Quality Assurance Project Plan

Kewaunee Marsh
Arsenic Impact Investigation
CD Besadny Fish & Wildlife Area
Kewaunee County, Wisconsin

STS Project No. 4-27393E

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Groundwater Monitoring
CD Besadny Fish and Wildlife Area
Town of Pierce, Kewaunee County, Wisconsin
Great Lakes National Program Grant No. GL96528001-0

Prepared by: STS Consultants, Ltd.

Prepared for: Wisconsin Department of Natural Resources

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Wisconsin Department of Natural Resources
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1.0 PROJECT MANAGEMENT

1.1 Project/Task Organization

1.1.1 Overview

STS Consultants, Ltd. (STS) has prepared this Quality Assurance Project Plan (QAPP) on behalf of the Wisconsin Department of Natural Resources (WDNR) to outline field and laboratory procedures related to groundwater monitoring on a 13-acre study site in the CD Besadny Fish and Wildlife Area, subsequently referred to as Kewaunee Marsh.

The WDNR, with financial assistance through the Great Lakes National Program Office (GLNPO) authorized preparation of this QAPP to support the environmental assessment of the Kewaunee Marsh. Procedures outlined in this document are consistent with applicable professional technical standards, US Environmental Protection Agency (EPA) guidance, and specific project goals and requirements. This QAPP presents the organization, functional activities, and specific quality assurance (QA) and quality control (QC) activities associated with the groundwater sampling to be conducted at Kewaunee Marsh. This QAPP also includes specific protocols, which will be followed for sample handling and storage, Chain of Custody, and laboratory and field analysis.

The goal of the environmental monitoring is to facilitate environmental remediation planning and specifically address the feasibility of cleanup alternatives. Details of the QA/QC program are presented and supporting documentation is appended.

1.1.2 Wisconsin Department of Natural Resources

Ms. Annette Weissbach (Remediation & Redevelopment Program Project Manager)

- Authorize STS to proceed on the study work.
- Approve the work scope and QAPP
- Review and monitor project progress.
- Communicate with media and municipal officials.

Mr. James Killian (Watershed Bureau Project Manager)

- Technical Review

1.1.3 STS Consultants, Ltd.

Ms. Jan Tesch, Senior Project Manager:

- Coordinate work and provide status reports to WDNR Project Manager.
- Coordinate field data collection work
- Collect and maintain data.
- Update existing Health & Safety Plan.
- Prepare project progress reports for the WDNR.
- Coordinate activities of STS personnel.
- Review interpretation of data.
- Review investigation results.
- Report investigation results to WDNR.
- Provide conclusions and recommendations.
- Attend project progress meetings with WDNR.

Mr. Paul J. Killian, P.E., Principal Engineer:

- Provide direct supervision to the STS Project Manager.
- Assist with project scoping.
- Review project contracts and conditions.
- Review field and analytical test results with team to discuss conclusions and recommendations prior to report preparation.
- Provide conclusions and recommendations.
- Provide final review of all critical documents.

1.1.4 Wisconsin State Laboratory of Hygiene

Mr. George Bowman (Wisconsin State Laboratory of Hygiene, Chemistry Management Supervisor, ESS - Inorganic Chemistry Department)

- The Wisconsin State Laboratory of Hygiene will perform arsenic speciation analysis on samples collected by the WDNR and STS.

1.1.5 USFilter/Enviroscan Services

Mr. James Salkowski (Laboratory Director USFilter/Enviroscan Services [USFilter])

- USFilter will provide groundwater and surface water chemical analysis.

1.1.6 Environmental Protection Agency (EPA)

Ms. Mary Beth Giancarlo Ross (EPA GLNPO Project Manager)

- Grant oversight and management

Mr. Louis Blume (EPA GLNPO Quality Assurance Project Manager)

- EPA QA/QC management

1.2 Project Summary

1.2.1 Problem Definition

A historic arsenic release is a continuing threat to groundwater and surface water within the Kewaunee Marsh. Current concentrations of arsenic exceed soil, groundwater, and surface water quality criteria and represent a direct contact risk.

The purpose of this sampling effort is to collect and interpret site data to support a feasibility study to determine remediation methodologies which may be implemented on the project site.

1.2.2 Background

The CD Besadny Wildlife Area consists of over 22,000 acres of state-owned property including wetlands, farmland, forest, and stream habitats located in Pierce Township, Kewaunee County, Wisconsin. In August 1992, the WDNR was notified that a wetland area within the wildlife boundaries was either devoid of vegetation or showed signs of significant vegetative stress. Subsequent sampling indicated elevated concentration of arsenic in soil and groundwater samples collected for within the wetland area.

The impacted area is located in the SW 1/4, Section 7, T23N, R25E, Town of Pierce, Kewaunee County, and is approximately 1 mile northwest of State Highway 42 (STH 42) along a former railroad track once operated by Fox Valley & Western Railroad. Investigation efforts by both the WDNR and the railroad suggest that the subsurface degradation is a result of a surface release of sodium arsenate, which presumably occurred sometime between 1938 and 1950. During the 1930s and 1940s, sodium arsenate was used as an insecticide on the cherry producing orchards of Door County and the surface release may have been the result of a train derailment, which reportedly occurred in this area.

Since the initial WDNR sampling in October 1992, the arsenic-impacted wetland area had been characterized by collecting and analyzing numerous soil, groundwater, and surface water samples. Site assessment and corrective action efforts to date have included the following:

1994	Preliminary assessment of site conditions completed by STS on behalf of Fox Valley & Western Railroad. The investigation included collecting surface water samples, groundwater samples from shallow groundwater monitoring wells installed on the site, sediment samples, and soil pore water.
1996	Interim actions completed to reduce the direct contact risk in the area of greatest impact included installation of an approximate 3.25-acre textile/wood chip cover over visibly impacted areas of the marsh and enclosing an approximate 15-acre area with a security fence. Additional groundwater and surface water monitoring points were established downgradient and sidegradient to the cover. Groundwater and surface water samples from the Kewaunee River were collected and analyzed by the WDNR. Water levels of the Kewaunee River were monitored.
1997	Groundwater and surface water modeling was done to estimate the environmental fate and migration potential of arsenic. The model indicated that the transport of arsenic in the groundwater at the site is very slow, with the model predicting that the maximum concentration of arsenic in the groundwater would reach the Kewaunee River in approximately 2,800 years. The groundwater model generally simulated dissolved arsenic migration based on estimated sorption through the saturate organic soil. The results indicated that the maximum stormwater arsenic concentration was 28.3 micrograms per liter (µg/L); the highest downstream arsenic concentration was 15.6 µg/L. The surface water model predicted arsenic migration in surface water using the ration of arsenic mass and total runoff from regional sub-basins.
2000	A baseline risk assessment was completed by the WDNR for the Kewaunee Marsh site. The WDNR published its Baseline Ecological Risk Assessment (BERA) of the Arsenic Contaminated Wetland Associated with the CD Besadny Fish and Wildlife Area and the Kewaunee River. The BERA was conducted to determine the present and future risks to wildlife, birds, and aquatic resources from exposures to arsenic in the soil, sediment, groundwater, and surface water following implementation of the interim action at the site. The BERA also documented the degree of uncertainty and quality of the data available in performing the risk assessment and indicated that further investigation of soil, sediment, groundwater and surface water is warranted to determine if the interim action cover would be sufficient to protect the environmental and public health.
2001	In 2001, the WDNR Bureau of Watershed Management conducted additional investigations at the site, including installation of ten new groundwater monitoring wells. Ten surface water samples and ten soil/sediment samples were collected and the water levels in the Kewaunee River were monitored to determine if a correlation existed between groundwater levels in the marsh and the water level of the Kewaunee River.
2002/2003	STS continued site investigation work to evaluate the potential contaminant transport mechanisms in the marsh. Additional groundwater monitoring wells were installed on the project site, and soil, groundwater and surface water, and sediment samples were collected for analysis. Work also included defining geochemical conditions throughout the study area, an evaluation of the condition of the interim cover to determine whether it is sufficient to protect the environment and human health, and creation of a Geographical Information System database to manage the environmental data collected from the site. A Site Assessment and Remedial Actions Alternatives Report were prepared describing study findings, which also included an evaluation of remedial action options and recommendations.

Report references are provided in Section 5.0.

1.3 Project/Task Description

1.3.1 Project Description

The project scope of work will include the continued monitoring of groundwater in the study area to advance the following goals further:

- Define groundwater conditions near the apparent source of the release,
- Characterize the potential impact to the Kewaunee River,
- Monitor surface water quality and flow in primary drainage ditches within the impact zone,
- Define contaminant transport mechanisms, and
- Obtain samples for completing soil column studies to determine sediment/water distribution of arsenic.

This QAPP is limited to groundwater sampling and chemical analysis tasks; those efforts funded by GLNPO.

An arsenic speciation study will also be completed to determine the predominant oxidation state of the arsenic contamination on the project site for use in evaluating possible remediation methods. The predominant oxidant state of arsenic in soil in groundwater is pentavalent (+5) and trivalent (+3). Pentavalent arsenic or arsenate is less toxic than arsenite (trivalent arsenic). Arsenite is the reduced state of inorganic arsenic and is considerably more toxic, more soluble, and more mobile than the oxidized state.

1.3.2 Sampling Design Rationale

The sampling plan will utilize existing groundwater monitoring wells and established surface water sample collection points to further define arsenic transport mechanisms in the project area of the Kewaunee Marsh, with the ultimate use of the data being applied to determine the feasibility of potential remediation technologies.

Previously collected data indicates that elevated arsenic concentrations are most prevalent in the upper 5 feet of soil on site. The movement of groundwater both horizontally and vertically through the subsurface soil contributes to arsenic migration. Additionally, dissolution of arsenic and the transport of suspended solids by surface water runoff is also a significant factor in arsenic transportation toward the Kewaunee River. Previous monitoring programs have identified soil

conditions on the project site and the direction of groundwater flow, and provided a preliminary indication of the rate of arsenic transport though the project site.

Groundwater monitoring wells have been installed in a well distributed pattern over the 13-acre site. The wells are located upgradient and sidegradient to groundwater flow direction, near the source area of the contamination (now beneath the interim cover), and downgradient of the source. Some of the networked wells on the project site are nested to define the vertical changes in groundwater quality, while others are single well installations.

Previous investigations of soil and sediment conditions concluded that approximately 20 feet of organics, organic silts, and silty clays are found above low permeability clay soil on the project site. Nested wells, consisting either of a grouping of three wells with screens placed at 3 to 5, 8 to 10, and 18 to 20 feet below ground surface (bgs) or two wells with screens placed at 3 to 5 and 8 to 10 feet bgs, where installed to intersect different vertical zones of the organic soils.

Individual or nested groundwater wells located upgradient or sidegradient to the source will be sampled annually to monitor conditions on the perimeter of the impact zone. Concentration levels of arsenic in upgradient and sidegradient wells are expected to remain consistent. Change in concentration would likely indicate a change in groundwater flow patterns. The well network in and downgradient of the source area will be sampled on a quarterly basis to determine seasonal influences to groundwater quality. Analytical data from the existing groundwater well network will adequately characterize groundwater quality at various depths and reflect changes in arsenic concentrations laterally and vertically through the marsh.

Surface water on the project site is known to transport arsenic via in both dissolved and suspended form. The primary routes of surface water transport should be identified in order to determine a suitable remediation for surface water collecting on the project site. Sampling points are located along two separate shallow drainage swales within the marsh that direct surface water through the marsh toward the Kewaunee River. Samples obtained from the two swales are expected to be representative of typical surface water flow transport mechanisms through the marsh.

Groundwater and surface water samples obtained from the project site will be analyzed for arsenic, sulfate, sodium, iron, nitrate, dissolved oxygen, field ph, field conductivity, temperature, and redox potential. These parameters were selected to identify oxidation-reduction conditions

throughout the study area to assist in defining arsenic transport mechanisms which will influence site remediation planning and implementation.

Sampling locations are shown on Figure 1. The following table is a summary of the proposed sampling program.

Program Summary

Sampling Point	Frequency	Parameter Analysis
MW02-3, 3i, 3d MW02-4, 4i, 4d MW02-5, 5i MW02-6, 6i MW02-7, 7i MW02-8 GW01-2 GW01-3 GW01-5 GW01-6 GW01-8 MW04-9 MW04-10 MW04-11 MW04-12 MW04-13	Quarterly	Arsenic Sulfate Sodium Iron Nitrate Dissolved Oxygen (DO) Field pH, Conductivity, Temperature, and Redox Potential
MW02-1, 1i, 1d MW02-2, 2i MW02-7d MW02-8i GW01-1 GW01-4 GW01-10	Annually	Arsenic Sulfate Sodium Iron Nitrate DO Field pH, Conductivity, Temperature, and Redox Potential
Surface Water SW1 SW2 SW3 SW4 SW5 SW6	Semi-Annual	Arsenic Sulfate Sodium Iron Nitrate DO Field pH, Conductivity, Temperature, and Redox Potential

1.3.3 Data Uses and Expected Measurements

Soil, groundwater, and surface water are known to be impacted on the project site. The data obtained from this study will be used to characterize the arsenic concentration present on the site to define the degree and extent of groundwater degradation.

1.3.4 Data Quality Objectives for Measurement Data

The overall Quality Assurance objective is to develop and implement procedures for field sampling, Chain of Custody, laboratory analysis, and reporting, which will provide results that are legally defensible in a court of law. Specific procedures for sampling, Chain of Custody, laboratory instrument calibration, laboratory analysis, reporting of data, internal quality control, audits, preventive maintenance of field equipment, and corrective actions are described in other section of this QAPP.

Data quality objective from measurements during this project will be addressed in terms of precision, accuracy, representation, completeness, and comparability (PARCC).

- Precision is a measure of the degree to which two or more measurements are in agreement.
- Accuracy is the degree of agreement between an observed value and an accepted reference value.
- 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 circumstances.
- Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is also dependent on similar QA objectives.
- Representation expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

The numerical PARCC parameters will be determined from the project Data Quality Objectives (DQOs) to insure that they are met. The Data Quality Objectives of a project are determined through a process of planning steps designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for the intended application. A description of general DQO planning steps is included as Appendix A.

1.3.4.1 Field Precision Objectives

Field precision is assessed for groundwater through the collection and measurement of field duplicates at a rate of one duplicate per twenty analytical samples (as required by the WDNR). Field measurements for this project include obtaining groundwater elevations, field pH, field temperature, specific conductivity, redox potential, and dissolved oxygen (DO). The QA precision objectives for field measurements are in Table 1.

1.3.4.2 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of Relative Percent Differences (RPD) for duplicate samples and Relative Standard Deviation (RSD) for three or more replicated samples. The equations to be used for precision in this project can be found in Section 4.3.1 of this QAPP. Precision control limits are provided in Table 2.

1.3.4.3 Field Accuracy Objectives

Accuracy in the field is assessed through the use of field and trip blanks and through the adherence to all sample handling, preservation, and holding times. Field blanks will be collected and analyzed at a rate of 1 blank per 20 analytical water samples. Trip blanks for water analysis will be prepared and analyzed at a rate of one trip blank per shipping container. The QA accuracy objectives for field measurements are listed in Table 1. The Standard Operating Procedures (SOPs) for instruments used to obtain the field measurements of water quality parameters are found in Appendix B.

1.3.4.4 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of Matrix Spikes (MS) or Standard Reference Materials (SRM) and the determination of percent recoveries. The equation to be used for accuracy in this project can be found in Section 4.3.3 of this QAPP. Accuracy control limits are given in Table 2.

1.3.4.5 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 4.3.5 of this QAPP. Field completeness for this project will be greater than 90%.

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 4.3.5 of this QAPP. Laboratory completeness for this project will be greater than 95%.

1.3.4.6 Measures to Ensure Representation of Field and Laboratory Data

Representation is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the Sampling and Analysis Plan (SAP) is followed and that proper sampling techniques are used. The portion of each water sample to be analyzed for arsenic speciation will be placed in a glass container with dry ice/solvent slurry preservation in a manner to minimize volatilization. Other samples will be collected in appropriate containers based on analyses and placed on ice immediately after collection.

1.3.4.7 Measures to Ensure Representation of Laboratory Data

Representation in the laboratory is ensured by using proper analytical procedures, meeting sampling holding times and analyzing and assessing field duplicate samples. The sampling network, discussed in the groundwater and surface water SAP is designed to provide data representative of the site conditions. The SAP is included in Appendix C.

1.3.4.8 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the SAP is followed and proper sampling techniques are used.

1.4 Instructions for Documentation and Records

Records that will be generated as part of this subsurface investigation are a critical aspect of a successful project.

1.4.1 Documents and Records

Sample Collection Records: These records will show that proper sampling protocol was performed in the field. This documentation will include the sample number, sample collection points, names of the persons conducting the activity, maps and diagrams, equipment/methods used, climatic conditions, and unusual observations. Field notebooks and field note forms will be used to record raw data and record changes in planned activities. The field books will include numbered pages with data and signature lines.

QC Sample Records: These records will be kept on the field sampling forms and will include information regarding field, trip, and equipment rinsate blanks and duplicate samples. They also will include sample integrity and preservation information.

Field Analysis Records: The field analysis records will include Chain of Custody records, sample receipt forms/sample tracking forms, tabulated data summaries, and raw data for field samples and QC samples.

Fixed Laboratory Records: Fixed laboratory records that will be compiled if available and appropriate will include Chain of Custody records, sample receipt forms, log books, tabulated data summary forms, and raw data for samples, standards, QC samples, and corrective action reports.

Data Handling Records: The document protocols used in data reduction, verification, and validation will be kept in the project log book.

1.4.2 Data Reporting Package Format and Documentation Control

1.4.2.1 Field Documentation

Field documentation refers to the record of data collection activities performed at the site. Documentation should be completed in sufficient detail so that persons going to the facility could reconstruct a particular situation without reliance on memory. Field documentation will be provided using a combination of field book entries and field log forms as described below.

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

The title page of each log book will contain the following:

- Person to whom the log book is assigned,
- Log book number,
- Project name,
- Project start date, and
- End date.

Entries into the log book 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 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 log book.

Measurements made and samples collected will be recorded. All entries will be made in ink, signed, and dated; and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark, which is signed and dated by the sampler.

Field data collection information will be recorded in a bound field notebook with pre-numbered pages and appropriate sampling logs, as described below. The following information will be documented in the field notebook:

For each site visit: The site name and location, date, starting and ending times, weather, purpose for site visit, names of all people involved in sampling activities, level of personal protection used, documentation of adherence to protocol, any changes made to planned protocol, names of visitors to the site during sampling and reason for their visit, unusual observations, and signature of the person recording the information.

For each groundwater sample: The groundwater sampling information will be tabulated and will include the following: well number; condition of well including depth to bottom of well and height of PVC stick-up; depth to groundwater; the calculated amount of four well-volumes; the actual amount of water purged from the well; whether the well purged dry; time of sample collection; field measurements such as pH, temperature, conductivity and dissolved oxygen (DO) and redox potential; general field observations, such as color, odor, and turbidity of the collected sample will

be recorded in the field notebook. The field notebook will also reference the Chain of Custody document numbers used to track sample delivery to the laboratories.

1.4.2.2 Laboratory Documentation

The following will be included in the laboratory data package:

- Case narrative
- Calibration (initial/continuing) summary and raw data
- Mass spectrometer tuning data (if appropriate)
- Gas chromatogram (if appropriate)
- Mass spectra (if appropriate)
- Quality control summary forms and raw data
- ICP, AA, and graphite furnace data outputs (if appropriate)
- Inter-element correction data (if appropriate)
- Blank data results
- Method and instrumental detection limit results

1.4.3 Data Reporting Package Archiving Retrieval

The final data package will be kept at the offices of STS as described in Section 2.4.4.

1.4.4 Project Schedule

A tentative project schedule is provided in Table 3. Project personnel should be contacted regarding significant schedule changes.

1.4.5 Special Personnel and Training Requirements

Field personnel entering the project site will be required to have OSHA 40-Hour Health & Safety training.

2.0 MEASUREMENT DATA ACQUISITION

The purpose of the QAPP is to produce reliable data that will be generated throughout the investigation by:

- Ensuring data validity and integrity;
- Assuring and providing mechanisms for ongoing control of data quality;
- Evaluating data quality in terms of PARCC; and
- Providing usable, quantitative data for analysis, interpretation, and decision making.

2.1 Sample Network Design and Rationale

This work is a continuation of the previous groundwater and surface water monitoring efforts on the project site to document current arsenic concentrations and the progression of the contaminant plume toward the Kewaunee River.

The sampling plan will utilize existing groundwater monitoring wells and established surface water sample collection points to further define arsenic transport mechanisms in the project area of the Kewaunee Marsh with the ultimate use of the data being applied to determine the feasibility of remediation design.

A summary of the sampling program, including sample matrices, analytical parameters, and frequency of sample collection is provided in Table 4.

Figure 1 is a site map showing the location of soil borings, surface soil and water sampling locations, and groundwater monitoring wells on the project site.

2.2 Analytical Method Requirements

The components of data acquisition for the investigation are discussed in detail in the QAPP for the project. Sample collection, preparation, and decontamination procedures are also referenced in the QAPP. Sample preservation, holding times, and volume requirements as specified by SW-846 for samples collected as part of this project will be strictly adhered to by the laboratory. The surface water and groundwater samples will be analyzed for known and suspected contaminant parameters common to past activities and RECs associated with each subject property.

All environmental media samples will be collected and analyzed in accordance with the SW-846 methods as discussed below. Bottle/containers utilized for the collection of samples will be provided by the vendor selected to supply bottles/containers for the project. Bottles provided will be cleaned by the vendor to USEPA specifications. Also, sample collection activities will conform to STS's standard procedures as presented in Appendix B. Trip blanks prepared in accordance with USEPA Methods will be requested from the vendor supplying the bottles. The trip blanks will be sent with the sample containers by the selected vendor. SOPs from the vendor will be supplied upon request. This project will utilize the analytical services of the Wisconsin State Laboratory of Hygiene (WSLH), Environmental Division. Laboratory methods and applicable target detection levels are provided in Table 2.

STS field personnel will review and/or have available to them a copy of the USEPA publication, Sampler's Guide to the Contract Laboratory Program (EPA/540/12-96/032) to assist in understanding the protocols associated with sample collection. Appropriate preservatives will be added by the bottle vendor prior to shipment to STS.

2.3 Sample Handling and Custody Requirements

The admissibility of environmental data as evidence in a court of law is dependent upon custody of the data, among other factors. Custody procedures will therefore be used to document the relevance and authenticity of data collected during the investigation at each site. The data requiring custody procedures includes both field samples and data files, which can include field books, logs, and laboratory reports.

Various aspects of sample handling and shipment, as well as the proposed sample identification system and documentation, are discussed in the following subsections.

2.3.1 Sample Handling

The sampling procedures to be used in this site investigation will be consistent for the purpose of this project. The procedures used for groundwater sampling are specified in the SOPs provided in Appendix B.

Sample containers will be individually wrapped with foam or bubble-wrap material to reduce the potential for breakage during transit. Secondary containment, consisting of sealable, plastic

Ziplock® bags, will be placed around each sample container to prevent the release of a sample in the event of breakage. Sample containers and preservation and holding time requirements are summarized in Table 5.

Samples will be handled with minimum contact and gloved hands. New, disposable vinyl gloves will be used to handle each sample. Samples will be chilled in the field and during transport to the laboratories using frozen water. The cold packs will be placed into plastic sealable Ziplock® bags to contain leakage and condensation. Insulated shipping containers (coolers) for transporting samples to the laboratory shall be clean and undamaged. The drain shall be sealed to prevent leakage.

The signed custody record will be enclosed within and secured to the inside top of each shipping container. 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 at least two locations.

Samples will be transported to the laboratory by overnight carrier the same day the samples are collected in the field.

2.3.2 Sample Identification System

A specific identification number will be assigned to each sample prior to collection. The sample identification system is described in general below:

A specific sample number will be assigned to each sample prior to collection. The sample number will consist of the following:

- Project code: The project will be identified with a six digit code unique to the project.
- Location Code: each sample will be identified with a code which indicates the well location where the sample was collected. The specific sample location numbers are identified in Figure 1.

- Sample Type Code: each sample will be identified with a code which indicates the type of sample as follows:

  GW = groundwater

  SW = surface water

  AS = arsenic speciation

  B = field blank

  D = field duplicate

- The sample collection date will be identified using six digits representative of the date (041015)

- Examples of sample numbers:

  GW-27393E-MW04-13, 041015 is a groundwater sample collected under Project 27393E from Location MW-04-13 on October 15, 2004.

  SW-27393E-SW-2, 041015 is a surface water sample collected under Project 27393E from Location SW-2, Well SB-15 on October 15, 2004.

  SW-27393E SW2D, 041015 is a duplicate sample.

Sample labels will be attached to each individual sample bottle. The label will include the field sample number (described above), date/time of collection, type of analysis, sampler initials, and STS project number. Labels will be annotated with waterproof, permanent ink.

2.3.2.1 Sample Labeling and Analysis Scheduling for Samples Shipped to Analytical Laboratory

Sample labels will be prepared by STS field personnel. A sample label will be attached to each individual sample bottle. The label will include the field sample number (as described in the preceding section), date/time of collection, type of analysis, and sampler initials. Labels will be annotated with waterproof, permanent ink. An example label is included in Appendix C.

The laboratory will be notified of impending sampling events. The laboratory will verify receipt of samples by fax to the STS Project Manager. The laboratory will schedule the sample analysis so that analyses are completed within the method holding times.

2.4 Sample Custody

Custody is one of several factors which are necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field

sample collection, laboratory analysis, and final evidence files. Final evidence files are maintained under document control in a secure area of STS.

The custody sequence can be divided into three major segments: collection (field), laboratory analysis, and final evidence files. Within any of these segments, a sample or evidence file is in someone's custody if:

1. It is in his/her actual physical possession,
2. It is in his/her view, after being in his/her actual possession,
3. It is in his/her physical possession, and he/she has placed it in a secure (locked) location, or
4. It is in a designated secure area.

2.4.1 Field-Specific Custody Procedures

The sample packaging and shipment procedures summarized below will insure the samples will arrive at the laboratory with the Chain of Custody intact. An example Chain of Custody form is provided in Appendix D.

Field procedures are as follows:

(a) The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly shipped. As few people as possible should handle the sample.

(b) All bottles will be labeled with a sticker showing the sample number, preservative used, date, time, analysis to be performed on the sample, and project number.

(c) The STS Project Manager must review all field activities to determine whether proper custody procedures were followed during the fieldwork and decide if additional samples are required. He or she should notify the Ms. Annette Weissbach of a breach or irregularity in Chain of Custody procedures.

(d) The Project Geologist/Environmental Technician will maintain as appropriate field log book and field notes to document location, time and type of sample(s) collected and to document site specific conditions and should designate the sample numbering system utilized during the investigation.

Transfer of Chain of Custody and shipment procedures are as follows:

(a) Samples will be picked up and delivered to the analytical laboratory via private carrier and accompanied by a properly completed 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 procedure will be used to transfer custody of samples from the sampler to another person, to a mobile laboratory, to the permanent laboratory, or to/from a secure storage area.

(b) A Chain of Custody record identifying the contents of the cooler will accompany each cooler. The top two copies of the Chain of Custody forms (white and green copies) should be placed in a plastic bag and taped to the inside cover of the cooler. The remaining three copies will be kept in the STS project file.

(c) Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis. Shipping containers will be secured with strapping. The strapping tape will be placed in at least two locations on the cooler.

(d) A bill of lading will be used to document the pick-up/delivery of samples by the commercial courier service. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the seals remain intact.

(e) Samples will be packaged according to EPA guidelines by using appropriate packing material and ice.

Samples shipped to the laboratory will be documented on the Chain of Custody form. The completed forms will include all required information as identified on the applicable form. An example of the completed form is included in Appendix D. The completed form will be enclosed in a sealable bag and taped to the inside lid of the cooler that contains the samples listed on the form.

2.4.2 Documentation

Sample labels will be attached to each individual sample bottle. The label will include the field sample number as described in the SAP (Appendix C), date/time of collection, type of analysis, sampler initials, and the assigned project number. Labels will be annotated with waterproof, permanent ink. A sample label is included in Appendix D.

The WSLH will be notified and analytical work will be scheduled. A minimum of one week before sampling, sample containers will be ordered from the lab. Sample containers are anticipated to be received by the sampling technician within one week of order. Samples will occur and samples collected will be shipped to the laboratory. When samples are received at the laboratory, their condition is noted on the Chain of Custody form and a copy of the completed Chain of Custody will be faxed back to STS to document receipt.

2.4.3 Laboratory Chain of Custody Procedures

The Chain of Custody procedures for WSLH and USFilter are described in QA/QC Manuals provided in Appendix E.

2.4.4 Final Document Files

The final document files will be the central repository for all documents which constitute evidence relevant to sample and analysis activities as described in the QAPP. STS will be the designated custodian of the evidence file (excluding the original laboratory data package), and will maintain the contents of the evidence files for the site, including all relevant records such as: reports, logs, field notebooks, other field records, correspondence, Chain of Custody documents, and regulatory communications. The final evidence file will be secured in a limited access area under the custody of the STS facility manager. The original laboratory data package will be maintained as a final evidence file by STS.

Collectively, the final evidence file of STS will include at a minimum:

- Field log books
- Field data and deliverables
- Photographs
- Drawings
- Soil boring logs
- Laboratory data deliverables
- Data validation reports
- Data assessment reports
- Progress reports, QA reports
- All custody documentation

The final document file will be maintained in a secure, limited access area for a minimum of three years after submittal of the final report.

2.5 Quality Control Requirements

All sample containers utilized for this project will be procured from the analytical laboratory, along with an adequate supply of certified trip blank samples. Sample containers will be cleaned to

EPA specifications. Where preservatives are required for water samples, the analytical laboratory will include the appropriate type and volume of preservative in the sample container.

Field blank, trip blank, method blank, duplicate, standard reference materials, and matrix spike samples will be analyzed to assess the quality of the data resulting from the field sampling and analytical programs.

Field and trip blanks, consisting of distilled/de-ionized water, will be submitted to the analytical laboratory to provide a means to assess quality of the data resulting from the groundwater field-sampling program. Field blanks are analyzed to check for procedural contamination introduced during sampling which may cause sample contamination. Field blanks will be generated at a rate of one for every ten or fewer groundwater samples, with a minimum of one per day.

Trip blanks are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Trip blanks are prepared prior to the sampling event and are kept with the investigative samples throughout the sampling event. They are then packaged for shipment with other samples and sent for analysis. There will be one trip blank included in each sample-shipping container. At no time after their preparation are the sample containers opened before they reach the laboratory. Trip blanks for groundwater samples will consist of distilled water.

Method blanks are generated within the laboratory and used to assess contamination resulting from laboratory procedures. A method blank will be run each day, or at frequencies specified in the laboratory SOPs for each analysis.

Duplicate samples are analyzed to check for sampling and analytical reproducibility. Duplicate samples will generally be collected at a frequency of one for every ten or fewer investigative samples of a liquid matrix, as per WDNR requirements.

The QC level of effort for the field measurements consists of daily instrument calibration in accordance with manufacturer specifications, and using an appropriate calibration standard.

2.5.1 Field QC Requirements

The standardization and QA information for dissolved oxygen (DO), pH, field temperature, reduction oxidation potential, and field conductivity field measurements are described in Appendix B of this QAPP. Field parameter measurements will be obtained using a closed flow-through cell with Hariba Water Quality Monitoring System on downhole probes; Oakton Portable Meter or Orion Meter. Hariba U-22 SOPs will be followed when using the closed flow-through cell. Horiba SOP will be supplied upon request. In the event a probe monitor could not be used to obtain DO measurements, a chemetrics colormetric test will be used.

QA/QC procedures for measuring water levels will include verification that the instrument is operating and taking duplicate measurements at 10% of the sampling points.

Assessment of field sampling precision and bias will be made by collecting field duplicates and field blanks for laboratory analysis. Collection and frequency of samples will be in accordance with the applicable procedures in Section 2.5 of this QAPP.

2.5.2 Laboratory QC Requirements

A complete listing of project target compounds, project quantitation limits, and current laboratory-determined detection limits for each analyte group can be found in Table 2 for each analyte group described above, respectively.

The laboratory SOPs include a QC section which addresses the minimum QC requirements for the analysis of specific analyte groups. Section 2.5 of this QAPP specifies the rate at which associated QC samples will be taken for each analyte group or matrix.

Internal quality control procedures for WSLH are specified in Sections 7 and 10 of the WSLH QA/QC manual (Appendix E). Internal quality control procedures for USFilter are specified in Section 7 through 10 in the USFilter QA/QC Manual (Appendix E). These specifications include the types of QC checks required (method blanks, reagent/preparation blanks, matrix spike and matrix spike duplicates, duplicate/replicate analysis, calibration standards, internal standards, surrogate standards), the frequency of audits, the specific calibration check standards, compounds and concentrations to be used, and the quality control acceptance criteria for audits.

2.6 Calibration Procedures and Frequency

This section describes the calibration procedures and the frequency at which the procedures will be performed for both field and laboratory instruments.

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

To reduce downtime and interruption in field sampling and laboratory testing, it is necessary to adhere to a schedule of routine preventative maintenance. This section presents a summary of the maintenance schedule in-place for field and laboratory instrumentation.

2.6.1 Field Instruments/Equipment

Equipment which will be used for 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 ensure that maintenance requirements are being observed. Field notes from previous sampling trips will be reviewed, so notations on any prior equipment problems are not overlooked and all necessary repairs to equipment have been carried out.

Calibration of field instruments is governed by the specific SOP for the applicable field analysis method, and such procedures take precedence over the following general discussion.

Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. Calibration checks will be performed daily. Instruments will include a water level indicator, pH meter, conductivity meter, and DO meter. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service (see additional calibration details as provided in the SOP included in Appendix B).

Preventive maintenance is routinely performed on all field instrumentation. Field personnel are trained in routine maintenance procedures. Equipment which requires maintenance and/or service outside the capabilities of the in-house personnel are performed by the manufacturer or another qualified source.

Specific preventative maintenance procedures to be followed for field equipment are those recommended by the manufacturer. Field instruments will be checked and calibrated before use. Calibration checks will be documented on the field logbooks.

The maintenance schedule and troubleshooting procedures for field instruments are indicated in Table 6.

Critical spare parts will be kept on site to reduce downtime. Backup instruments and equipment will be available on site or within one day shipment to avoid delays in the field schedule.

Detailed logbooks document the preventative maintenance and repairs performed on each instrument.

2.6.2 Laboratory Instruments

Calibration procedures for a specific laboratory instrument will consist of initial calibration (three or five points), initial calibration verification, and continuing calibration verification. The SOP for each analysis performed in the laboratory describes the calibration procedures, their frequency, acceptance criteria, and the conditions that will require calibration. In all cases, the initial calibration will be verified using an independently prepared calibration verification solution.

The laboratory maintains a sample log book for each instrument which will contain the following information: instrument identification, serial number, date of calibration, analyst, calibration solutions run, and the samples associated with these calibrations.
Calibration procedures and frequency are specified in Chapters 11 and 12 of the WSLH Environmental Health Division Quality Assurance Procedures and Policies (Appendix E) and Sections 5 and 6 of the USFilter QA/QC Manual (Appendix E).

Preventive maintenance practices employed by the WSLH are described in Chapter 11 of their QA/QC Manual (Appendix D). Section 8 describes preventive maintenance practices employed by USFilter.

2.7 Data Management

2.7.1 Data Reduction

2.7.1.1 Field Data Reduction Procedures

Direct reading instruments will be used in the field. The data generated by the instruments will be written into field logbooks immediately after the measurements are taken. Errors, if any, in the logbook will be crossed out with a single line and corrected in the space adjacent to the original (erroneous) entry. The error/correction will be initialed and dated by the field personnel making the correction.

2.7.1.2 Laboratory Data Reduction Procedures

Data reduction and reporting for samples analyzed by the state lab will be performed according to specifications in the SOPs for the specific methods and as described in Chapter 8 of the WSLH lab's QA/QC Manual (Appendix E). The data will be reviewed by the lab analyst, who will check the sample data and the associated QA data for adherence to QA procedures and requirements. Data which do not meet the QA acceptance criteria will be appropriately flagged.

2.7.2 Data Reporting

Data cannot be adequately evaluated unless supported by a complete data package. The field and laboratory data packages should include the elements specified below and be reported according to the procedures outlined.

2.7.2.1 Field Data Reporting

Field data reporting will be conducted through transmission of: 1) report sheets containing tabulated results of field measurements; 2) documentation of field calibration activities; and 3) documentation of field sampling locations and activities/circumstances, which may potentially affect the data quality and accuracy.

2.7.2.2 Laboratory Data Reporting

The laboratory data generated by the state lab will not be reported until it has been reviewed. The QA manager will perform a final review of the laboratory's report summaries and case narratives to determine whether the report meets the specified requirements. The laboratory report format of the WSLH is documented in Chapter 8 of the WSLH lab's QA/QC Manual (Appendix E) and in Section 12 of the USFilter QA/QC Manual (Appendix E).

3.0 PEFORMANCE AND SYSTEM AUDITS AND RESPONSE ACTIONS

The purpose of a quality control audit is to provide an objective, independent assessment of a measurement effort. It ensures that field and laboratory data generating, data gathering, and measurement activities produce reliable and useful results. Cases can occur in which inadequacies are identified in the measurement system. In such cases, audits provide the mechanism for implementing corrective action. In the event an audit questions the validity of the field or laboratory's test results, the quality assurance (QA) auditor can take corrective action.

Performance and system audits of both field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the SAP and QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits.

3.1 Field Performance and System Audits

3.1.1 Internal Field Audits

Field activities will be monitored internally by the STS Quality Assurance/Quality Control (QA/QC) officer. The QA/QC officer will not be involved with the actual physical sampling, but will be knowledgeable regarding the project objectives and QA/QC project requirements.

The QA/QC officer's field audit responsibilities will include the following:

1. Document the various sample program QA/QC objectives, including performance requirements, number of samples required, precision, accuracy, measurable deliverables, and schedules.
2. Verify that sampling personnel are suitably trained and informed regarding the project objectives and QA/QC objectives.
3. Verify sampling personnel conformance with SAP and QAPP.
4. Verify proper equipment decontamination procedures are followed.
5. Examine sampling records.
6. Examine field instrument operating records.
7. Verify that sufficient quality control samples are taken and taken properly.
8. Verify that sampling objectives are being met and Chain of Custody procedures are being followed,

9. Document QA activities including: sampling personnel, procedures utilized, time, dates, location, and justification for any deviations from the SAP and/or QAPP

Internal field audits will be conducted at least once near the beginning of the sampling program. The field audit checklist to be used for this project is included as Appendix F.

3.1.2 External Field Audits

External field audits may be conducted by the WDNR.

External field audits may be conducted at any time during the field operations. These audits may or may not be announced and are at the discretion of the WDNR.

External field audits will be conducted according to the field activity information presented in the QAPP.

3.2 Laboratory Performance and Systems Audits

The WSLH is externally audited approximately every two years by WDNR Certification personnel. Certification by the State of Wisconsin is conducted on an annual basis. A copy of the WSLH most current certification is provided in Appendix E with their QA/QC Manual. Their certification expires August 31, 2005. The lab performs internal audits on an annual basis. The lab's internal audit procedures are described in the WSLH QA/QC Manual included in Appendix E. USFilter is externally audited every two years by WDNR certification personnel and every two years by the Environmental Accreditation Program. Other external audits by the EPA and others are completed on a periodic basis. USFilter state laboratory certification expires on August 31, 2005.

3.3 Response Actions

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out of QC performance, which can affect data quality. Corrective action can occur during field activities, laboratory analyses, data validation, and data assessment. All corrective action proposed and implemented will be documented in the regular QA reports to management. Corrective action should only be implemented after approval by the Project Manager, or his designee, the field operations manager. If immediate corrective action is required, approvals secured by telephone from the Project Manager should be documented in an additional memorandum.

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 will be responsible for notifying the Project Manager, who in turn will notify the STS Project Manager, who may notify the WDNR Project Manager depending upon the type of noncompliance problem identified. Implementation of corrective action will be confirmed in writing through the same channels.

Any non-conformances with the established quality control procedures in the QAPP or SAP will be identified and corrected in accordance with the QAPP. The STS Project Manager or his designee will issue a non-conformances report for each non-conformance condition.

Corrective actions will be implemented as 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 from the STS or WDNR Project Manager.

3.3.1 Field Corrective Action

Corrective action in the field can be needed when the sample network is changed (i.e., more or less samples, sampling locations other than those specified in the QAPP, etc.) or sampling procedures and/or field analytical procedures require modification due to unexpected conditions. Technical staff and project personnel will be responsible for reporting all suspected technical deficiencies or non-conformances by reporting the situation to the Project Manager or designee. This manager will be responsible for assessing the suspected problems in consultation with the Project QA Manager. If they determine that the situation warrants reportable non-conformances requiring corrective action, 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-conformances reports and corrective actions taken;
- Ensuring non-conformances reports are included in the final site documentation project files.

If appropriate, the Project Manager will ensure that no additional work that is dependent on the non-conforming activities 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 Project Manager or his designee is responsible for all site activities. In this role, the manager at all times is required to adjust the site programs to accommodate site-specific needs. When it becomes necessary to modify a program, the responsible person notifies the manager of the anticipated change and implements the necessary changes after obtaining the approval of the manager.

The change in the program will be documented in the field books. The Project Manger must approve the change in writing or verbally prior to field implementation, if possible. If unacceptable, the action taken during the period of deviation will be evaluated in order to determine the significance of any departure from established program practices and action taken.

The Project Manager is responsible for the controlling, tracking, and implementation of the identified changes. Reports on all changes will be distributed to all affected parties.

Corrective action resulting from internal field audits will be implemented immediately if data may be adversely affected due to unapproved or improper use of approved methods. The QA officer will identify deficiencies and recommended corrective action to the Project Manager. Implementation of corrective action will be performed by the field operations manager and field team. Corrective action will be documented in QA reports to the entire project management.

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 the STS or WDNR Project Manager.

3.3.2 Laboratory Corrective Action

Corrective action in the laboratory may occur prior to, during, and after initial analyses. A number of conditions such as broken sample containers, multiple phases, low/high pH readings, or potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with lab analysts and section leaders, it may be necessary for the laboratory QC Coordinator to approve the implementation of corrective action. The standard operating procedures (SOPs) specify some conditions during or after analysis that may automatically trigger corrective action or optional procedures. These conditions may include dilution of samples, additional sample extract cleanup, automatic re-injection/re-analysis when certain quality control criteria are not met, etc. A summary of method-specific corrective actions are found in the SOPs (Appendix B).

Corrective action procedures are described in Chapter 12 of the WSLH QA/QC Manual and Section 8 of the USFilter QA/QC Manual (Appendix E) and in the SOP for Filing a Corrective Action Form (Appendix B).

3.4 Corrective Action During Data Validation and Data Assessment

The facility may identify the need for corrective action during either the data validation or data assessment. Potential types of corrective actions may include re-sampling by the field team or re-injection/re-analysis of samples by the laboratory.

These actions are dependent upon the ability to mobilize the field team, whether the data to be collected is necessary to meet the required quality assurance objectives (e.g., the holding time for samples is not exceeded, etc.). When the STS QA manager identifies a corrective action situation, it is the Project Manager who will be responsible for approving the implementation of corrective action, including re-sampling, during data assessment. All corrective actions of this type will be documented by the QA manager. If re-sampling is deemed necessary due to laboratory problems, the STS Project Manager must identify the necessary approach for the additional sampling effort.

3.5 Reports to Management

For the duration of the project, monthly progress reports will be prepared by the STS Project Manager or designee. The Progress Reports will be submitted to the WDNR for review. The final report will be submitted by the STS Project Manager. Reports will contain activities completed during the month and significant findings. At project completion, draft and final projects reports will be issued. The final project report will include a description of field activities, data tables, figures, geologic cross-sections, and a discussion and summary of the results and recommendations for additional investigation and/or remediation, as appropriate.

4.0 DATA VERIFICATION, VALIDATION, AND USABILITY

The section describes the QA activities that will be performed to ensure the data is scientifically defensible, properly documented, of known quality, and meets the project objectives. Data validation and verification criteria have been developed to identify and qualify data that does not meet the measurement performance criteria established in Section 3.0 of this QAPP. The first steps in the process are data reduction and reporting. The data, supported by a complete data package, is then evaluated for validity and usability.

4.1 Data Review, Validation, and Verification Requirements

The activities described in this section are intended to determine the degree to which the collected data meet the project objectives. In order to do so, the following must be considered.

Sampling Design - Each sample will be checked by the Field Technician for compliance with the sample collection specifications, including sample type, and location with respect to the vertical and horizontal planes.

Sample Collection Procedures - Deviations from the prescribed sample collection procedures (see the SAP) will be identified by the Field Technician and documented as samples are collected or, if deviations are discovered at a later date, upon discovery. QA audits of field procedures to be conducted by the STS QA Officer will also include documentation of adherence or deviation from prescribed procedures.

Sample Handling - Sampling personnel, laboratory personnel, and project management personnel should evaluate sample containers and preservatives for their appropriateness with respect to the nature of the sample and the type of analysis to be performed. The samples should be checked for proper labeling and chain of custody records to verify that the sample is representative of the point from which it was collected.

Deviations from prescribed sample handling procedures (Section 2.3 and SAP) should be documented on Chain of Custody forms and/or field log books and/or laboratory log sheets. If any events occur during sample handling and processing which may potentially affect the integrity of the samples, such events should be noted.

Analytical Procedures - The appropriateness of the analytical procedures applied to each sample should be verified by reference to this QAPP and appropriate attachments. QA audits of field and laboratory procedures will be conducted by the QA Officer to determine whether the field and laboratory analytical procedures were implemented as specified. Deviations from prescribed procedures (see Section 2.2 and SOPs in Appendix B) should be documented and evaluated to determine the potential effects, if any, of such deviations.

Quality Control - Quality control procedures are specified in Section 2.5. Out of control situations and the corrective action taken should be documented by the QA Officer. This documentation will be evaluated to determine the effect, if any, on data quality.

Calibration - Section 2.6 specifies the requirements for calibration of field and laboratory instruments. Documentation of calibration will be evaluated by the QA Officer and by the analytical laboratory's QA Officer, as appropriate to determine whether calibrations:

- Were performed within the acceptable timeframe prior to sample analysis.
- Were performed in the proper sequence.
- Included the proper number of calibration points.
- Were performed using standards at concentrations that bracket the range of reported results or results were flagged accordingly.
- Included the appropriate checks for system and calibration stability.

Data generated between a suspect calibration event and the subsequent satisfactory recalibration should be flagged appropriately.

4.2 Data Validation (usability) and Verification Methods

This section describes the processes by which the data will be validated and verified.

4.2.1 Validation of Data Generated by Analytical Laboratories

Data generated by the WSLH will be validated by the WSLH QA Manager as specified in Chapters 8 and 12 of their QA/QC Manual (Appendix E). Data generated by USFilter will be validated by the USFilter QA/QC Officer as specified in Sections 2, 7, and 11 of the USFilter

QA/QC Manual. Each analytical report will be reviewed for compliance with the applicable method and for the quality of the data reported.

4.2.2 Validation of Field Data

The field data will be validated by the STS QA Officer who will evaluate the following:

- Holding times (field analyses should be performed on the day of sample collection as batches of samples are collected).
- Initial and routine calibration results (where appropriate).
- Duplicate analyses.
- Results of field blanks.

Each of these elements will be evaluated against the established limits for precision and accuracy (see Table 2). Data falling outside the established limits will be appropriately flagged, and an assessment will be made as to the effect on data quality.

4.2.3 Data Verification

The purpose of data verification is to ensure that conclusions can be correctly drawn from the data, or to identify those data points which may not be representative of the subject site. Data verification requires documentation of sample and data integrity and independent inspection of that documentation.

Field sampling personnel will be responsible for documenting sample collection, sample handling, and field analysis. Field personnel will document any events which may affect sample integrity and representation, such as potential mis-labeling or a delay in sample preservation. The STS Project Manager and/or field sampling personnel will be responsible for documenting sample shipment and transport. The courier or shipping agency will provide a Bill of Lading and collect signatures upon delivery, but the STS Project Manager will be responsible for collating this documentation and ensuring it provides adequate documentation of the sample's history.

Laboratory personnel will be responsible for documenting sample receipt and sample condition upon receipt. The laboratory personnel will document any events occurring in the laboratory which may affect sample integrity and representation, such as the potential for mis-identification at the laboratory or samples received too warm.

The STS QA Officer will verify that all sample results have been received, that quantitation limits have been met, and will evaluate the aforementioned documentation to make a determination as to the effects, if any, on data quality and data integrity. The results of this evaluation will be summarized in a brief technical memorandum.

4.3 Reconciliation with Data Quality Objectives

The field and laboratory data collected during this investigation will be used to evaluate the nature and degree of contamination at the subject site. Only data generated in association with QA results meeting the objectives described in Section 1.0 will be considered useable for decision making purposes. The data obtained will be both qualitatively and quantitatively assessed on a project-wide, matrix-specific, parameter-specific, and unit-specific basis. This assessment will be performed by STS personnel and the results presented and discussed in detail in the Final Site Investigation Report. STS will determine if the correct type, quality, and quantity of samples were taken to support environmental decision making for the project. Factors to be considered in this assessment of field and laboratory data will include, but are not limited to, the following:

- Were all samples appropriately collected and handled using the methodologies and SOPs described in the QAPP?
- Were all proposed analyses performed according to the SOPs provided in the QAPP?
- Were samples obtained from all proposed sampling locations and depths?
- Do any analytical results exhibit elevated reporting limits due to matrix interferences or contaminants present at high concentrations?
- Were any analytes not expected to be present at the facility, or a given unit, identified as target parameters?
- Were all field and laboratory data validated according to the validation evaluation protocols, including project-specific QC objectives proposed in the QAPP?
- Which data sets were found to be un-usable based on the data validation results?

- Which data sets were found to be usable for limited purposes based on the data validation results?
- What effect do qualifiers applied as a result of data validation have on the ability to implement the project decision rules?
- Has sufficient data of appropriate quality been generated to obtain site closure?
- Can valid conclusions be drawn for all matrices at each unit and/or area under investigation?
- Were the project-specific decision rules used as proposed during the actual investigation?
- For any cases where the proposed procedures and/or requirements have not been met, has the effect of these issues on the project objectives been evaluated?
- Based on the overall findings of the investigation and this assessment, were the original project objectives appropriately defined? If not, have revised project objectives been developed?

From the results of this data assessment, additional samples may be conducted to address new situations which are identified or to address previous inquiries, which require more detailed information in order to meet the data quality objectives outlined in Section 1.3.3. Sample data sets which are deemed not usable for decision-making purposes may be re-collected. In addition, additional project objectives may be identified from the sampling results. This assessment process is an interactive one; throughout the site investigation, project objectives may be altered to meet the needs of the project and to more efficiently use the available project time and funds.

The Data Quality Assessment (DQA) Process is described in "Guidance for the Data Quality Assessment Process: Practical Methods for Data Analysis," EPA QA/G-9, July 1996. EPQ QA/G-9 will be used to guide the data assessment on this project. The DQA Process will consist of five steps:

1. Review DQAs and Sampling Design.
2. Conduct Preliminary Data Review.
3. Select Statistical Test.
4. Verify Assumptions.
5. Draw Conclusions from the Data.

While the formal DQA process presented in this document may not be followed in its entirety, a systematic assessment of the data quality will be performed. This process will include a preliminary data review. Data will be presented in tables and figures to identify the trends, relationships (correlations), and anomalies.

4.3.1 Precision

Spiked samples are prepared by choosing a sample at random from each sample shipment received at the laboratory, dividing the sample into equal aliquots, and then spiking each of the aliquots with a known amount of analyte. The duplicate samples will then be included in the analytical sample set. The splitting of the sample allows the analyst to determine the precision of the preparation and analytical techniques associated with the duplicate sample. The Relative Percent Difference (RPD) between the spike and duplicate spike will be calculated and plotted. The RPD is calculated according to the following formula:

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

where:

S = First sample value (original or matrix spike value);

D = Second sample value (duplicate or matrix spike duplicate value).

4.3.2 Accuracy/Bias

In order to assure the accuracy of the analytical procedures, an environmental sample will be randomly selected from each sample shipment received at the laboratory and spiked with a known amount of the analyte or analytes to be evaluated. In general, a sample spike will be included in every set of 20 samples tested on each instrument. The spike sample will then be analyzed. The increase in concentration of the analyte observed in the spiked sample due to the addition of a known quantity of the analyte and compared to the reported value of the same analyte in the un-spiked sample determines the percent recovery. Daily control charts will be plotted for each commonly analyzed compound and kept on instrument-specific, matrix-specific, and analyte-specific bases.

The percent recovery for a spiked sample is calculated according to the following formula:

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

where:

A = The analyte concentration determined experimentally from the spiked sample;

B = The background level determined by a separate analysis of the un-spiked sample;

C = The amount of the spike added.

4.3.3 Sample Representation

In order to meet the needs of the data users, project data must meet the measurement performance criteria to sample representation specified in Section 1.3.3.

QC check and sample data will be reviewed to assess sample representation. If field duplicate precision checks indicate potential spatial variability, then this may trigger additional scoping meetings and subsequent re-sampling in order to collect data that are more representative of a non-homogeneous site.

The Data Assessment Report will discuss and compare overall representation for each matrix, parameter, and concentration level. Data Assessment Reports will describe the limitations on the use of project data when overall non-representative sampling has occurred or when non-representative sampling is limited to a specific sampling group, data set, matrix, analytical parameter, or concentration level. If data are not usable to adequately address environmental questions and/or support project decision making, then the Data Assessment Report will address how this problem will be resolved and discuss potential need for re-sampling.

4.3.4 Sensitivity and Quantitation Limits

In order to meet the needs of the data user, project data must meet the measurement performance criteria for sensitivity specified. Low point calibration standards should produce a signal at least ten times the background noise level and should be part of a linear calibration curve. Document the procedures for calculating MDLs and QLs.

Overall Sensitivity and Quantitation Limits: If Data Validation Reports indicate that sensitivity and/or QLs were not achieved, then the impact of that lack of sensitivity and/or higher QLs on data usability will be discussed in the Data Assessment Report.

The Data Assessment Report will discuss and compare overall sensitivity and QLs from multiple data sets collected for the project for each matrix, analytical parameter, and concentration level. The Data Assessment Report will describe the limitations on the use of the project data if project-required sensitivity and QLs were not achieved for all project data or when it is limited to a specific sampling or laboratory/analytical group, data set, matrix, analytical parameter, or concentration level.

When project-required QLs are not achieved and project data are not usable to adequately address environmental questions (i.e., determining if regulatory/technical action limits have been exceeded) and to support project decision making, then the Data Assessment Report will address how this problem will be resolved and discuss the potential need for re-sampling. In this case, the Data Assessment Report will clearly differentiate between usable and un-usable data for the users.

4.3.5 Completeness

(In order to meet the needs of the data users, project data will follow the measurement performance criteria for data completeness outlined in Section 1.5.3.)

Completeness is the ratio of the number of valid sample results to the total number of samples analyzed with the specific matrix and/or analysis. Following completion of the analytical testing, the percent completeness will be calculated by the following equation:

$$\text{Completeness} = \frac{\text{(number of valid measurements)}}{\text{(number of measurements planned)}} \times 100$$

Overall Completeness - The Data Assessment Report will discuss and compare overall completeness of multiple data sets collected for the project for each matrix, analytical parameter, and concentration level. The Data Assessment Report will describe the limitation on the use of the project data if project-required completeness was not achieved for the overall project or when it is limited to a specific sampling or laboratory/analytical group, data set, analytical parameter, or concentration level.

When project-required completeness is not achieved and sufficient data are not available to adequately address environmental questions and support project decision making, then the Data

Assessment Report will address how this problem will be resolved and discuss the potential need for additional re-sampling.

4.3.6 Comparability

In order to meet the needs of the data users, the measurement performance criteria for comparability outlined in Section 1.3.3 will be followed.

For long-term monitoring projects, data comparability is extremely important. Project data will be compared to previously generated data to determine the possibility of false positives and/or false negatives. Variations detected in the data may reflect a changing environment or indicate sampling and/or analytical error. Comparability criteria will be established to evaluate these data sets in order to identify outliers to trigger re-sampling as verified.

The Data Assessment Report will discuss and compare overall comparability between multiple data sets collected for the project for each matrix, analytical parameter, and concentration level. The Data Assessment Report will describe the limitation on the use of project data when project required data comparability is not achieved for the overall project or when it is limited to a specific sampling or laboratory/analytical group, data set, matrix, analytical parameter, or concentration level.

If screen/confirmatory comparability criteria are not met, then this will be documented in the Data Assessment Report and the effect on data usability will be discussed. If oversight split sampling comparability criteria are not met, then this will be documented in the Data Assessment Report and the effect on data usability will be discussed. If data are not usable to adequately address environmental questions and/or support project decision making, then the Data Assessment Report will address how this problem will be resolved and discuss potential need for re-sampling.

Finally, if long-term monitoring data is not comparable, the Data Assessment Report will address whether the data indicates a changing environment or the anomalies are a result of sampling and/or analytical error. If data is not usable to adequately address environmental questions and/or support project decision making, then the Data Assessment Report will address how this problem will be resolved and discuss potential need for re-sampling.

4.3.7 Data Limitations and Actions

Sources of sampling and analytical error will be identified and corrected as early as possible to the onset of sample collection activities. An ongoing data assessment process will be incorporated during the project, rather than just as a final step, to facilitate the early detection and correction of problems, ensuring that project quality is maintained.

5.0 REFERENCES

STS Consultants, Ltd., *Preliminary Assessment Report, Arsenic Spill Site, Kewaunee Marsh, Kewaunee County, Wisconsin,* July 1994. STS Project No. 20716XF

STS Consultants, Ltd., *Temporary Measures for Interim Action, Kewaunee Marsh Arsenic Site, CD Besadny Wildlife Area, Kewaunee County, Wisconsin,* November 1995. STS Project No. 20716XA

STS Consultants, Ltd., *Site Assessment and Remedial Actions Alternatives Report, Kewaunee Marsh Area, Kewaunee County, Wisconsin,* March 15, 2004. STS Project No. 4-27393A

Wisconsin Department of Natural Resources Bureau of Watershed Management, Water Quality Section, *Baseline Ecological Risk Assessment for the Arsenic Contaminated Wetland Associated with the CD Besadny Fish and Wildlife Area and the Kewaunee River,* April 2000.

Wisconsin Department of Natural Resources Bureau of Watershed Management, *Interim Results Summary, CD Besadny Fish and Wildlife Area, Kewaunee Marsh, Wisconsin,* August 2001.

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Tables

TABLE 1 - QA OBJECTIVES FOR FIELD MEASUREMENTS

	Parameter	Method Reference(1)	Precision(2)	Accuracy(3)	Field Completeness
	Water				
	Standing Water Levels	Solonist Water Level	±0.01 foot	0.005 foot	+90%
	Temperature	Indicator E-170 Mercury Thermometer or Electronic Temperature Probe	±0.5° Celsius (C)	1.0° C	+90%
Closed Flow-Thru Cell	Conductivity	E120.1, Electrometric	±15%	±15%	+90%
	pH	E150.1, Electrometric	±0.2 pH units	0.1 pH units	+90%
	Dissolved Oxygen	Colormetric Indigo Carimine Test Chemetrics K-7512	±0.5 ppm (1-6 ppm range) ±1.0 ppm (6-12 ppm range)	(5)	+90%
	Conductivity	AC electrode	±0.3%	±0.3%	+90%
	pH	Glass electrode	±0.1 pH	±0.1 pH	+90%
	Redox Potential	Platinum electrode	±15 mV	±15 mV	+90%
	Dissolved Oxygen	Diaphragm Gavanic	±0.1 mg/L	±0.2 mg/L	+90%

Notes:

(1) Methods: E = *Method for Chemical Analysis for Water and Wastes* (US EPA, 2983)

(2) Expressed as the acceptable deviation from the Scale.

(3) Expected based on equipment manufacturer specifications.

(4) Acceptable accuracy and precision based on the range of measured.

(5) This is a visual colormetric test. The repeatability of the color produced is much better than the human eye can determine.

ppm = parts per million

mg/L = milligrams per liter

Table 2

INORGANIC LIST WITH QUANTITATION LIMITS AND QA OBJECTIVES

Analyte	Method Reference	Detection Limits	Water matrix LOQ (µg/L)	% Recovery	% RPD Limits
Arsenic (As)	200.7/6010 200.8/6020	8.0 0.6	26.6 2.0	±5%	±10%
Sulfate	Ion Chromatograpy 300.0	1.0	3.33	±5%	±10%
Sodium	200.7/6010	500.	500.	±5%	±10%
Iron	200.7/6010	10.	10.	±5%	±10%
Nitrate	353.1 Ion Chromatograpy 300.0	100. 100.	333. 333.	±5%	±10%

µg/L = micrograms per liter

Table 3 - Tentative Project Schedule

Task	Completion Date
QAPP development and signoff.	November 2004
First round quarterly groundwater sampling including arsenic speciation sample collection and analyses.	November 2004
Surface water sampling and flow monitoring.	April 2004
Second round quarterly groundwater sampling.	February 2005
Third round quarterly groundwater sampling and annual sampling	April/May 2005
Fourth round quarterly groundwater sampling.	August 2005

Table 4 - Sample Program Summary

Sampling Point	Frequency	Parameter Analysis
MW02-3, 3i, 3d MW02-4, 4i, 4d MW02-5, 5i MW02-6, 6i MW02-7, 7i MW02-8 GW01-2 GW01-3 GW01-5 GW01-6 GW01-8 MW04-9 MW04-10 MW04-11 MW04-12 MW04-13	Quarterly	Arsenic (by GFAAS) Sulfate Sodium Iron Nitrate Field Dissolved Oxygen, pH, Conductivity, Temperature, Redox Potential
MW02-1, 1i, 1d MW02-2, 2i MW02-7d MW02-8i GW01-1 GW01-4 GW01-10	Annually	Arsenic (by GFAAS) Sulfate Sodium Iron Nitrate Field Dissolved Oxygen, pH, Conductivity, Temperature, Redox Potential
Surface Water SW1 SW2 SW3 SW4 SW5 SW6	Semi-Annual	Arsenic (by GFAAS) Sulfate Sodium Iron Nitrate Field Dissolved Oxygen, pH, Conductivity, Temperature, Redox Potential

Table 5

Sample Container, Preservation, and Holding Time Requirements

Matrix	Analysis	Container	Preservation	Holding Time
Water	Metals	One 1 liter polyethylene bottle	HNO_3 to pH <2	Six months
Water	Nitrate	One 1-liter amber glass bottle	H_2SO_4	48 hours
Water	Arsenic Speciation	One 1-liter polyethylene bottle	EDTA, HCL or by rapid freeze in dry ice/solvent slurry	

Table 6

Field Equipment Maintenance Schedule

Instruments	Maintenance Procedures/Schedule	Spare Parts in Stock
pH meter	1. Calibrate beginning at end of each day and as necessary during use. 2. Replace electrodes as needed.	1. pH buffers 2. Batteries 3. Spare electrodes
Conductivity Meter	1. Calibrate beginning at end of each day and as necessary during use. 2. Check redline and replace batteries if does not calibrate.	1. Batteries
Dissolved Oxygen Meter	1. Calibrate beginning and end of each day and as necessary during use. 2. Replace membrane as necessary.	1. Batteries 2. Membrane
Dissolved Oxygen Colormetric Analysis Chemetrics K-7512	1. Check expiration date and verify tube is in good condition.	1. Extra tubes.
Closed Flow-Thru Cell	1. Calibrate probes at end of each day and as necessary during use. 2. Replace probes as necessary.	1. Spare probe; membranes as necessary
Peristaltic Pump and Tubing	1. Check battery and replace when low. 2. Check tubing for wear and/or cleanliness.	1. Extra batteries 2. Tubing
Water Level Indicator	1. Check battery and replace when low.	1. Extra batteries
Temperature Probe	1. Check battery and replace when low.	1. Extra batteries

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Figure

Figure 1 - Sample Locations and Interim Cover Conditions (pocket)

LIMITS OF 1996 COVER WITH JANUARY 2002 SURFACE

RIVER WATER LEVEL 577.84
(11-13-02)

KEWAUNEE RIVER

WOODEN TRAIN TRESTLE

RIVER STAFF GAUGE
2.0 MARK ELEV. 579.34

sts-mp1
sts-mp2
sts-mp3
sts-mp4
sts-mp5
sts-mp6

WEIR

SCALE IN FEET
60' 0 60'

LEGEND

- EXISTING CHAIN LINK FENCE
- TREE LINE
- MARSH
- FORMER RAILROAD GRADE
- MW02-1d MONITORING WELL
- SPOT ELEVATION
- COVER PLACEMENT
- KM01-A SURVEY CONTROL POINT
- 1.2 COVER THICKNESS
- SW-1 2003 SURFACE WATER SAMPLE LOCATION
- SS02-16 2002 SURFACE SOIL SAMPLE LOCATION
- SB02-15 SHALLOW 5' SOIL BORING

NOTES:
HORIZONTAL DATUM IS WISCONSIN STATE PLANE (CENTRAL-4802) COORDINATE SYSTEM, NAD 83, U.S. FEET.

VERTICAL DATUMN IS IGLD 1985/NAVD 1988, U.S. FEET.

SURVEY SITE CONTROL POINTS SET BY WISCONSIN DEPARTMENT OF NATURAL RESOURCES

KM01-A NORTH 243595.30 EAST 2616993.19 ELEVATION 586.52

KM01-B NORTH 243768.19 EAST 2616689.40 ELEVATION 586.40

POINTS ARE 2' LONG , 1/2" REBARS WITH CAPS

FIGURE 1

DESIGNED BY PJK DATE 12-04
DRAWN BY DTB DATE 12-18-04
APPROVED BY PJK DATE 12-04
CADFILE X:\PROJECTS\427393EFIG1.DWG
XREF

C.D. BESADNY FISH AND WILDLIFE AREA
WISCONSIN DEPARTMENT OF NATURAL RESOURCES
KEWAUNEE, WISCONSIN

SAMPLE LOCATIONS AND INTERIM COVER CONDITIONS

STS Consultants Ltd.
Consulting Engineers
1035 Kepler Dr.
Green Bay, WI 54311
920.468.1978

STS PROJECT NO. 27393
STS PROJECT FILE
SCALE AS SHOWN
FIGURE NO. C-1

Wisconsin Department of Natural Resources
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Appendix A

Data Quality Objective Process

DATA QUALITY OBJECTIVE PROCESS

The Data Quality Objective (DQO) Process is a series of planning steps designed to ensure that the field activities, data collection operations, and resulting data meet the project objectives. The DQO process allows decision makers to determine the level of data quality needed for specific data collection activities. The DQO is based on the following seven-step process applied to the specific project:

1.0 State the Problem

The purpose of the project is to further characterize groundwater quality and define transport mechanisms of arsenic through groundwater and surface water pathways for evaluation of feasible remediation methods.

This project will facilitate the remedial action design for the Kewaunee Marsh arsenic site, a historic arsenic spill site in a state-owned wildlife area/wetland complex in Kewaunee County, Wisconsin. Approximately 13 acres of the CD Besadny Fish and Wildlife are within the Kewaunee Marsh study area contaminated by a suspected rail car derailment and resultant spill of sodium arsenite in 1943. Despite interim remedial measures in place since 1996, arsenic continues to migrate through the sediments both horizontally and vertically, and may be discharging to the adjacent Kewaunee River in both dissolved form (groundwater) and as suspended-sediment particulate via surface water runoff. Recent samples suggest that levels of arsenic reaching the river may be over 50 micrograms per liter, exceeding surface water quality standards. An effective and feasible remediation method to reduce the concentration of arsenic should be identified and implemented on the project site.

2.0 Identify the Decision

Specifically - What are the available options under consideration?

This project will facilitate a study to address the technical and financial feasibility of a combination of select remedy options identified as part of the previous investigative efforts. Results of past site investigations have concluded that wetland soils and sediment over approximately half the project site have elevated arsenic concentrations to depths of approximately 6 feet. The scope of work for this study will further determine the arsenic transport mechanisms of the study area, which in turn is expected to be used to evaluate remediation methods. Possible remediation methods include mechanical removal of impacted sediment, solidification/stabilization techniques which would convert the highly mobile arsenic to a more stable metal hydroxide, or use of a permeable reactive barrier system that would utilize the existing hydraulic gradient at the site to passively remove arsenic from surface and groundwater through a barrier catalyst.

To further define conditions for evaluation of possible remediation, the study must find answers to the following questions:

- At which depth is the arsenic movement the greatest?
- Is the arsenic changing form as it moves through the soil?

- Does surface water contribute significantly to the transport of arsenic on the site?
- How does the varying oxidation-reduction conditions across the site influence arsenic transport?

3.0 Identify Inputs to the Decision

What information is needed to make informed, defensible decisions?

Groundwater and surface water samples must be collected and analyzed from the impacted area to characterize groundwater and surface water quality. Other ionic constituents will influence arsenic speciation, solution/dissolution, adsorption, and subsequent transport.

4.0 Defining the Boundaries of the Study

What are the geographical extent and time and budget constraints for the project?

The extent of elevated arsenic concentrations on the study site and project boundaries have been determined through previous site investigative work. The impacted area is approximately 13 acres in size, extending from an abandoned railroad grade east to the Kewaunee River. A perimeter fence has been constructed around the area of elevated arsenic concentrations, and an interim barrier was constructed over the area of highest concentration. This work will focus on groundwater and surface water transport mechanisms of the arsenic over the study site, including the fenced area, and the area between the fenced portion of the site and the Kewaunee River. Limited financial resources are available and sampling should be completed within a reasonable period of time.

Funding for the investigation is limited. To economize, arsenic speciation analysis is being completed by the Wisconsin State Laboratory of Hygiene rather than a private laboratory

5.0 Developing a Decision Rule

Formulate "if...then" statements that relate the data to the decision they support.

If weather conditions do not allow use of a low flow sampling cell to complete field analysis, sampling will be postponed until weather conditions allow, or other methods of field analysis will be used, specifically sampling by dedicated bailer and down-well probes.

6.0 Specifying Limits on Decision Error

Estimate how much uncertainty will be tolerated in the site decisions.

Field analytical accuracy will be dependent on equipment accuracy standards. Equipment will be calibrated before use as specified in the SOP for each analysis.

7.0 Optimizing Design

Identify the most cost-effective means to gather the data needed. If obstacles exist, reassess all the steps of the DQO process to refine decisions and goals until a workable roadmap or decision tree is produced.

The study will collect data from existing groundwater well installations and previously established surface water monitoring points. The existing wells on the project site adequately define the impact area. The existing wells and sampling points are located upgradient, sidegradient, downgradient, and within the arsenic impact area. Wells located upgradient and sidegradient to groundwater flow direction will only be sampled annually, as arsenic concentrations are expected to change very little away from the direct path of migration. Other wells in the study area will be sampled quarterly. Use of the network of existing wells will limit the cost of collecting data by eliminating the need of additional wells. The only obstacle to gathering of data from the wells are seasonal conditions which could potentially limit site access (snow depth or standing water), prolonged cold temperatures could freeze the water in the wells, or cold temperatures may cause field analytical equipment to malfunction, which may cause inaccuracies in field measurements. Various methods of access, such as snowmobile or snowshoes, can be employed to access the site in the winter. Seasonal weather conditions are uncontrollable, with the only option to plan sampling events accordingly around seasonal conditions.

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Appendix B

Standard Operating Procedures

Arsenic Speciation Standard Operating Procedure

Monitoring well samples will be collected using low-flow sampling techniques to minimize mobilizing non-representative particulates into the groundwater samples. All materials that come in contact with the groundwater or surface water will be rigorously cleaned with high purity acids and rinsed with ASTM Type I water prior to use. The groundwater will be pumped through an in-line Meissner 0.4 um Alpha polypropylene capsule filter (or equivalent filter) and directly into acid-cleaned polyethylene or Teflon sample bottles to minimize aeration and potential change in the Arsenic redox species. Wells are carefully purged prior to sample collection, while continually monitoring key state variables (pH, redox, specific conductance, and temperature). Sample bottles will be filled sequentially and promptly preserved to stabilize the Arsenic (III/V) redox species. Arsenic samples destined for speciation will be preserved with EDTA, HCl, or by rapid freezing in dry-ice/solvent slurry (*McCleskey, et al., 2004*). Subsequent bottles will be preserved with nitric acid to a pH of <2 for total recoverable arsenic, iron, and sodium and with sulfuric acid for nitrate+nitrite nitrogen. Samples for sulfide and DOC are collected directly into amber glass bottles. The sulfide is pre-spiked with SAOB buffer for subsequent potentiometer measurement (ion-selective electrode). All sample bottles, filters, and sampling equipment will be cleaned and prepared in the Trace Metals Clean Lab (TMCL) located at the Wisconsin State Laboratory of Hygiene (WSLH) in Madison, Wisconsin. Each item will be placed in two plastic bags before leaving the TMCL and being transported to the field.

Surface water samples will be collected using "clean" sampling techniques as described in the Wisconsin Department of Natural Resources Field Procedures Manual and (*Shafer, et al., 1991 and Hurely, et al., 1996*) http://intranet.dnr.state.wi.us/int/es/science/ls/fpm/101-2.htm. Samples will be filtered and processed in the same manner as the monitoring well samples. Unfiltered samples will also be collected.

Arsenic speciation will be determined using high-performance liquid chromatography (HPLC) coupled with an inductively coupled plasma mass spectrometer (ICP-MS) (*Zheng, et al., 2003, and Le, et al., 2004*). Both inorganic and organic arsenic species will be reported for each sample. Total dissolved arsenic analyses will be determined using stabilized platform graphite furnace atomic absorption (Method 3113B) or by ICP-MS (Method 1638). Iron and sodium analyses will be determined by ICP-OES (EPA Method 200.7). Where available, certified reference material will be analyzed with each batch of samples. National Laboratory Accreditation Program (NELAP) guidelines will be followed, where applicable, for processing and analysis of all samples. Quality policies, practices, and principles are described in the WSLH Quality Assurance Manual.

State of Wisconsin will be submitting an addendum to this SOP, expected to be completed by January 2005.

Standard Operating Procedures for Groundwater Sampling

At least one week is recommended by the WDNR between well development and the first groundwater sampling event. The following section provides details relating to groundwater sampling.

Purging

Prior to collection of groundwater samples, the water level is again measured and each well is purged. The well purging procedures described herein are in accordance with the WDNR's *Groundwater Sampling Desk Reference*. If possible, four well-volumes of water are removed from the well using either a disposable bailer or low-flow sampling pump. If the well purges dry, the stagnant water is removed from the well and water is allowed to recharge into the well. Time permitting, the well is bailed or pumped dry again and allowed to recharge prior to collection of samples.

Typically, wells are purged using a Teflon© bailer or a disposable polyethylene bailer. In some instances, when it is necessary to remove a large volume of water from the well, a pump is used to purge the well. In these instances, a small submersible pump is used to purge the well. The pump and the hosing are decontaminated prior to inserting into the well.

Well Sampling

Typically, wells are sampled using either a low-flow sampling pump or a disposable polyethylene bailer. If a bailer is used in order to minimize disturbance of the water in the well, the bailer is slowly lowered to the water table using a rope tied to the top of the bailer. The bailer is allowed to fill from the bottom. Once the bailer is filled, it is gently brought to the surface and emptied into sample containers.

Duplicate samples are collected from each site at a minimum of 10% of the total number of samples collected. This procedure complies with WDNR quality assurance/quality control requirements.

Each cooler is sent to the laboratory with a trip blank. The trip blank is prepared by the laboratory by filing a volatile organic compound(s) (VOC) vial with distilled water and sealing the bottle. The bottle remains sealed and stays with the sample bottles throughout shipment from the laboratory, until it reaches the laboratory again. The water sample contained in the trip blank is analyzed by the laboratory to verify that the samples were not affected by contaminants during transportation.

VOC Sampling

A VOC sampling port is inserted into the bottom of the bailer to allow for regulation of water flow from the bailer. This allows for minimal sample disturbance.

The water is slowly discharged directly into the laboratory provided 40-ml VOC vials containing hydrochloric acid (HCl) preservative. The bottle is filled to a positive meniscus and covered with a cap fitted with a Teflon© septum. The bottle is inverted and gently tapped to verify that air bubbles are not present in the sample. Each bottle is labeled, typically with a label provided by the laboratory, with the well number, sample number, date, sampler's initials, project number, and preservatives added. After labeling, the samples are placed in a cooler on ice with the Chain of Custody forms for shipment to the analytical laboratory.

Semi-Volatile, Pesticide/Polychlorinated Biphenyls Sampling

The groundwater sampling for semi-volatiles will be completed using a low-flow purging and sampling protocol. The wells will be purged using a peristaltic pump approximately 100 milliliters per minute while monitoring the purge water for turbidity. Purging will be discontinued and the groundwater samples collected when the purge water turbidity stabilizes. All semi-volatile and pesticide/polychlorinated biphenyls (PCBs) samples will be collected using the peristaltic pump.

Metals Sampling

The sample portion used for metals analysis will also be collected using the low-flow sampling technique, as described above. After the semi-volatile, pesticide/PCB sample portion is collected (as described above) an in-line filter is connected to the sample discharge tubing. Filtering of the metals portion of the sample is required by the WDNR. The filter is a disposable 0.45-micron filter assembly connected directly to the discharge tubing of the peristaltic pump. The filtered water sample is discharged directly to the laboratory-supplied sample container, which contains nitric acid as a preservative. The bottle is filled, capped, and then inverted several times to mix the preservative into the sample. The samples are placed in a cooler on ice for shipment to the appropriate laboratory.

In-Field Testing

Typically, several in-field tests are conducted prior to completion of sampling at each well location. These tests include testing the conductivity, pH, and temperature of each sample after it is collected. The testing for pH, conductivity, and temperature are usually conducted using one instrument that records all three measurements. Various brands of instruments are available and used for conducting this testing. Water color, odor, and turbidity are also recorded by the technician in the field for each sample.

The water sample to be collected for in-field testing is collected at the time of well sampling. The sample is collected after the samples to be laboratory tested are collected and placed in coolers. The field-tested sample is collected using the same bailer used to collect the samples for analytical testing. The water is discharged from the bailer into an 8-ounce clear glass container. The instrument probe is inserted into the water sample and slowly swirled in the water until the instrument equilibrates. The measurements are then recorded in a field book. The visual observations noted at this time are also recorded in the field book.

Standard Operating Procedures for Decontamination

Drilling and Soil Sampling

To avoid cross-contamination between borings, the drilling equipment (i.e., augers and down-hole equipment) is decontaminated using a high pressure hot-water washer after each boring. The split-spoon sampler and stainless steel spoon/spatula are decontaminated using a wash of Alconox© soap and clean water, followed by a rinse with clean water. Equipment is scrubbed with a brush during each step of the decontamination process to remove soil particles which may adhere to equipment.

Groundwater Sampling

Typically, disposable bailers are used during well sampling. A new bailer is used to sample each well; therefore, there is no need to decontaminate down-hole equipment between locations. Similarly, if low-flow sampling is conducted, a new section of tubing will be used for each well sampled. The in-field testing equipment (pH, conductivity meter, temperature meter, and m-scope) are decontaminated between samples using a double rinse of distilled water. The water is containerized with the decontamination water generated during the advancement of the boring/well or purge water.

If disposable bailers are not used at the site, the Teflon© bailer is decontaminated using a wash of Alconox© soap and distilled water, followed by a double rinse using distilled water. The bailers are scrubbed with brushes during the washing process, and then during the first rinse, to remove sediment or other particles which may adhere to the bailer.

New rope and gloves are used at each well location; therefore, no decontamination of the equipment is necessary. If sample filters are used (i.e., for metals analysis), a new disposable filter and new tubing are used for each sample.

PURGING AND SAMPLING PROCEDURES

The **goal of purging** is either to remove stagnant water from the well or prevent stagnant water from entering samples as you are collecting them. Stagnant water does not represent groundwater.

The **goal of sampling** is to collect unaltered samples that represent the physical and chemical composition of groundwater.

- Purge and sample wells in order of least-to-most contaminated. (This is not necessary if you use dedicated or disposable equipment.) If you do not know this order, sample the upgradient wells first, then the furthest downgradient or sidegradient wells, and finally the wells closest to, but downgradient, of the most contaminated area.

- Wait at least one week before sampling a newly constructed and developed well; waiting a month or more may be appropriated for wells constructed in silt or clay.

- When using a bailer, purge and sample **slowly and carefully**. Use a bottom emptying device to decant samples from the bailer.

- Do **not** use cotton or cloth rope or line. Use stainless steel cord, Teflon® coated cord, nylon, or equivalent cord that can be decontaminated between each well or use disposable rope or line.

- Decontaminate all equipment and accessories between each use in each well. Store and transport all equipment in clean containers.

- Place a clean plastic sheet or other protective covering around the base of the well to prevent the equipment form contacting the ground. If you do not use a protective covering, ensure that your equipment does not touch the ground or a contaminated surface.

Wells that do NOT Purge Dry

This section applies to wells that take less approximately than one hour for their water levels to recover, or nearly so, after they have been purged.

The following purging and sampling procedures are recommended for wells that do not purge dry. The first procedures (A) consistently yields the **highest level of data quality**. The last procedure (C) may yield a **lower level of data quality**.

A. Low-flow Purging <1 liter/minute (L/min) (0.26 gallons per minute [gpm]). Low flow Sampling <300 milliliters per minute (ml/min) (0.3 L/min or 0.1 gpm) and Monitoring Indicator Parameters for Stability in a Closed Flow-Through Cell.

1. SLOWLY lower the pump to the **middle** of the well's screened area. (A dedicated system is recommended.) Securely fasten the power cable and sample tubing at the top of the well. Connect the power source, controller box, gas source, etc. to the pumping equipment.

2. Connect the sample tubing to the water entry point of the closed flow-through cell.

   Closed Flow-Through Cell

   Air pockets may exist in the upper neck of each porthole that has a probe inserted into it. This is not a problem. Just make sure the probe's sensors are completely submerged in water during use.

Avoid exposing the flow-through cell to extreme heat and sun in the summer and freezing temperatures in the winter.

3. Set up and calibrate all indicator parameter instruments and place each probe into its respective port of the closed flow-through cell.

4. Set the pump controller to the desired purging rate (i.e., < 1 L/min). Do not use a valve to reduce the flow from a pump; valves can cause an "orifice" effect that can cause sample agitation and alteration.

5. Record the "purging time start" and start purging the well at a rate of 1 L/min or less. During purging, the water level in the well should not decrease significantly and should stabilize after purging for a few minutes. If the water level continues to decline while purging, decrease the purging rate if possible. Record the "purging flow rate" as an average. Use a graduated beaker, cylinder, calibrated bucket, or other device to measure the flow rate while purging and sampling.

6a. Purge the well until you have taken at least three consecutive readings, spaced approximately 2 minutes or approximately 0.5 well-volumes or more apart, which are within the following ranges for the following indicator parameters:

Dissolved Oxygen (DO)	± 0.2 milligram per liter (mg/L)
Specific Conductance	± 5.0 µmhos/centimeter (µmhos/cm) for values > 1,000 µmhos/cm
pH	± 0.1 pH units
Temperature	± 0.1 degrees C
Turbidity	< 5 NTUs (**Required** if metal samples will not be filtered. **Recommended** if sorptive compounds or elements are collected. **Optional**, but recommended, if other compounds or elements are collected.)
Eh (optional)	± 30 mv

Stable dissolved oxygen, specific conductance, and turbidity readings are considered the most reliable parameters for indicating that stagnant water has been replaced by formation water. You may adjust the ± ranges and which indicator parameters you use to indicate that stagnant water has been replaced by formation water to reflect site-specific data, geochemistry, and hydrogeologic conditions.

Turbidity stabilization and NTU readings below 5 are required if you will not be filtering metal samples. In addition, monitor turbidity stabilization when collecting sorptive, hydrophobic, or high octanol water partition coefficient (Kow) compounds or elements.

OR

6b. Purge the well until the readings for indicator parameters listed above (or well-specific indicator parameters) vary within ±10% over three or more consecutive readings, spaced approximately two minutes or approximately0.5 well-volumes or more apart.

7. Record the final three stable readings for each indicator parameter on the "Well-Specific Field Sheet - Monitoring Wells" (Appendix A) or use your own customized data sheet. Record indicator parameter data measured before stabilization on graph paper or customize your own data sheet.

8. Record the "volume purged," "purging time stop," purged dry (Y/N)," and any problems purging.

9. Collect samples as described under Section 2.5. Record "sample flow rate" as an average, "time sample collected," and any other pertinent information related to the sampling event.

B. Purging FOUR Well-Volumes with a Standard Pump and Sampling with a Pump or Grab Sampler.

1. SLOWLY lower the pump to the **middle** of the screened area of the well. Securely fasten the power cable and sample tubing. Connect the power source, controller box, gas source, etc. to the pumping equipment.

2. Use **Equation 1** or **Table 1** (see the following this section) to calculate the number of gallons to remove four well-volumes from the well. Record this data as "four well-volumes."

3a. Using a **pump** *to purge* and *sample* the well: Record the "purging time start" and start purging the well. Minimize the well drawdown; it should stabilize before sampling. If the water level continues to decline during purging, try using a lower purging rate. Use a graduated beaker, cylinder, calibrated bucket, or other device to calculate the flow rate while purging and sampling.

3b. Using a **pump** to *purge* the well and then using a **grab sampler** to *sample* the well: Record the "purging time start" and start purging the well with the pump's inlet at the top of the water column. As you are purging, **slowly lower the pump**, so that after four well-volumes are purged, the pump's inlet is near the bottom of the well (within approximately 1 foot). Important note: Before collecting samples with a grab sampler, you must lower the pump while purging the well, thus removing any stagnant water before collecting samples.

4. Record "purging flow rate" as an average, "volume purged," "purging time stop," "purged dry (Y/N), and any problems purging.

5a. If you use a **pump** to collect samples, the sampling flow rate should be as low as possible and preferably less than the purging flow rate.

5b. If you use a **grab sampler**, try not to disturb the samples. If you use a bailer, use a bottom emptying device to decant your samples.

6. Collect samples as described under Section 2.5. Record "sample flow rate" as an average, "time sample collected," and any other pertinent information related to the sampling event.

C. Purging FOUR Well-Volumes with a Bailer and then Sampling with a Bailer or Other Grab Sampler.

1. Use **Equation 1** or **Table 1** (see the following section) to calculate the number of gallons needed for removing **four** well-volumes. Record this data as "four well-volumes."

2. Record the "purging time start." Lower and raise a decontaminated bailer in and out of the water column **very slowly** and purge four well-volumes.

TIP: To hasten purging and sampling with a bailer, tie an overhand knot, string, or other easily removable market to the rope or cable just of the well's depth to water. You can then rapidly lower the bailer into the well to just above the water column, then **gently and slowly** lower it into, then out of, the water column.

3. Use a calibrated bucket or other device to keep track or the volume of water you remove. Purge four well-volumes.

4. Record "volume purged," "purging time stop," "purged dry (Y/N)," and any problems purging.

5. Sample the well by **slowly and gently** lowering the bailer until it is submerged and in the middle of the well screen. Do not allow the bailer to contact the bottom of the well. **Very slowly and carefully** raise the bailer out of the water column and to the surface. Do not bang it against the side of the well (typical of the "helicoptering" technique).

6. Collect samples as described under Section 2.5. Use a bottom emptying device to decant samples from a bailer. Record "time sample collected" and any other pertinent information related to the sampling event.

LOW-FLOW SAMPLING

Wells that Purge Dry

This section applies to wells that take ~ 1 or more hours to recover, or nearly so, after they have been purged dry, or nearly so.

A. Low-flow Purging and Sampling in a WATER TABLE WELL (water level intersects the well screen)

1. Slowly lower the pump to the ***lower portion*** of the screened area of the well but without setting it at the very bottom of the well. Secure the power cable and sample tubing. A dedicated system is recommended over a portable system.

2. Record the "purging time start" and start purging the well at < 300 ml/min or <0.1 gpm. Purge until drawdown reaches the top of the pump or until the pump runs dry, then shut the pump off immediately! (***Caution!*** Some pumps can be damaged by running them dry - follow the manufacturer's instructions.) Record the "purging flow rate" as an average.

   Note: A pressure transducer or electric water level indicator can assist in determining when drawdown reaches the top of the pump. If you use an electric water level indicator, lower the probe tip to the well pump and turn the instrument on before pumping. Start pumping, then shut off the pump when the water level indicator shuts off.

3. Allow the well to recover, or nearly so. If time permits, purge the well a second time and allow the water to recover again before sampling. To save time, purge a well the first time, move on to the next well and purge it, then come back to the first well to purge it again before sampling. (With portable equipment - decontaminate first.) Record "volume purged," "purging time stop," "purged dry (Y)," and any problems purging.

4. Collect samples as described under Section 2.5. (Collect samples within 24 hours of purging, if possible.) Record "sampling flow rate" as an average, "time sample collected," and any other pertinent information related to the sampling event.

B. Low-flow Purging and Sampling in a PIEZOMETER (water level is above the top of well screen)

1. SLOWLY lower the pump to the ***lower portion*** of the screened area of the well but do not set the pump on the very bottom of the well. Secure the power cable and sample tubing at the top of the well. A dedicated system is recommended over a portable system.

2. Record the "purging time start" and start purging the well at <300 ml/min or <0.1 gpm. Purge the well until the water level is just below the top of the well screen. (Use a pressure transducer, water level indicator or similar method.) Shut off the

pump and record the "purging flow rate" as an average, "volume purged," "purging time (stop)," "purged dry (Y)," and any problems purging.

3. Allow the well to recover, or nearly so, then begin collecting samples as described under Section 2.5. (Collect samples within 24 hours of purging, if possible.) If the water level in the well reaches the top of the screen before all samples are collected, shut off the pump, allow the well to recover again, then resume collecting the rest of the samples. Record "sample flow rate" as an average, "time samples collected," and any other pertinent information related to the sampling event.

C. Purging and Sampling with a BAILER, or other grab sampler, in a Water Table Well or Piezometer

1. Record "purging time start" and bail the well dry, or nearly so. Take extra care to purge the well very slowly and very gently. Do not allow the bailer to contact the bottom of the well.

2. Allow the well to recover, or nearly so. If time permits, purge the well dry, or nearly so, a second time. Record "volume purged," "purging time stop," "well purged dry (Y)," and any problems purging.

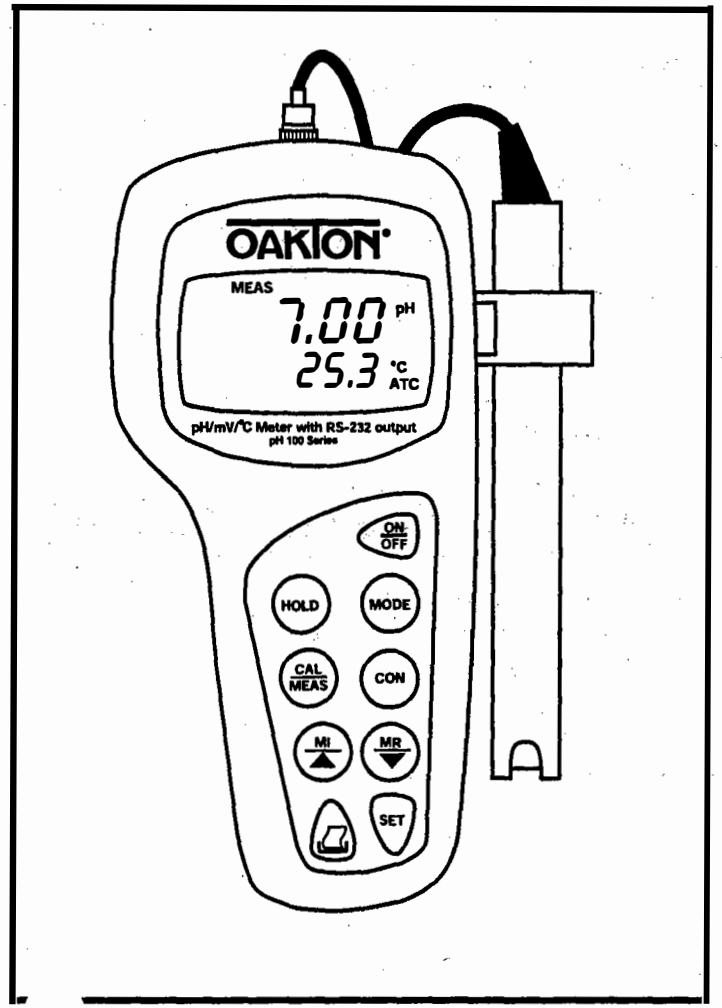
3. Collect samples as described under Section 2.5, within 24 hours of well recovery, if possible. Use a bottom-emptying device to decant samples from the bailer. Record "time sample collected" and any other pertinent information related to the sampling event.

1. Introduction

Thank you for selecting an OAKTON meter. The OAKTON pH/mV/°C portable meter is a microprocessor-based instrument that measures pH, mV, and temperature. This meter has many user-friendly features—all of which are completely accessible through the water-resistant membrane keypad.

Your meter includes two electrode holders, batteries and OAKTON PC Datalog Assist Software on a 3½" disk.

Please read this manual thoroughly before operating your meter.



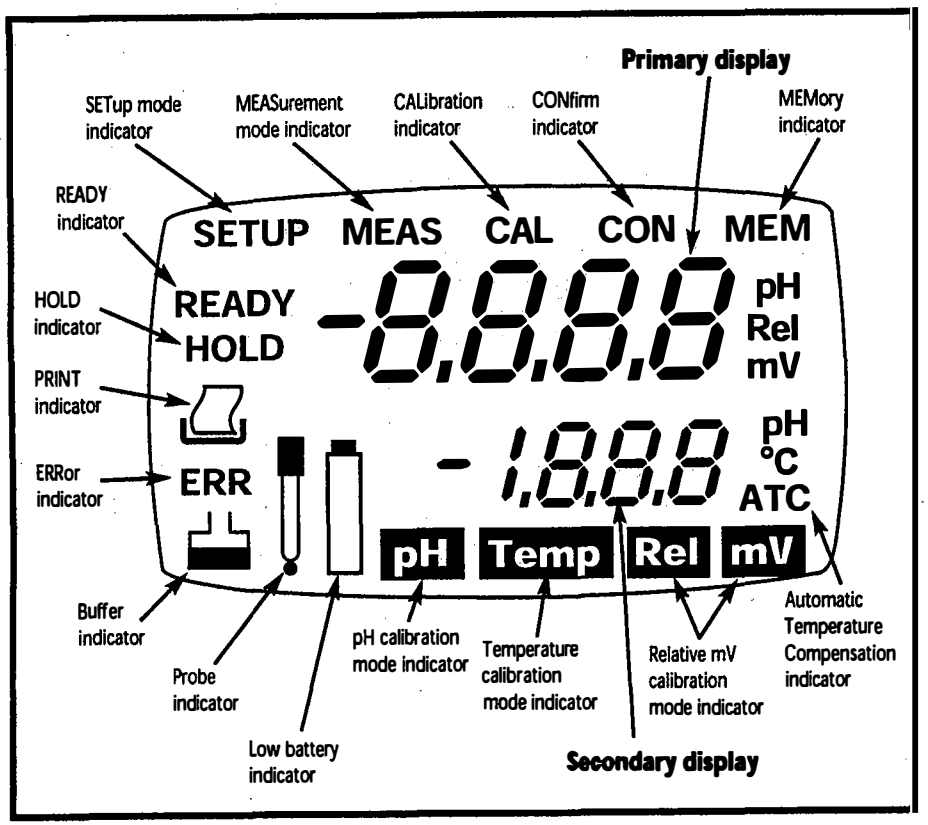
2. Display and Keypad Functions

2.1 Display

The LCD has a primary and secondary display.

- The primary display shows the measured pH or Relative mV reading.
- The secondary display shows the temperature of the reading in °C.

The display also shows error messages, keypad functions and program functions.



2.2 Keypad

The large membrane keypad makes the instrument easy to use. Each button, when pressed, has a corresponding graphic indicator on the LCD.

ON/OFF.....Powers and shuts off the meter.

HOLD .....Freezes the measured reading. To activate, press **HOLD** while in measurement mode. To release, press **HOLD** again

MODE .....Selects the measurement parameter (pH, mV or relative mV). Press MODE to toggle between pH, mV and relative mV mode.

CAL/MEAS.....Toggles user between Calibration and Measurement mode.

- If you were in pH Measurement mode, press **CAL/MEAS** to enter pH Calibration mode.
- If you were in mV Measurement mode, press **CAL/MEAS** to enter mV Calibration mode.

NOTE: Manual Temperature compensation is accessible from temperature mode; see page 17 for instructions.

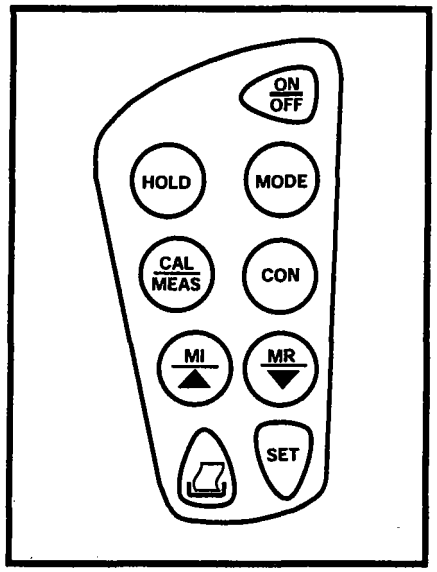
CON.....Press to confirm your calibration values in Calibration mode.

MI/MR .....▲/▼ scrolls values up and down in Calibration mode.

MI/MR work in the measurement mode. MI (memory input) stores the measured value into memory. MR (memory recall) recalls the sets of values stored in the memory.

(PRINT).....Sends measurement to either the printer or computer.

SET.....Enters the SETUP mode. Lets you customize the preferences and defaults.

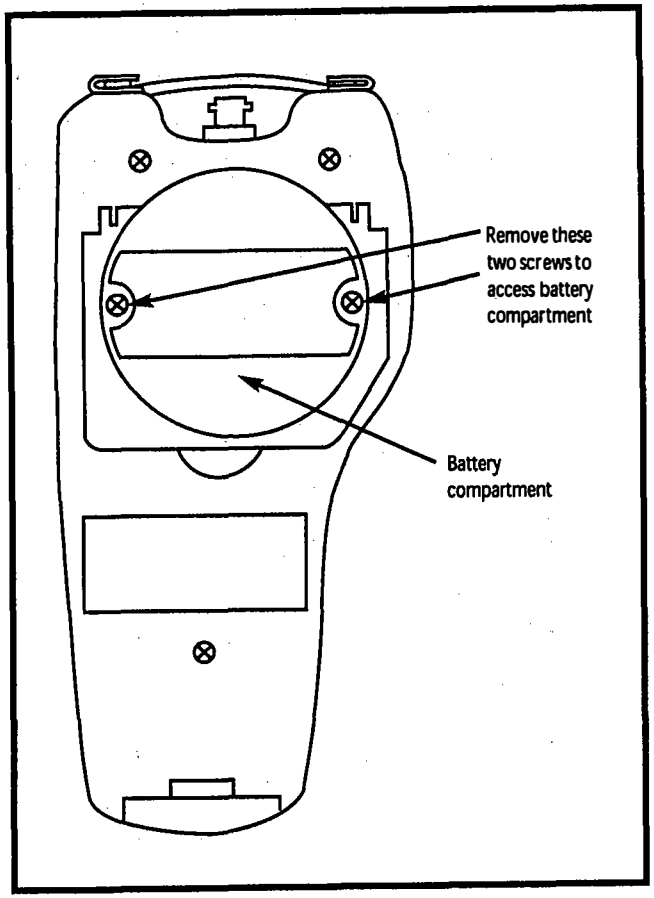


3. Preparation

3.1 Inserting the Batteries

Four AAA batteries are included with your meter.

1. Use a Phillips screwdriver to remove the two screws holding the battery cover. See figure below.
2. Lift meter stand to expose battery cover. Remove battery cover.
3. Insert batteries. Follow the diagram inside the cover for correct polarity.
4. Replace the battery cover into its original position using the two screws removed earlier.



3.2 Connecting the Electrode and Temperature Probe

The OAKTON pH/mV meter uses any standard pH, ORP, or ISE electrode with a BNC connector and separate temperature probe or an "All-in-One" combination pH/temperature probe.

NOTE: Keep connector dry and clean. Do not touch connector with soiled hands.

To connect the pH, ORP or ISE electrode:

1. Slide the BNC connector of the probe over the BNC connector socket on the meter. Make sure the slots of the connector are in line with the posts of the socket. Rotate and push the connector clockwise until it locks

See figure **A**

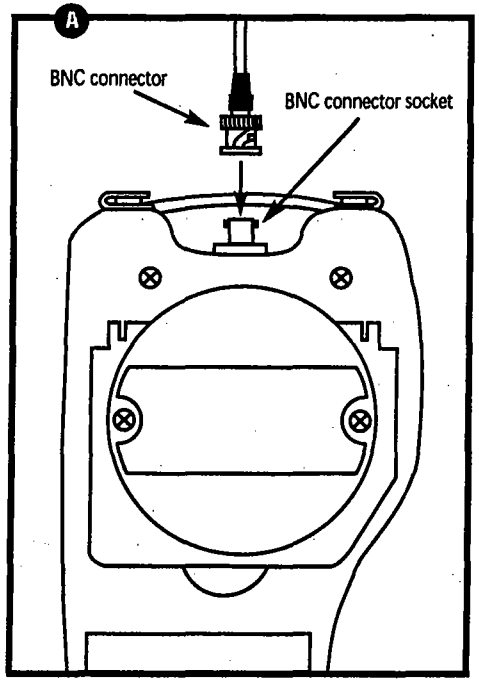
2. To remove probe, push and rotate the connector counter clockwise . While holding onto the metal part of the connector, pull probe away from the meter.

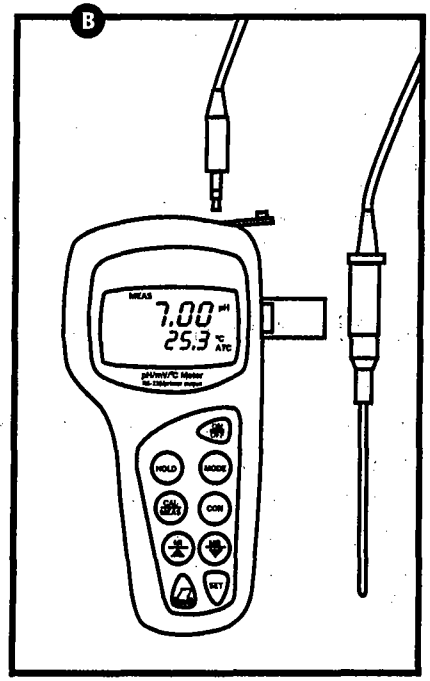
CAUTION: Do not pull on the probe cord or the probe wires might disconnect.

To connect the temperature probe:

1. Plug temperature in the phone jack as shown in figure **B**

Note: You should calibrate your temperature probe when you replace the probe and when using an OAKTON® "All-in-One" combination pH and temperature probe. See Temperature Calibration on pages 14-16 for instructions.



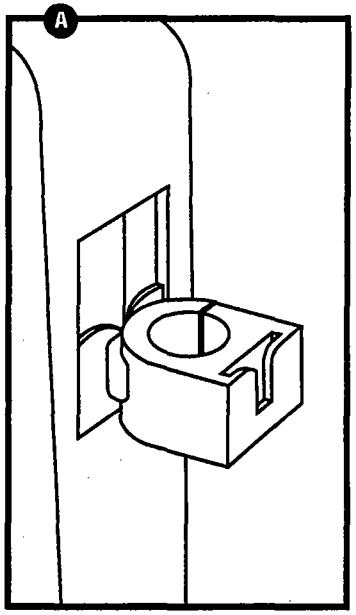


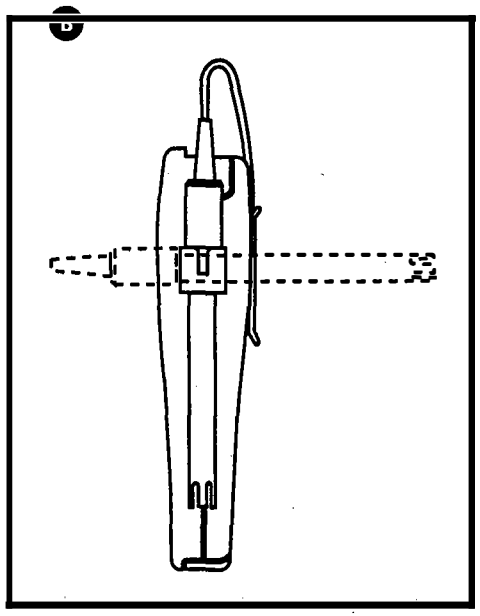
3.3 Attaching the Electrode Holder to the Meter

1. Place the electrode holder with the flange facing the slot on the meter. See Figure A
2. Gently slide the flange of the holder into the slot on the meter. Make sure the holder is secured properly into the slot.

You can attach the electrode holder in different positions. See figure B

This flexibility facilitates one-hand operation.





3.1 Inserting the Electrode into the Electrode Holder

Two electrode holders are included with your meter.

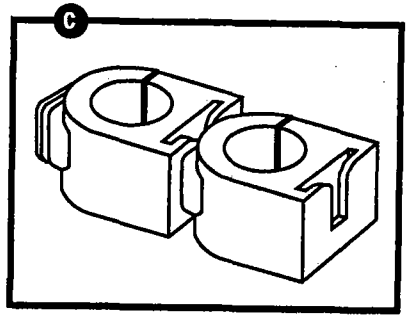
NOTE: Do not use excessive force when inserting electrodes into the holders.

1. Insert the pH electrode into the opening of the holder until the top housing of the electrode touches the top of the holder.

NOTE: The holder is designed for probes 12 mm in diameter. Electrodes larger than 12 min may not fit in the holder. Forcing the electrode into the opening may damage the holder or your electrode.

5.3 Attaching Two Electrode Holders

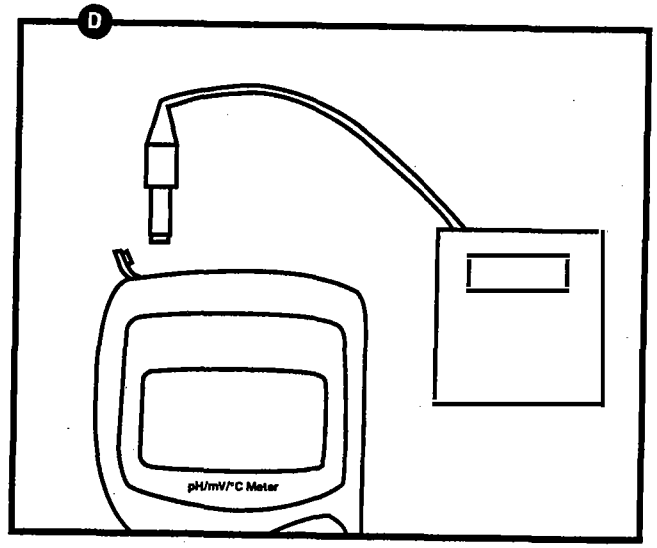
1. Align the flange of the second electrode holder with the slot of the first holder. See Figure C
2. Slide the flange of the second holder into the slot of the first holder until the tops of the holders are aligned and secure.



3.3 Connecting the AC Adapter

The AC adapter is not included with your meter; order separately on page 42.

1. Insert the AC jack as shown in figure D below.
2. Switch off the meter before plugging the adapter into the power source. This safety precaution protects the software in your meter.
3. Press the ON/OFF button to switch meter on.



4. Calibration

4.1 Important Information on Meter Calibration

When you recalibrate your meter, old pH, Rel mV and mV are replaced on a point by point basis. For example, if you previously calibrated your meter at pH 4.01, 7.00, and 10.01, and you recalibrate at pH 7.00, the meter retains the old calibration data at pH 4.01 and pH 10.01. To view current calibration points see Program 2 in the SETUP section, page 24.

To completely recalibrate your meter, or when you use a replacement probe, it is best to set the meter to its factory defaults and recalibrate the meter at all points. To reset the meter to its factory defaults see the SETUP section Program 1 (P 1.1), page 23.

For information on how to calibrate your meter:
- See section 4.3 on pages 11-12 for pH calibration
- See section 4.4 on page 13 for Relative mV calibration
- See section 4.5 on pages 14-16 for Temperature Calibration of replacement temperature probes or replacement "All-in-One" electrodes

4.2 Preparing the Meter for Calibration

Before starting calibration, make sure you are in the correct measurement mode. When you switch on the meter, the meter starts up in the units you shut it off in (either pH, Rel mV, or mV). For example, if you shut the meter off in "Rel mV" units, the meter will read "Rel mV" units when you switch the meter on.

The pH/mV meter uses any standard pH, ORP, or ISE electrode with a BNC connector and separate temperature probe or an ALL IN ONE combination pH/temperature probe.

Be sure to remove the protective electrode storage bottle or rubber cap of the probe before calibration or measurement. If the electrode has been stored dry, wet the probe in tap water for 10 minutes before calibrating or taking readings to saturate the pH electrode surface and minimize drift.

Wash your probe in deionized water after use, and store in pH 4.0 or 7.0 electrode storage solution.

Do not reuse buffer solutions after calibration. Contaminants in the solution can affect the calibration, and eventually the accuracy of the measurements.

4.3 pH calibration

This instrument is capable of up to 5-point pH calibration to ensure accuracy across the entire pH range of the meter. You can perform 1-, 2-, 3-, 4- or 5-point calibration with standard pH buffers 1.68, 4.01, 7.00, 10.01, and 12.45.

We recommend that you perform at least a 2-point calibration using standard buffers that bracket (one above and one below) the expected sample range. You can also perform a 1-point calibration, but make sure that the buffer value is close to the sample value you are measuring.

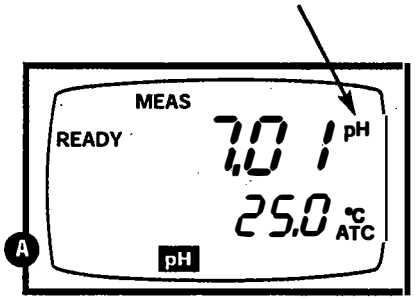
This meter features five preprogrammed pH buffers (pH 1.68, 4.01, 7.00, 10.01, and 12.45). The meter automatically recognizes and calibrates to these standard buffer values, which makes pH calibration faster and easier. See pages 44-45 for information on our high-quality OAKTON pH buffers.

All new calibration data will over-ride existing stored calibration data.

Calibrating for pH:

1. If necessary, press the MODE key to select pH mode. The pH indicator appears in the upper right hand corner of the display.

See figure A

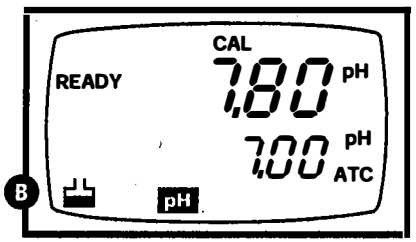


2. Rinse the probe thoroughly with de-ionized water or a rinse solution. Do not wipe the probe; this causes a build-up of electrostatic charge on the glass surface.

3. Dip the probe into the calibration buffer. The end of the probe must be completely immersed into the sample. Stir the probe gently to create a homogeneous sample.

4. Press CAL/MEAS to enter pH calibration mode. The primary display will show the measured reading while the smaller secondary display will indicate the pH standard buffer solution.

See figure B



NOTE: If using a pH buffer other than pH 7, press the ▲ or ▼ keys to scroll up or down until the secondary display value is the same as your pH buffer value (pH 1.68, 4.01, 7.00, 10.01, or 12.45).

5. Wait for the measured pH value to stabilize. The READY indicator will display when the reading stabilizes.

See figure C

6. After the READY indicator turns on, press CON to confirm calibration. A confirming indicator (CON) flashes and disappears. The meter is now calibrated at the buffer indicated in the secondary display.

See figure D

The secondary display automatically scrolls to the next buffer calibration option.

- If you are performing multipoint calibration, go to step 7.
- If you are performing one-point calibration, go to step 10.

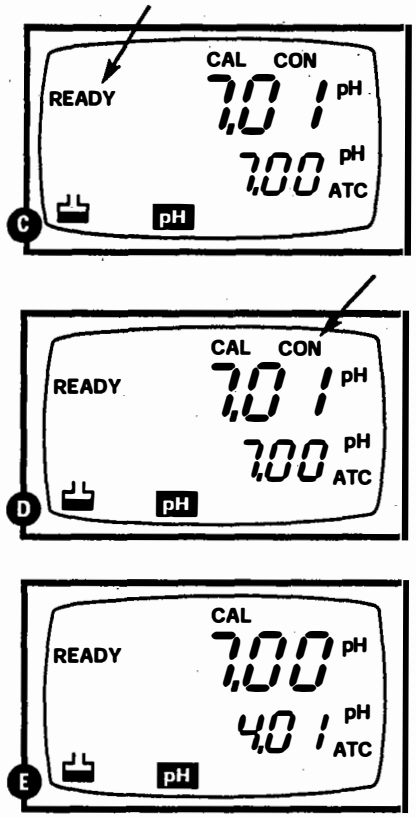
7. Press the ▲ or ▼ keys to select the next buffer value you want to calibrate (pH 1.68, 4.01, 7.00, 10.01, or 12.45).

See figure E

8. Rinse the probe with de-ionized water or a rinse solution, and place it in the next pH buffer.

9. Follow steps 5 to 8 for additional calibration points (up to 5 values).

10. When calibration is complete, press CAL/MEAS to return to pH measurement mode.



Notes

To exit from pH Calibration mode without confirming calibration, DO NOT press CON in step 6. Press CAL/MEAS instead.

If the selected buffer value is not within ±1.0 pH from the measured pH value: the electrode and buffer icon blink and the ERR annunciator appears in the lower left corner of the display. These indicators also flash if the buffer used is not the same as the buffer value on the secondary display.

4.4 Relative mV Calibration

1. While in the measurement function, press MODE to enter the relative mV mode. The primary display indicator shows "Rel mV". If you have never calibrated mV or if the meter has been reset, the value shown is the same as the absolute mV value. Once calibrated, the value shown will be relative mV. The secondary display shows the temperature.

See figure A

2. Press the CAL/MEAS key. The CAL indicator appears above the primary display. The primary display shows the measured reading and the secondary display shows the temperature.

See figure B

3. Press the ▲ or ▼ keys to the mV value to be added or subtracted from the reading.

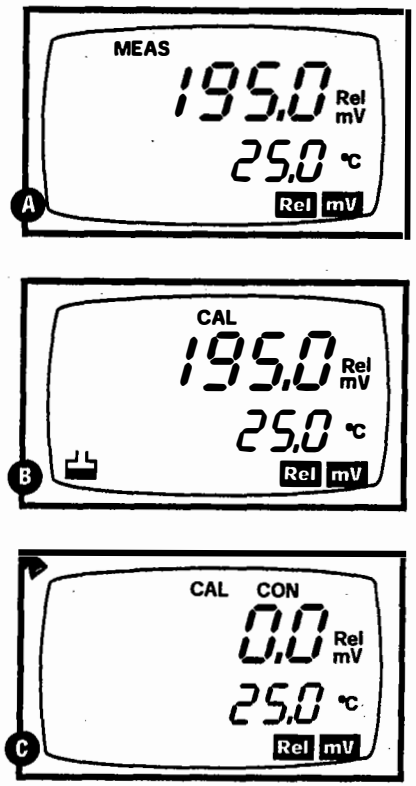
4. Press the CON key to confirm calibration.

NOTE: If you press CON without entering a mV (without scrolling with the ▲ or ▼ keys) the meter will subtract the entire reading value displayed–that is, it will zero the mV reading. The LCD will then show 0 mV.

See figure C

5. Press CAL/MEAS to return to the measurement mode. The primary display now shows the relative mV reading. To recall the selected base value, use the SETUP mode Program 2. See page 24 for instructions.

6. To reset all calibration values in memory to the factory default settings, use the SETUP mode Program 1 (P 1.1). See page23.

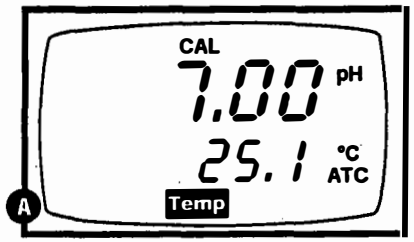


4.5 Temperature Calibration

1-point temperature calibration

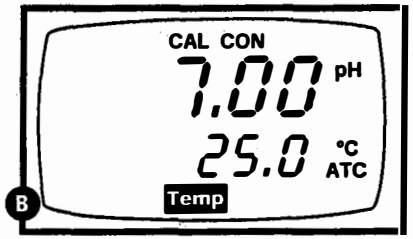
1. Connect ATC probe (or temperature connector of the "All-in-One" electrode) to the phone jack. The ATC annunciator will appear at the right-hand side of the LCD.
2. Press the **MODE** key to select temperature mode (**Temp**).
3. Press the **CAL/MEAS** key to enter calibration mode. The primary display shows the measured pH value and the secondary display shows the temperature.

See Figure A



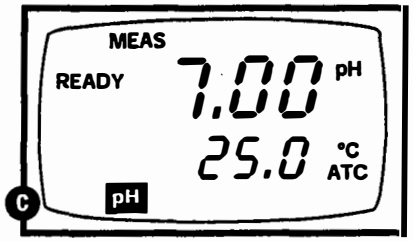
4. Dip the ATC probe (or "All-in-One" electrode) into a solution of known temperature (i.e. a temperature bath). Allow some time for the temperature probe to stabilize.
5. Scroll up or down with the ▼ and ▲ keys to set the correct temperature value (i.e. the temperature of the temperature bath). You can adjust the reading in increments of 0.1°C. Note that the current input reading can only vary ±5°C from the reading originally displayed on the meter.
6. Once you have selected the correct temperature, press the CON key. A confirming indicator (CON) flashes on the display for one second, then disappears.

See Figure B



7. Press the **CAL/MEAS** key to return to pH measurement mode.

See Figure C



2-point temperature calibration

In most cases 1-point temperature calibration will give you accurate temperature measurement and compensation. If you notice inaccurate measurements at high or low temperatures after 1-point calibration, you may need to do a 2-point calibration. Perform the following procedure:

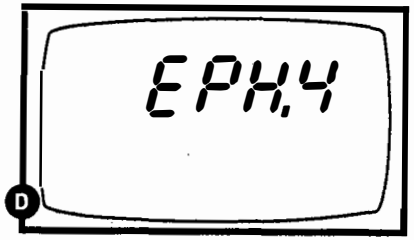
A. Preparing Temperature Baths

1. Prepare two temperature baths for temperature calibration.
2. Set the temperature of the baths. Using a good reference thermometer, preferably certified, set one bath at 0°C ±3.0°C and the second bath at 77-80°C .

NOTE: Make sure to set the baths within the pH electrode operating temperature. For the epoxy-body All-in-One probe (included with meter), do not set the high temperature bath above 80°C. If you are using a glass-body "All-in-One" electrode or a separate electrode and metal ATC probe, set bath to 90.0°C ±3.0°C.

B. Entering 2-point Calibration Mode

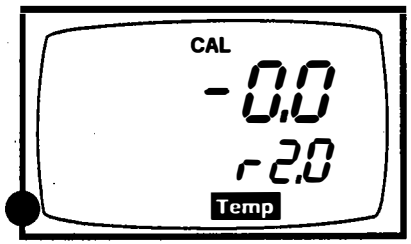
1. Attach the temperature probe or electrode to the meter.
2. Turn the meter off.
3. Press the **CAL/MEAS** key.
4. Without releasing the **CAL/MEAS** key, press the **ON/OFF** key.
5. When the LCD lights up, release the **ON/OFF** key.
6. Wait for 2 seconds, then release the **CAL/MEAS** key. The upper display will show **"EPH.3"** or **"EPH.4"** (depending on the software version number). The meter is now in 2-point Calibration Mode.



See Figure D

7. Press the CON key three times to enter temperature low point calibration.

C. Temperature Low Point Calibration

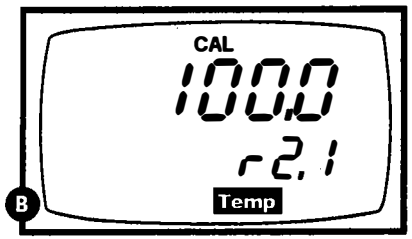


The primary display will show "0.0" and the secondary display will show "r2.0".

See Figure A

1. Insert the temperature sensor or electrode with built-in temperature sensor into the low temperature bath.
2. Press the ▼ and ▲ keys to set the primary display temperature to match the low temperature bath's temperature.
3. Press the CON key to store the selected value. The display will show the A/D value for the temperature. This value appears as a number on the display.
4. Press the CON key to store the A/D value when the number stabilizes.

D. Temperature High Point Calibration



The primary display will show "100.0" and secondary display will show "r2.1".

See Figure B

1. Insert the temperature sensor or electrode with built-in temperature sensor into the high temperature bath.

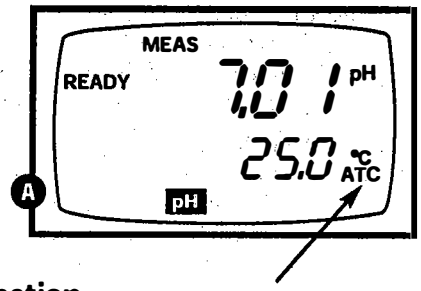
NOTE: To avoid breaking the electrode with temperature shock, allow your electrode to reach room temperature before putting it in a high temperature bath.

2. Press the ▼ and ▲ keys to set the primary display temperature to match the high temperature bath's temperature.
3. Press the CON key to store the selected value. The display will show the A/D value for the temperature. This value appears as a number on the display.
4. Press the CON key to store the A/D value when the number stabilizes.. The meter displays all LCD segments.
5. Press the ON/OFF key to switch off the meter.

5. Measurement

This meter is capable of taking measurements with automatic or manual temperature compensation. Automatic temperature compensation only occurs when the temperature probe is plugged into the meter or when using an OAKTON "All-in-One" electrode. If there is no temperature probe plugged into the meter the default temperature is automatically 25°C. You can manually set the temperature to match your working conditions using a separate thermometer.

5.1 Automatic Temperature Compensation

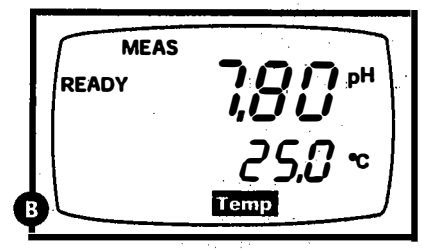


For automatic temperature compensation (ATC) simply plug the temperature probe into the meter (see page 7). The ATC indicator will show on the LCD.

See figure A

NOTE: If you are using a temperature probe, the probe must be submersed in the liquid you are measuring.

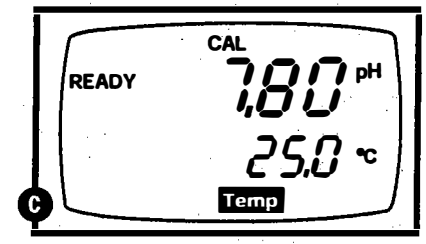
5.3 Manual Temperature Compensation



1. Turn the meter on. Make sure meter is set to the pH measurement mode. Press MODE to select temperature mode.

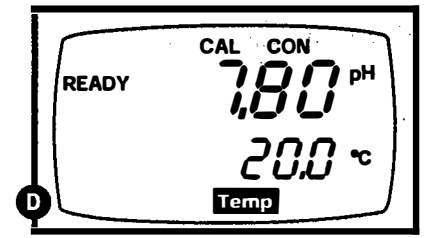
See figure B

2. Press the CAL/MEAS key to enter calibration mode. The CAL indicator will appear above the primary display. The primary display shows the measured pH value and the secondary display shows the temperature.



See figure C

3. Check the temperature of your sample using an accurate thermometer.
4. Press the ▲ or ▼ keys to set the temperature to the measured value.
5. Press CON to confirm the selected temperature. The CON indicator flashes for one second and then disappears.



See figure D

5. Press CAL/MEAS to return to the pH measurement mode.

The meter is now prepared for temperature compensation without the temperature probe.

5.3 Taking Measurements

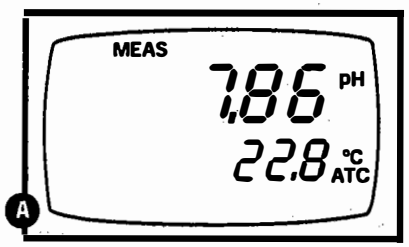
The **READY** indicator appears on the display when the reading stabilizes. The reading holds until the measured value exceeds the tolerance (± 0.02 pH; ± 0.8 mV <400; ±1.2 mV >400), then the **READY** annunciator turns off.

NOTE: Be sure to remove the electrode soaker bottle or protective rubber cap on the electrode before measurement.

To take readings:

1. Rinse the probe with deionized or distilled water before use to remove any impurities adhering to the probe body. If the pH electrode has dehydrated, soak it for 30 minutes in OAKTON electrode storage solution or a 2M–4M KCl solution.

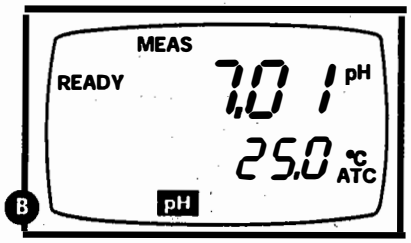
2. Switch on the meter. The **MEAS** annunciator appears on the top center of the LCD. The ATC indicator appears in the lower right hand corner to indicate Automatic Temperature Compensation (See page 17 for setting Manual Temperature Compensation).



See Figure A

3. Dip the probe into the sample.

NOTE: When dipping the probe into the sample, the sensor or the glass bulb of the electrode must be completely immersed into the sample. Stir the probe gently in the sample to create a homogenous sample. *Be sure to tap probe to remove air bubbles. Air bubbles will cause errors in the reading.*

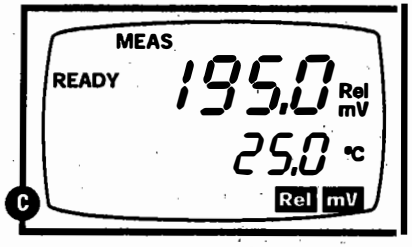


pH measurement mode display

4. Allow time for the reading to stabilize. Note the reading on the display. When the reading is stable, the **READY** annunciator appears.

5. To toggle between pH, Rel mV, and mV readings, press the **MODE** key.

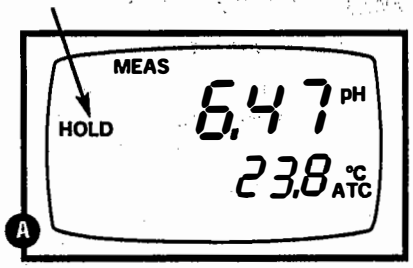
See figures B and C



Relative mV measurement mode display

6. HOLD function

This feature lets you freeze the value of the pH, Rel mV or mV and temperature readings for a delayed observation. **HOLD** can be used any time when in **MEAS** mode.



1. To hold a measurement, press the **HOLD** key while in measurement mode. "HOLD" will appear on the display.

See Figure A

2. To release the held value, press **HOLD** again. Continue to take measurements.

NOTE: This meter will hold a reading for up to 30 minutes, because it features automatic shutoff after 30 minutes to conserve batteries.

7. Memory and data input functions

3.1 Memory Input

Data is stored in sets:

- pH and temperature
- relative mV and temperature
- mV and temperature

This meter can store up to 16 sets of data in any combination of values (pH, mV, and relative mV). For example you can store 7 pH, 5 mV, and 4 relative mV values. The meter uses the last-in-first-out (LIFO) method of memory management within each of the three modes, but not between the modes when used in combination. For example, a pH reading will not replace a relative mV or mV reading. pH readings can only replace pH readings. Relative mV readings can only replace relative mV readings. mV readings can only replace mV readings.

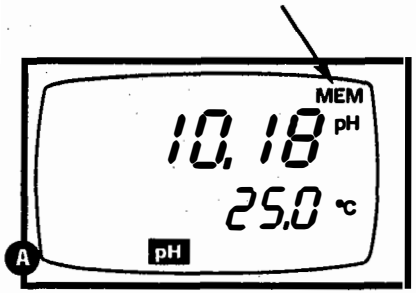
1. During any measurement function (MEAS), press the MI key to input any data into the memory
2. MEM will flash, for a few seconds on the display. The meter stays in measurement mode.
3. If the memory is full, the first value stored will be erased to create space for the new value.

7.2 Memory Recall

This function recalls the previous readings stored in the memory. You can only access MR in the MEASurement mode. In pH mode, MR recalls stored pH values; in Rel mV mode, MR recalls stored relative mV values; and in mV mode, MR recalls stored mV values.

1. Set the mode to the parameter that you wish to recall (pH, relative mV, or mV).
2. Press the MR key once to recall the last reading stored. MEM will flash on the display.

See Figure A



3. Press the MR key again to recall the next to the last reading stored, and so on. NOTE: Readings stored in memory are retained even if the unit is turned off. To erase all readings stored in Memory use the SETUP mode Program 1 (P1.0 or P1.1) on page 23.
4. To prevent accidental memory clearing use the SETUP mode Program 1 (P1.0 and P1.1) and turn the memory clear and reset OFF, see SETUP section page 23 for instructions.
5. To exit Memory Recall, press the CAL/MEAS key to return to the measurement mode.

8. Setup Functions

The SETUP mode lets you customize the meters preferences and defaults through a series of advanced programs.

The SETUP mode has four main programs:

- **Memory clear and reset:** clears or resets all stored values.
- **Electrode Data:** provides diagnostic information on electrode offset and slope, and relative mV offset value. Shows calibration data for both pH and temperature
- **Meter functions:** activates the "Ready" and "Auto-Off" functions. Sets pH resolution.
- **Communication data:** sets up communication parameters for use with a printer or computer.

8.1 SETUP Functions at a Glance

Program	Function Keys	Activation	Options Settings	Default
P1.0	Memory Clear	MR & MI	ON, OFF	OFF
P1.1	RESET	MR & MI	ON, OFF	OFF
P2.0	Electrode Offset	-	Indication only	0.0 mV
P2.1	Electrode Slope	-	Indication only	100.0
P2.2	Displays pH Calibration buffer data	MR & MI	1.68, 4.01, 7.00, 10.01, 12.45 Indication only	"---" No calibration performed
P2.3	Last pH Calibration Temperature	-	Indication only	25.0 °C
P2.4	Relative mV Offset	-	Indication only	0.0 mV
P3.0	pH Resolution	MR & MI	0.1, 0.01	0.01
P3.1	READY	MR & MI	ON, OFF	ON
P3.2	Auto Shut Off	MR & MI	ON, OFF	ON
P4.0	Baud Rate	MR & MI	2.4, 4.8, 9.6, 19.2 Kbps	9.6 Kbps
P4.1	Parity	MR & MI	1, 2, 0	2
P4.2	Stop Bit	MR & MI	1, 2	2

8.2 General Instructions for All SETUP Programs

Please read the next four sections before operating SETUP functions. Refer to "SETUP functions at a glance" page 22 , for a quick review.

1. To enter SETUP mode, press SETUP key while in any measurement mode (pH, relative mV, or mV). The meter automatically enters Program 1, Option 0 (P1.0). You can only access SETUP through the measurement mode.
2. Use MI/MR keys to select options, if changes are required
3. Press CON to confirm the option in each program. The meter then automatically scrolls to the next program in sequence.
4. To exit the program, press CAL/MEAS and return to the measurement mode.

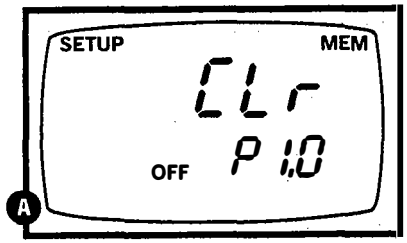
8.3 Program 1: Memory Clear and Reset

Program 1 has two options.

IMPORTANT: OFF is the default setting for both Memory Clear and Reset. Read this section carefully before changing options. Accidentally selecting the wrong option will wipe out all memory.

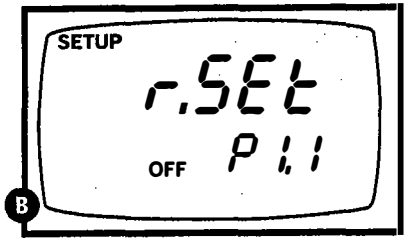
P1.0 Memory Clear: Selecting ON clears all measurement values committed to memory (all 16 values despite the mode). Press MI or MR to select ON, if desired. Once memory is cleared, this program will return to the default setting OFF. Clear memory each time you need to store a new series of values, to avoid confusing the old memory values with the new ones. Press CON.

See Figure A



P1.1 Reset: Selecting ON resets the entire meter. The meter immediately switches off, and you must power ON before proceeding with any other functions. If reset is required, press MI or MR to select ON and then press CON to activate. Reset clears all data: memory values for pH, Rel mV, and mV; and all calibration data. Once memory is cleared, all settings will return to the default setting. Press CON to continue to the next program.

See Figure B



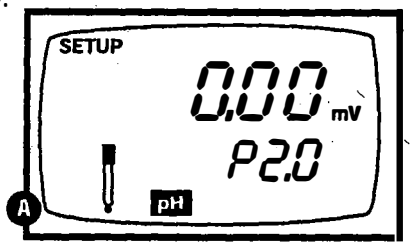
8.4 Program 2: Electrode Data

Program 2 has five options that lets you check the electrode parameters for diagnostic purposes.

NOTE: The electrode icon shows on the display.

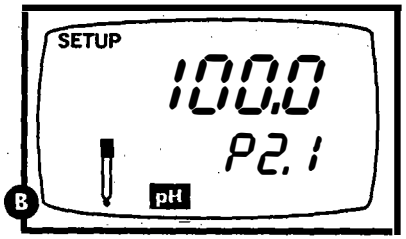
P2.0 Electrode Offset: Shows the pH electrode offset value in mV. The offset is based on the 7.00 pH buffer calibration. If you do not calibrate any buffer, the primary display shows 0.00 mV. No options to select. Press CON.

See Figure A



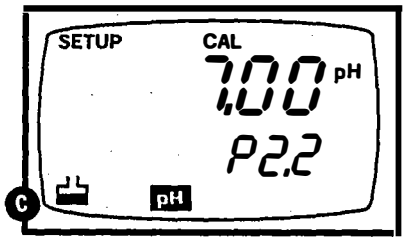
P2.1 Electrode slope: Shows electrode slope in percentage. Slope displayed is the average slope based on the calibrations. Default setting is 100.0. If you did not perform any calibrations, the display will show 100.0. No options to select. Press CON.

See Figure B



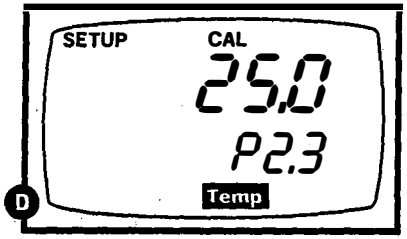
P2.2 Calibration Data: Records calibrations made on the meter, and lets you view all current calibrations points (up to 5). Use MI or MR key to scroll through the five calibrations. Press CON when finished.

See Figure C



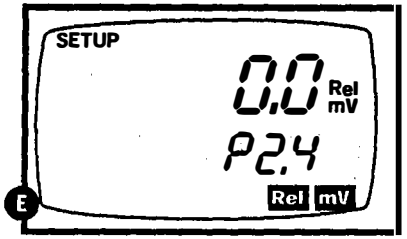
P2.3 Calibration Temperature: Indicates temperature at the last calibration. Default setting is 25°C. No options to set. Press CON.

See Figure D



P2.4 Relative mV Offset Value: Shows the relative mV base value in mV. Based on your selected value during calibration for REL mV measurement. Default setting is 0 mV. If you did not perform any calibration, the display will show 0 mV. No options to select. Press CON.

See Figure E

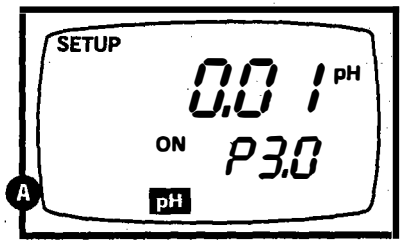


8.5 Program 3: Selectable Meter Functions

Program 3 has three options for customizing.

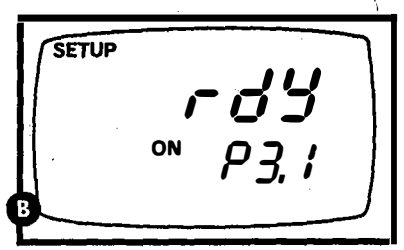
P3.0 Resolution: In the pH mode, select resolution of 0.1 or 0.01 pH. 0.01 pH is the default setting. Press MI or MR to switch to 0.1 or 0.01 pH (0.1 pH resolution is optimal for fast pH checks). Press CON

See Figure A



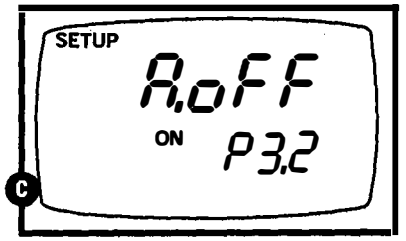
P3.1 READY selection: Use the MI or MR key to turn ON or OFF the READY option. READY indicator will display when the reading stabilizes within ±0.02 pH (±0.8 mV <400 mV; ±1.2 mV >400 mV). The default setting is ON. Use MI or MR key to select ON or OFF. Press CON.

See Figure B



P3.2 Auto-Off: To conserve energy, this function automatically shuts off the meter within 30 minutes after the last key has been pressed. Default setting is ON. Use MI or MR key to select ON or OFF.

See Figure C



STANDARD OPERATING PROCEDURE
pH (Orion Model 230A)

This SOP describes the use of the Orion Model 230A pH meter for measurement of pH in water samples. General instruction for measurement of pH in aqueous samples (EPA Method 9040A) and in soil and solid waste samples (EPA Method 9040A) are described in SOP No. FM-001-M, "pH - General Procedures."

1.0 INSTRUCTIONS (see Note 1)

1.1. **Meter Setup and Check** (see Figure 1)

1.1.1 Attach BNC Shoring Plug (attached to meter by a head chain) to the BNC connector on top of meter. Press **power** key. If "bat" is displayed during second and third display, the battery is weak and should be replaced (see Note 2). Press **power** key again to turn meter off.

1.1.2 Meter self-test: Press the **power** key and the **yes** key in rapid succession to start the self-test. The meter will automatically go through a series of tests. When "7" appears in the lower half of the display followed by "0" press each of the keypad keys in succession. The meter will then turn itself off. See Note 3 for more information on the self-test.

1.1.3 Electrode Attachment (see Note 4)

1.1.3.1 Remove BNC shoring plug and attach BNC and ATC connectors from the Triode electrode.

1.1.3.2 Remove electrode form storage solution and rinse thoroughly with distilled water.

1.1.3.3 Slide the protective sleeve down to expose the electrode filling hole and add electrode filling solution (Section 2.3) if needed.

1.2. Daily Calibration - The meter should be calibrated using two buffers at least once per day. The meter calibration should be checked periodically during use (once every 5 to 10 samples or once every hour) to check meter and electrode calibration. A single buffer (near the pH of the samples) can be used for calibration checks. Record calibration data and calibration checks to verify meter operation (see Note 5).

The auto-calibration method can be used for routine pH analysis of samples at or near room temperature. For precise work or for measurement of pH of cold (less than 10° C) or warm (more than 30° C) samples, use the manual method.

1.2.1 Autocalibration

1.2.1.1 Press power key to turn meter on.

1.2.1.2 Place electrode in pH 7 buffer and press cal key.

1.2.1.3 When the reading is stable, "READY" will be displayed. Press yes key.

1.2.1.4 When "P2" is displayed, remove the electrode, rinse thoroughly, and place in a pH 4 or pH 10 buffer (see Note 5).

1.2.1.5 When the reading is stable, "READY" will be displayed. Press yes key.

1.2.1.6 The meter will display "SLP" and the measured electrode slope (in percent). The slope should be between 92% and 102% (see Note 6).

"MEASURE" will be displayed and meter is ready for samples.

1.2.2 Manual Calibration

1.2.2.1. Press power key to turn meter on.

1.2.2.2. Place electrode in pH 7 buffer. Press cal key.

When pH reading is stable, read the displayed temperature. Refer to the temperature/pH chart for the buffer being used to determine the actual pH at the temperature used (see Note 7).

1.2.2.3. Press the λ or V key. The first digit of the displayed pH value will flash. Scroll (using the λ or V keys) until the first digit is correct. Then press the yes key. The second digit will now flash. Scroll until the second digit is correct and press the yes key. Do the same for the third digit.

1.2.2.4. When "P2" is displayed, remove the electrode rinse thoroughly and place in a pH 4 or pH 10 buffer (see Note 5).

1.2.2.5. Repeat Steps 1.2.2.2. and 1.2.2.3.

1.2.2.6. The meter will display "SLP" and the measured electrode slope (in percent). The slope should be between 92% and 102% (see Note 6).

1.2.2.7. "MEASURE" will be displayed and meter is ready for samples.

1.3 Sample Measurement

1.3.1 Place the electrode in the sample. If "MEASURE" is not displayed, press **measure** key.

1.3.2 When the reading is stable, "READY" will be displayed. A small amount of missing may help reduce the time to reach a stable reading (see Note 8). Record the pH and temperature readings.

1.3.3 Remove the electrode, rinse thoroughly with distilled water, and shake or blot off excess water before placing in next sample. For temporary storage between readings, the electrode can be kept in distilled water or in electrode storage solution.

1.3.4 Discard the sample. Do not use the sample for conductivity measurements or return to the sample bottle.

1.4 Preparation for Transport or Long-Term Storage

1.4.1 Place a few drop of electrode storage solution in the rubber cap and place on the tip of the electrode.

1.4.2 Slide the electrode sleeve over the filling hole. Disconnect the electrode from the meter. Be sure power key is off.

1.5 Notes:

Note 1 This meter has several features that the operator can modify if desired. If not modified, this meter will:

a. Automatically turn off the power if a keypad key had not been pressed in 10 minutes (automatic shutoff does not affect calibration).

b. A "beep" will be heard whenever:

- a key is pressed
- a "READY" signal is displayed
- an operator assistance code is displayed.

c. A "READY" will be displayed when the pH reading is stable.

Any of the above can be cancelled and some additional features added by using the SETUP mode. See pages 13 through 16 of the attached instrument manual for making these changes.

Note 2 The display of "bat" anytime during calibration or measurement indicates a weak battery which should be replaced with either a 9-volt alkaline or lithium battery.

Note 3 See Chapter IX - Troubleshooting" (page 47) of the instrument instructions for description of each step in the meter self-test. If the meter finds an error during the self-test, an Operator Assistance Code (such as E-2, E-3, etc.) will be displayed. See page 48 of the meter instruction for code descriptions. (Note that the display of "E-7" may indicate either a keypad problem or that the operator failed to test the keypad.)

Note 4 A Triode electrode is described here. A combination pH electrode with a BNC connector and a separate ATC probe can also be used.

Note 5 The pH 10 buffer is easily contaminated by absorption of CO_2 from the air. When using this buffer for calibration, be sure to use fresh buffer solutions. The pH 4 buffer is much more stable.

Note 6 If the measured slope is not between 92% and 102%:

- Re-calibrate with fresh buffers.
- Use manual calibration method.
- Adjust temperature to about 20 to 25° C.
- Clean electrode.

Note 7 If a temperature/pH chart is not available, use the chart on page 7 of the attached "Triode pH Electrode Instruction Manual."

Note 8 Some samples, such as samples having very low ionic strength, may require a long time to reach a steady reading. For these samples, "READY" may not be displayed. Record the pH to the nearest 0.1 pH unit. Check the electrode response with a buffer solution; if the response is normal, the slow response is sample related. If response in the buffer is also slow, the electrode may need cleaning or replacement.

2.0 EQUIPMENT AND MATERIALS

2.1 Orion Model 230A pH meter

2.2 Orion Model 91-57 BN Triode pH electrode

2.3 Electrode Filling Solution, 4MKCl saturated with AgCl

Orion Part No. 900011 (This is the same filling solution used for the Cole-Parmer pH Wand.)

2.4 Electrode Storage Solution. Orion Part No. 910001 or add about 1 gram KCl to 200 milliliters pH 7 buffer.

2.5 Buffer solutions having nominal pH values of 4, 7, and 10. Two solutions of each buffer are recommended:

- A working buffer for calibration and pH checks.
- A stock solution of fresh, unused buffer.

2.6 A temperature/pH chart for each buffer.

2.7 Probe Cleaning Solutions:

- Liquid household detergent. 0.1 to 0.5% in water.
- 1:1 methanol:water solution
- Dilute HCCCl.0.1M

Note: See page 8 of Triode pH Electrode Instruction Manual for other cleaning solutions.

2.8 Distilled or deionized water.

2.9 Tissue or paper hand towels for blotting electrode tip after rinsing.

3.0 ROUTINE EQUIPMENT CHECKS AND MAINTENANCE

3.1 The meter should be calibrated with two buffers at least once per day. Check calibration frequently during use, once every 5 to 10 samples or once per hour and re-calibrate if necessary.

3.2 Electrode Preparation: The following procedure is used to prepare a new electrode for use and for treating an electrode that has been cleaned using some of the stronger cleaning agents.

3.2.1 Fill the electrode with fresh electrode filling solution (Section 2.3).

3.2.2 Suspend upright in air for 15 minutes to thoroughly wet the reference junction.

3.2.3 Soak the electrode in electrode storage solution (Section 2.4) for at least one hour.

3.3 Probe Cleaning: The following methods are listed in order of increasing severity:

3.3.1 Rinse with distilled water.

3.3.2 Soak electrode tip in dilute detergent solution (Section 2.7) for a few minutes and thoroughly rinse with distilled water.

3.3.3 Rinse electrode tip with methanol or isopropanol solution (Section 2.7), thoroughly rinse with distilled water, and soak in electrode storage solution (Section 2.4) one hour.

3.3.4 Soak electrode tip in dilute HCl (Section 2.7) for 15 to 30 minutes, rinse thoroughly with distilled water, and soak in electrode storage solution (Section 2.4).

3.3.5 See Page 8 of Triode pH Electrode Instruction Manual for other cleaning procedures.

4.0 TROUBLESHOOTING PROCEDURES

4.1 Meter Troubleshooting: See Chapter IX Troubleshooting" (pages 47-51) of the attached meter instruction manual.

4.2 pH Electrode: See Page 9 and 10 of the attached Triode pH Electrode Instruction Manual.

4.3 General Troubleshooting Guide: The following general procedures may be useful in correcting observed problems.

4.3.1 No display on meter when POWER key is pressed.

- Dead battery - replace battery.
- Faulty meter - repair meter.

4.3.2 Stable pH reading, but no response to pH change.

- Poor probe connection - check connections.

- Faulty probe or meter - try another probe known to be good, if available. If operation is okay, the old probe was faulty and should be replaced. If not okay, meter may need repair.

4.3.3 Meter cannot be calibrated to pH 7

- Grossly dirty probe - clean probe (see Section III.C.)
- Faulty probe - replace probe.

4.3.4 Meter cannot be calibrated to second buffer.

- Contaminated buffer solution - recalibrate using fresh buffer solution for both buffers.
- Broken glass bulb in tip of probe - replace probe.
- Dirty probe tip - clean probe (see Section 3.3)
- Plugged or dried up reference junction - replace electrode filling solution (Section 2.3) and perform electrode preparation procedures (Section 3.2).

4.3.5 Slow electrode response, drifting, or unstable readings.

- Poorly mixed sample - mix sample.
- Sample is not aqueous - pH of non-aqueous samples cannot be measured.
- Aqueous sample has a very low ionic strength (low dissolved solid) - report pH to nearest 0.1 unit.
- Dirty probe tip - clean probe (see Section 3.3).
- Plugged or dried up reference junction - replaced electrode filling solution (Section 2.3) and do electrode preparation procedure (Section 3.2).
- Long exposure to pH less than 0.5 or more than 13 - soak probe in several fresh solutions of electrode storage solution (Section 2.4), thoroughly rinse, and re-check. If ineffective, replace probe.

5.0 SAFETY REQUIREMENT

Some of the probe cleaning solutions are acidic and should be handled accordingly.

6.0 REFERENCES

6.1 Copies of the pertinent parts of the following instruction manuals are attached:

6.1.1 Orion pH Meter Instruction Manual (only parts of the manual pertaining to Model 230A meter are included in the attachment).

6.1.2 Orion Triode pH Electrode Instruction Manual.

6.2 See SOP No. FM-001-M for general instructions for measurement of pH in aqueous samples (EPA Method 9140A) and in soil and solid waste samples (EPA Method 9145A).

7.0 CONTACTS FOR ADDITIONAL HELP

7.1 STS Contacts:

7.2 Manufacturer contacts: For information on Orion meters or electrodes, call the Orion Product Service Department at (800) 225-1480 or (617) 242-3900.

Note: A return authorization number **must** be obtained from Orion before returning any item for repair.

8.0 FORMS

There are no specific forms for pH data.

STANDARD OPERATING PROCEDURES
SPECIFIC CONDUCTANCE AND TEMPERATURE

Method: Specific Conductance, µmhos @ 25°C
Reference: EPA 1979, page 120.1: Standard Method, 15th Edition, pages 70-73.
Detection Limits: 2.5 µmhos /cm @ 25°C; -2°C
Optimum Range: 0 to 50,000 µmhos /cm
Sample Handling: Determined in field while sampling.
Reagents & Apparatus:

1. Conductivity meter (YSI Model 33) and probe.
2. Distilled water in squirt bottle.
3. Calibration solution set (1,000 and 10,000 µmhos/cm)

Calibration Procedure

1. Check meter zero with the mode switch in the off position. If the meter does not read zero, adjust the meter screw to read zero.

2. Turn mode switch to "Red Line." Use the Red Line control knob to adjust meter needle to line up with the red line on the meter face. Replace batteries if this adjustment cannot be made.

3. Check the suitability of the conductivity probe by measuring the conductivity of the a standard conductivity solution.

Operation Procedure

1. Calibrate the meter as described above.

2. Immerse the probe in the sample to be tested. Turn mode switch to "Temperature" and read temperature from meter while gently stirring.

3. Immediately after measuring temperature, turn the mode switch to one of the appropriate conductivity scales (X100, X10, or X1) and read the meter scale. Multiple the meter reading by the selected scale factor and record the measured conductivity and temperature.

4. Report the conductivity measured along with the measurement temperature or report the conductivity corrected to 25° C using the calculation described below.

5. Rinse the probe in distilled water and blot off excess rinse water between samples.

Data Calculation and Reporting

$$C25 = \frac{C_T}{1 = 0.02\,(T-25)}$$

Where: C25 is the specific conductance (µmhos/cm) at 25°C.
C_T is the measured conductivity (µmhos/cm).
T is the measured temperature (°C) of the samples.
Report the corrected specific conductance as µmhos/cm at 25°C.

Quality Control

Conductivity Measurements:

The frequency, acceptance criteria, and correction action for quality control (QC) checks recommended for this method are listed below:

QC Check	Frequency	Acceptance Criteria	Corrective Action
Calibration	Daily	± 10% of standard	1. Clean probe. 2. New standard. 3. Charge batteries. 4. Repair or replace probe.
Duplicate Analysis	10%	RPD < 15%	1. Obtain third value. 2. Flag value.
QC Check Sample	10%	RPE < ± 15%	1. Check calibration (zero and red line). 2. Use new standard. 3. Clean probe.

RPD = Relative Percent Difference.
RPE = Relative Percent Error.

The calibration check is performed by measuring the conductivity of a standard conductivity solution. (Standards of 1,000 and 10,000 µmhos/cm are typically used. Use the standard closest in value to the samples to be tested.) The calibration check performed immediately after meter calibration should be within 10% of the standard value (be sure to correct for temperature). If it is not, one or more of the following may be the cause of the discrepancy:

- The standard solution may be contaminated.
- The probe may be dirty.
- The probe may be damaged.

Temperature Measurement

It is recommended that the accuracy of the temperature measurement be verified before each major field survey by comparing the instrument temperature reading to a laboratory thermometer reading using a well-mixed water bath at temperatures bracketing the temperatures expected in the field test.

Preventative Maintenance and Precautions

1. Oil or air bubbles adhering to the measurement surfaces of the probe will result in faulty readings. Remove air bubbles by moving the probe in the sample container (swirling the probe or alternately removing and re-immersing the probe). Oil material may be removed by using a detergent solution. To remove chemical fouling, the probe may have to be re-platinized or replaced.

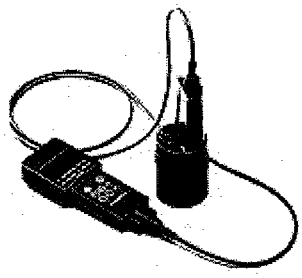
2. If samples cannot be measured immediately, cool the sample to 4°C and measure conductivity within 24 hours.

3. The formula used for temperature correction is somewhat matrix dependent. For most accurate results, measurements should be made as close to 25°C as possible.

Analytical Instruments

Products

Water Quality Checker
U-10



Contact

- U-10
- Specifications
- Details
- Detailed Specifications

Parameters		pH	Conductivity
Principle		Glass electrode	Alternating four-electrode
Range		0-14 pH	0-100 mS/cm
Resolution	Standard	0.1 pH	(mS/cm)(mS/cm) 0 -1:0.01 1 -10: 0.1 10-100: 1
	Expanded	0.01 pH	(mS/cm)(mS/cm) 0-1:0.001 1 -10:0.01 10-100: 0.1
Repeatability		±0.05 pH	±1% of full scale
Temp. compensation		0-50°C	0-50°C
Calibration		Auto one-point calibration, manual two-point calibration	Auto one-point calibration, manual two-point calibration
Display		LCD	
Printer output		Centronics	
Power		Battery 6F22 (S-006P)x1 pc.	
Sensor assembly		Standard: sensor with 2 m cable, Optional: sensor with 10 m cable	
Weight		Main unit: approx. 400 g (0.9 lb), Sensor assembly (2 m cable): approx. 800 g (1.8 lb)	

Parameters		Turbidity	Dissolved oxygen
Principle		Scattering/ transmitting light	Membrane/ galvanic cell
Range		0-800 NTU	0-19.9 mg/L
Resolution	Standard	10 NTU	0.1 mg/L
	Expanded	1 NTU	0.01 mg/L
Repeatability		±3% of full	±0.1 mg/L

	scale	
Temp. compensation	-	0-40°C
Calibration	Auto one-point calibration, manual two-point calibration	Auto one-point calibration (air), manual two-point calibration
Display	LCD	
Printer output	Centronics	
Power	Battery 6F22 (S-006P)x1 pc.	
Sensor assembly	Standard: sensor with 2 m cable, Optional: sensor with 10 m cable	
Weight	Main unit: approx. 400 g (0.9 lb), Sensor assembly (2 m cable): approx. 800 g (1.8 lb)	

Parameters		**Temperature**	**Salinity**
Principle		Thermistor	(Alternating four-electrode)*
Range		0-50°C	0-4 %
Resolution	**Standard**	1°C	0.1 %
	Expanded	0.1°C	0.01 %
Repeatability		±0.3°C	±0.1%
Temp. compensation		-	-
Calibration		-	-
Display		LCD	
Printer output		Centronics	
Power		Battery 6F22 (S-006P)x1 pc.	
Sensor assembly		Standard: sensor with 2 m cable, Optional: sensor with 10 m cable	
Weight		Main unit: approx. 400 g (0.9 lb), Sensor assembly (2 m cable): approx. 800 g (1.8 lb)	

*Salinity is calculated from the conductivity data.

Oxygen CHEMets®

1 - 12 ppm

Sampling

The most critical part of any dissolved oxygen test is sampling. It is difficult to obtain an aliquot which accurately reflects the oxygen content of a sample. Exposure to the high oxygen content of "air" will cause a sample to approach saturation. Biological activity may cause rapid oxygen depletion. Dipping and pouring operations should be performed with as little agitation as possible. A two foot sub-surface sampler/snapper is available to minimize sample contamination (see Reorder Information).

Test Procedure

1. Fill the sample cup to the 25 mL mark with your sample (fig. 1).
2. Place the CHEMet ampoule in the sample cup. Snap the tip by pressing the ampoule against the side of the cup. The ampoule will fill, leaving a small bubble to facilitate mixing (fig. 2).
3. Mix the contents of the ampoule by inverting it several times, allowing the bubble to travel from end to end each time. Wipe all liquid from the exterior of the ampoule. Wait **2 minutes** for color development.
4. Hold the comparator in a nearly horizontal position while standing directly beneath a bright source of light. Place the CHEMet ampoule between the color standards moving it from left to right along the comparator until the best color match is found (fig 3). If the color of the CHEMet ampoule is between two color standards, a concentration estimate can be made.

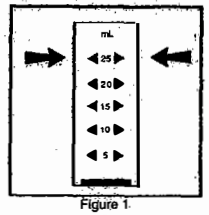


Figure 1.

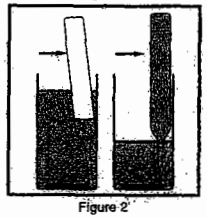


Figure 2

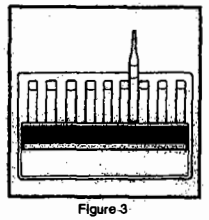


Figure 3

Test Method

The Dissolved Oxygen CHEMets®[1] test employs the indigo carmine method[2,3]. In an acidic solution, oxygen oxidizes the yellow-green colored leuco form of indigo carmine to form a highly colored blue dye. The resulting blue color is proportional to the dissolved oxygen concentration in the sample. Test results are expressed in ppm (mg/Liter) dissolved oxygen as O_2.

1. CHEMets is a registered trademark of CHEMetrics, Inc. U.S. Patent No. 3,634,038
2. ASTM D 888 - 87, Dissolved Oxygen in Water, Test Method A–Colorimetric Indigo Carmine
3. Gilbert, T. W., Behymer, T. D., Castaneda, H. B., "Determination of Dissolved Oxygen in Natural and Wastewaters," American Laboratory, March 1982, pp. 119-134

Safety Information

Read MSDS before performing this test procedure. Wear safety glasses.

Important Note

The CHEMet ampoules contain a reagent which will deteriorate upon prolonged exposure to light. They will remain stable only if stored in the dark.

Reorder Information — Cat. No.

***Test Kit**, complete*	*K-7512*
***Refill**, 30 CHEMet ampoules*	*R-7512*
***Sample Cup**, 25 mL, package of six*	*A-0013*
***Comparator**, 1-12 ppm*	*C-7512*
Sub-surface Sampler/Snapper	*A-0139*

Kits are available for dissolved oxygen analysis at other levels.

CHEMetrics

Rt. 28, Calverton, VA 20138-0214 U.S.A.
Phone: (800) 356-3072; Fax: (540) 788-4856
E-Mail: prodinfo@chemetrics.com
www.chemetrics.com

2084-5

Tesch, Jan J.

From: Teresa Neale [tneale@chemetrics.com]
Sent: Monday, August 16, 2004 12:22 PM
To: Tesch, Jan J.
Cc: Melanie Anderson
Subject: K-7512 Dissolved Oxygen Test Kit accuracy

Jan,
Here is the accuracy claim on the K-7512 Dissolved Oxygen Test Kit:

+/- 0.5 ppm in the 1-6 ppm range
+/- 1.0 ppm in the 6-12 ppm range

I do not have a numerical precision value. This is a visual colorimetric test, the repeatability of the the color produced is much better than the human eye can determine.

I hope this is helpful information.

Teresa

Teresa A. Neale
Director of Operations &
Product Support
CHEMetrics, Inc.
Calverton, VA 20138 USA
Phone: (540)788-9026
Fax: (540)788-4856
email: tneale@chemetrics.com

SOP-950 - Metals by ICP

Determination of: EPA Method 200.7/6010 for the determination of Trace Metals Analysis of Water and Wastes by ICP Emission Spectroscopy. This method describes a technique for the sequential multi-element determination of trace elements in solution.

Scope/Purpose: To provide a method for which soluble, suspended, and total metals in drinking water, surface, water, domestic, and industrial wastewater can be analyzed. Also, to provide a method for the analysis of total elements in soils, sludges, and other solid wastes.

Reagents: SPEX Multi-Element Plasma Standards; single-element Standards; and Fisher-Baker Standards; nitric acid.

Equipment: Inductively Coupled Plasma (ICP) spectrophotometer by SA Jobin Yvon, Model Ultrace 138; Gilson Autosampler; Gateway P5-120 PC; JYESS software; volumetrics; pipettes; and polyethylene bottles.

References:

Code of Federal Regulations Title 40, Part 136, Appendices A and B, US Government Printing Office, Washington D.C. (20402), 1991, EPA 200.7.

Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, Office of Research and Development, June 1991, EPA 200.7.

Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Environmental Monitoring and Support Laboratory, 26 West Martin Luther King Drive, Cincinnati, Ohio (45268), Revised 1983 including EPA-600/4-84-017, March 1984, EPA 200.7.

Test Method for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. (20460), November 1986 including December 1987 and November 1990 updates. EPA 6010.

Quality Assurance/Quality Control Manual, US Filter/Enviroscan, 1997.

JY User's Manual, Reference 31-088-409 \

Interferences:

Positive interferences are shown on Table 1: Interference Study on JY-138 Ultrace.

The negative interferences have little significance if the level of interferants is low or analyte concentration is high. The negative responses area a result of baseline fluctuation caused by the interferant of by a peak that occurs on or close to the background point. Most of the time, the background points can be changed; but in a few cases, they are unavoidable.

In either case (positive or negative interference), the problem usually can be solved by diluting eh reporting a higher detection limit for the analyte or by choosing an alternate wavelength. In either case, the detection limit will be higher than what is normally reported.

A summary of negative results is below:

1,000 ppm Ca, Mg, Al; 400 ppm Fe causes a negative result for the following:	
Se (196.090) of 0.058 ppm	Sn (242.170) of 0.062 ppm
Sb (206.833) of 0.027 ppm	Tl (276.787) of 0.085 ppm
Ag (328.068) of 0.027 ppm	
100 ppm Cr, Cu, Ni, Ti, V Mn cause a negative result for the following:	
Sn (242.170) of -0.508 ppm	Mg (279.079) of -0.797 ppm
Fe (238.204) of -0.153 ppm	
400 ppm Fe causes a result for Cu (324.754) of -1.028 ppm	

ppm = parts per million

Table 1 - Interference Study on JY-138 Ultrace

Element	Interferant	Concentration	Element Result (ppm)	Cause of Interference
Zn (213.856)	Cu	100	0.205	Peak of Cu on Zn line.
Zn (213.856	Ni	100	1.243	Peak of Ni on Zn line.
As (220.353)	Al	!,000	0.600	Tailing of Al causes high reading for As; cannot use two B.G. points due to other interferences.
Pb (220.353)	Al	1,000	0.009	Tailing from nearby pea. Problem solved by using two B.G. points.
Be (313.042)	V	100	0.116	V has peak close to Be peak; causes tailing and high result for Be.
V (292.402)	Fe	400	0.134	Small peak very close to V line caused high result.
V (292.402)	Ti	100	0.100	Small peak close to V line.
Co (228.616)	Ti	100	0.202	Small peak close to Co line.
Sb (206.833)	Cr	100	0.825	Tailing from nearby peak.
Se (196.090)	Cr, Cu, Ti, V. Mn, Ni	100	0.011	Small peak which may be noise at this level.

Sampling, Preservation, and Holding Time:

Sample Matrix	Container	Sample Preservation	Holding Time
Drinking Water	100-ml glass or plastic	HNO_3 to pH < 2.0	6 months
Wastewater	100-ml glass or plastic	HNO_3 to pH < 2.0	6 months
Soils	10 grams of sample	Glass/no preservation	6 months

Guidelines:

The following steps are outlined for the startup and operation of the JY-138 ICP.

Instrument Startup:

1. Turn valves on the argon tank to start gas flow (approximately 80 pounds per square inch [psi]).
2. Check pressure on nitrogen tank (@ red line on the gauge).
3. Press **P1, G1,** and **NEBU** keys on the front of the instrument to start argon flow.
4. Hook up pump tubes and start pump to aspirate water.
5. Wait a few minutes, then push the **NEBU** key again.
6. Turn OFF the pump and SLOWLY release the pressure from the argon humidifier using the black handle located on the inside left corner of the torch box.

   Note: The water level in the humidifier should be between the black lines.
7. After completing Steps 1-6, push the **START** key to ignite the torch.

   Note: The nebulizer flow must be **OFF** to start the torch.
8. Wait about a minute and press the **NEBU** key again to restart the pump.

   NOTE: THE INSTRUMENT MUST BE WARMED UP FOR APPROXIMATELY ONE HOUR BEFORE ANALYZING ANY SAMPLES.

Instrument Shutdown:

1. Press the **STOP** key and allow the instrument to cool down.
2. Press **P1, G1,** and **NEBU** keys to shut off.
3. Turn **OFF** the pump and unhook the pump tubes.
4. Turn the valves on the argon tank to shut off flow.

Computer Software:

This is a summary of the software for the JY-138 ICP. For specifics, refer to the J-Yess Version 4.0 Software Manual.

1. Turn on the computer and monitor to bring up the Windows screen.
2. Exit or close the messages on the screen. Double click on JY software icon.
3. Continue by touching any key to get the first menu screen.
4. Pressing the left pointing arrow will give the Main Menu of the software.
   Note: A zero order search will automatically be done when you press enter.
5. The "Shift F3" with no option change will automatically open the Autosampler option.

6. Press "F10" to choose Autosampler.
   Note: This unit has a Gilson 222 Autosampler.

7. Press the "escape" key twice to return to the Main Menu.

   The Main Menu contains the following options:

   - Methods
   - Preparation
   - Analysis
   - Results

Methods:

The Methods option is used to set up and modify the analytical files. The options TMISC, TRCRA, TBESRB, and TWAR are described in detail on Table 2, Method Summary for the JY-138 Ultrace.

Preparation:

The Preparation option contains Auto-attenuate, Auto-search, Profiles, and Calibration:

Auto-attenuate sets the high voltage for the photo-multiplier tube for each element in the file or selected ones. Use the high standard for this option.

Auto-search locates the exact peak position and transfers the information to the software for the location of the offset peaks. Press "F10" to continue with the next element.

Note: This option is used for each method at the beginning of each day's run. If more than one peak appears on the scan, the software will give a list of possible elements for each line.

Profiles allow you to see a scan of samples, standards, etc. The background points may be chosen for the analysis from the scan.

Calibration allows for the calibration of the method. Standard concentrations are listed on a separate summary page. Determine which standard will be analyzed by selecting "yes" or "no."

- To SAVE the modification press "F8."
- To SELECT elements press "F3."
- To DISPLAY calibration curve press "F3."
- To PRINT press "Shirt, Print Screen."

  Note: If a standard is not selected, you must exit the current calibration mode and start a new calibration procedure.

THE INSTRUMENT MUST BE CALIBRATED BEFORE SAMPLES MAY BE ANALYZED.

Analysis:

The software program allows for individual analysis or group analysis. The **Analysis** option allows for a single sample to be analyzed. The **Sample File** option allows for the analysis of one or more metals.

Results:

The **Results** option allows for the review of completed analysis.

EXIT SOFTWARE PROGRAM:

To exit the program, go to the DOS prompt and type "exit." Press "Start/Shut Down" option to turn off computer. Note: The monitor must be shut off separately.

Instrument Maintenance:

Flush the nebulizer with deionized water and air after each use. Check for clogging or chips and replace, if necessary.

Quality Assurance/Quality Control (QA/QC) Information:

Each sample run (10 samples) shall have a check standard, matrix spike, matrix spike duplicate, and calibration blank every 10 samples. A reagent blank is analyzed daily for each prepped matrix.

Default limits of 25% for duplicates and 75% to 125% for matrix spikes are used until enough data has been collected to establish new limits. Samples shall be spiked at various concentrations depending on the elements of interest and the sample concentration. Spikes or duplicates out of control shall be repeated, or the data shall be flagged on the benchsheet and final report.

NOTE: IF CHECK STANDARDS DO NOT FALL WITHIN THE LIMITS OF 5% AND 10% FOR EPA 200.7 AND EPA 6010, RESPECTIVELY, SAMPLES MAY NOT BE RUN UNTIL PROBLEM IS CORRECTED.

Glassware is washed thoroughly, rinsed with acid, and then with distilled water. All QC data is recorded appropriately and is easily retrievable.

Calibration Standards:

The calibration standards are made up from the 1,000 ppm plasma stock solutions. Add one milliliter of 1:1 HNO_3 per one liter of total standard volume. All dilutions are made with Modulab deionized water. The final standard solutions are stored in properly labeled polyethylene bottles. The calibration concentration tables used for each task file are listed in Table 2, Method Summary for JY-138 Ultrace.

The calibration standards are verified after every calibration using the SPEX multi-element plasma standards. The standards are analyzed at different concentrations depending on the element being checked and the concentration range of the task file. The SPEC standard must be within 10% of true value.

Reagent Blanks:

The reagent blanks are prepared by taking 100 ml of deionized water and carrying it through the complete metal digestion process. Blanks are run for the liquid prep, the solid prep, and for the recoverable prep. Reagent blanks are analyzed on every prepped sample run. The reagent blank is dependent on the sample matrix and prep performed.

Check Standards:

A check standard is used for each task file and has a concentration approximately in the middle of the calibration range. The check standard must be within 10% of the true value for EPA 6010 and 5% for EPA 200.7. The check standard is independent of the calibration standards.

Method Detection Limits:

Method detection limits are conducted according to Method EPA 200.7, EPA 6010, and 40 CFR, Part 136, Appendix B. Current MDL are attached to this procedure.

Calibration Instructions:

Calibrate the instrument according to JY-138 Ultrace User's Manual. The system is flushed out with a blank between each standard. An average intensity from three readings of each standard is used to reduce random error.

Reporting Data:

Samples that require dilutions are entered in the Autosampler file, and the final dilution result is checked against the initial analysis of the concentrated sample and a comparison is made between the two sample results. Data is reported in milligrams per liter (mg/L) to three significant figures.

Summary:

Below is an analysis summary of a sample set:

1. Start instrument, let warm up, and do the zero calibration.
2. Choose the Task file desired and set up Autosampler file from benchsheet (see attached).
3. Calibrate the instrument and verify calibration.
4. Analyze the samples. Review data for interferences and initial concentrations.
5. Print out a concentration and dilution report page. Review final data for errors in detection limits and dilution factors.
6. Enter final results into LIMS system.

Benchsheet and Final Report:

Use the following codes on benchsheets and final report pages:

chkstd	Check standard
blk	Calibration blank
RB (DATE) LIQ	Liquid prep reagent bland with date prepared.
RB (DATE) SOL	Solid prep reagent bland with date prepared
R (analytical #)	TCLP extract
(analytical #)r	Recoverable prep
(analytical #) FUS	Fusion
(analytical #) s	Soluble metals
(analytical #) DUP	Duplicated from the same prepped container
(analytical #) DUP PRE	Prepped in duplicate
(analytical #) +#	Spiked straight from the container
(analytical #) +# PRE	Spiked sample before prepped

Reviewed by: _____

Approved by: _____

SOP-970 - Metal Preparation

Determination of: Sample preparation of metals in waters, wastes, and toxicity characteristic leaching procedure (TCLP) extracts.

Scope/Purpose: To provide a standard method for the digestion/preparation of aqueous samples, EP, and mobility-procedure extracts, and wastes that contain suspended solids for analysis by GFAAA or ICP. The procedure is used to determine total metals.

Reagents: Concentrated nitric acid, concentrated hydrochloric acid

Equipment: Hot plates, black ribbon filter paper

References: Code of Federal Regulations Title 40, Part 136, Appendix C, Government Printing Office, Washington D.C. (20402), 1991.

Discussion:

This method describes the procedure for digesting a sample in order to make it suitable for analysis by either Inductively Coupled Plasma (ICP) or Graphite Furnace Atomic Absorption Spectroscopy (GFAAS). The sample is treated with nitric acid and heated to near dryness. It is then subjected to more nitric acid and is refluxed within its beaker. A final treatment of hydrochloric acid and deionized water dissolves any remaining residue. By treating the sample with nitric acid and heating, two things are accomplished. First, the acid and heat combine to break down most organic interferences within a sample's matrix. Second, metals will readily dissolve in nitric acid, leaching them away from elements that could form complex interferences. Due to the instruments inability to analyze solids, samples with a high turbidity or solids content must be digested.

There are two types of liquid samples digestion: total and recoverable. The total prep is a much more vigorous technique and is often needed to destroy meal complexes and leave them susceptible to atomization. Both procedures are described in detail in this document.

Specific Interferences

The analyst should be cautioned that the digestion procedures may not be sufficiently vigorous to destroy some metal complexes.

Precipitation could cause a lowering of concentrations and therefore less accurate results for some elements.

Total Metals Digestion Method:

1. Samples are evaluated upon receipt to determine if they require a digestion. Floating solids, discolored or murky samples, or smelly waters need to be digested prior to analysis. Samples not obviously in need of prepping should be evaluated by checking the turbidity. If the turbidity is > 1.0 NTU, then it should be digested.

2. Choose a measured volume of the mixed acid preserved sample appropriate for the expected level of metals. For most waters, use 100 milliliters (ml); for samples such as liquid sludges, use 10 ml. It is up to the discretion of the analyst as to the volume of sample used, but the consistency and amount of solids present are key indicators. Pipette the sample, if appropriate, otherwise use graduated cylinders. Be sure to rinse the graduated cylinder with a 1% acidified nitric acid deionized water solution after every use. if possible, wash the cylinder with a small portion of the sample before taking the volume to be used for digestion.

3. Transfer samples to a clean 250-ml beaker.

4. Mark the sample ID on the beaker and write the sample number and dilution on the benchsheet.

5. Add 3.0 ml of concentrated nitric acid.

6. Place the beaker on a hotplate set at 4 or 6 (depending on the hotplate you are using). Cover with a fluted watch glass and evaporate to near dryness without boiling or allowing the sample to go completely dry.

7. Remove and cool the beaker and add another 5.0-ml portion of concentrated nitric acid.

8. Cover the beaker with a small watch glass and return it to the hotplate. Heat so that a gentle reflux action occurs. The 3 or 5 setting on the hotplate should be sufficient.

9. Continue heating and adding acid until the digestate is light in color and does not change in appearance.

10. Again, evaporate to near dryness and cool the beaker.

11. Add 5 ml of concentrated hydrochloric acid and 15 ml of deionized water per 100 ml of final solution and warm the beaker for 15 minutes to dissolve any precipitate or residue resulting from evaporation. If the samples requests furnace lead, do not add the HCL as it negatively interferes with the GFAAS analysis of lead. If the sample in question is being spiked for silver, use an excess of HCL. An additional 5 ml should be sufficient. This helps prevent the spike from precipitating, which will improve the recovery.

12. Cool beaker and wash down the walls and watch glass with deionized water.

13. Gravity-filter the sample through Black Ribbon filter paper to remove any insoluble material that could clog the nebulizer on the ICP. The sample is to be filtered into a 100-ml volumetric flask and diluted to the mark using deionized water.

14. Pour the digested sample into a 120-ml plastic sample cup and mark the sample ID, dilutions, and date in permanent pen.

Recoverable Metals Digestion Method

1. Choose a measured volume of the well-mixed, acid-preserved sample appropriate for the expected level of elements and transfer to a 250-ml beaker. Be sure to rinse the graduated cylinder with acidified deionized water and a small portion of the samples being digested prior to use.

2. Mark the sample ID on the beaker with the grease pencil and write the sample number and dilution on the benchsheet.

3. Add 1.0 ml of concentrated nitric acid and 5.0 ml of concentrated hydrochloric acid. Hydrochloric acid is an interferant for low level lead analysis and should be left out of samples requesting furnace recoverable lead.

4. Place the sample on a hot plate on 4 and evaporate without boiling to about 25 ml.

5. Remove the sample from the hot plate and cool.

6. Gravity-filter out any insoluble materials through Black Ribbon paper into a 100-ml volumetric flask. Again, do not filter if the sample requests zinc.

7. Be sure to rinse the sides of the beaker, filter paper, and funnel to get all the digestate.

8. Adjust the volume to 100 ml with deionized water.

9. Pour into a sample cup labeled to tell the analyst the sample ID, dilution, and date digested.

Quality Assurance/Quality Control (QA/QC):

1. A reagent blank needs to be prepared every time samples are digested using any of these methods. Measure 100 ml of deionized water into a 250-ml beaker and treat in the same manner as a sample requesting a total prep. This method is much more vigorous and detailed than the recoverable procedure; and if contamination was to be introduced during digestion, it would more likely show up in the total prep procedure.

2. Matrix spike/matrix spike duplicates (MS/MSDs) are to be done after every ten samples.

3. An LCS/fortified blank is analyzed per batch of 20 samples or every analysis day.

4. GFAAS spiking levels need to be 20 micrograms per liter (μg/L) for lead, copper, selenium antimony, thallium, chromium, arsenic, and 2.0 μg/L for cadmium and silver. A spike mix may be prepared in advance to simplify the spiking procedure. This mix can be made by pipeting 1.0 ml. of 1,000 milligrams per liter (mg/L) stock standard of each of the metals needing 20 μg/L into a 1-liter volumetric, preserving with a 10.0 ml of nitric acid, and recording the mix into the reagent logbook. The mix for cadmium and silver is made by pipeting 1.0 ml of a 1,000-ml stock standard into a 1-liter volumetric and making a second dilution by pipeting 10.0 ml of the 1.0 mg/L working standard into a 100-ml volumetric. Preserve the mix with 1.0 ml of concentrated nitric acid and record all reagents and dilutions into the reagent logbook. From this point, 2.0 ml of the two standards are pipetted into the 250-ml beakers containing equal portions of the sample. They can now be digested using the prep procedure requested for the sample. After the transfer to the 100-ml volumetric and diluting the samples to mark with deionized water, the final concentration of the spike will be 20.0 μg/L for Pb, Cu, Se, Sb, T1, Cr, As, and 2.0 μg/L for Cd and Ag.

5. Spiking for ICP metals is done at much higher concentrations. Most metals can be spiked with the QC Standard 23, which can be purchased from several vendors. The Standard contains 100 mg/L of Sb, As, e, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, Sn, Li, P, S, V, and Zn. The spike is made by pipeting 1.0 ml of the Standard into the beakers containing the sample. The exact volume used is dependent upon the expected concentrations of the samples and request of the ICP analyst. A second Standard is used to spike for most of the remaining metals. QC Standard 7, which can also be purchased from several vendors, contains 2,000 mg/L K, 100 mg/L Al, Ba, B, Ag, and Na, and 50 mg/L Si. Pipet no more than 1.0 ml of this Standard into the sample. The reason for this is that it contains Ag, which shows a very poor spike recovery if a larger amount is used. A digestion note, use excess HCL in spikes containing silver, if possible. It seems to aid in the recovery of the spikes. The Si, Na, K, Ca, Mg, and Fe spike concentrations are too low using the volumes stated above. They need to be supplemented to produce a concentration that is discernible from the amount of analyte in the sample. Ca, Mg, Na, and K should be spiked at approximately 50 mg/L, and Fe and Si should be spiked at about 10 mg/L. Spike levels will vary depending on what concentrations are to be expected in the sample being spiked.

6. If a sample needs to be spiked with a metal not contained in either of the mixed Standards used, they can be done separately at a concentration dictated by the ICP analyst.

Batches:

Samples are give a batch number in order to track the metal digestions in our LIMS computer system. These numbers are written on the upper right corner of the benchsheet and entered when the dilutions are entered into the computer. Batch numbers are changed at the first of the month, and that number is used throughout the month for all liquid metal preps.

Glassware Cleanup and Storage:

1. After use, wash glassware using hot water and a concentrated general laboratory soap.
2. Immerse the washed glass into a nitric acid solution and allow them to soak for 24 hours.
3. The acid solution should be 5A10% nitric acid in order to effectively clean all surfaces of the glass.
4. Rinse the glass with distilled water and allow to dry.
5. In order to prevent contamination, label separate glassware for use with contaminated and dirty samples. Keep the two sets of glassware separated through the entire prepping and cleaning process.

Special Note: Separate glassware for metals from that used for total sulfur or sulfate. High concentrations of barium used here would contaminate the metals glassware.

Reviewed by: _____

Approved by: _____

SOP-980 - Soil Metal Preparation

Determination of: Metals in sediments, cakes, and soils.

Scope/Purpose: To provide a procedure for the acid digestion of solid samples for analysis by Inductively Coupled Plasma (ICP) and Graphite Furnace Atomic Absorption Spectroscopy (GFAAS) techniques.

Reagents: Reagent grade concentrated nitric acid. Reagent grade concentrated hydrochloric acid. Reagent grade 30% hydrogen peroxide.

Equipment: Hot plates, analytical balance capable or reading to 0.01 gram, Black Ribbon filter paper.

References: Test Methods for Evaluation Solid Waste Laboratory Manual, SW-846, Volume 1A, Chapter 3, Method 3050A, Revision 1, July 1992.

Methods for the Determination of Metals in Environmental Samples, EPA-600/4-91-010, Environmental Monitoring Systems Laboratory Office of Research and Development, US Environmental Protection Agency, June 1991.

Discussion:

A representative amount of a sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed in hydrochloric acid. These treatments serve to destroy meal complexes with organic substances and leach the metals away from insoluble materials. The insolubles are filtered out and what is left is a strongly acidic liquid that may be analyzed using either the ICP or GFAAS techniques.

Special Interferences:

The analyst should be cautioned that the digestion procedure may not be sufficiently vigorous to destroy some of the metal complexes.

Not using a homogeneous sample could cause less accurate results. It could also cause a large deviation between duplicate samples.

Precipitation could cause a lowering of concentrations, and therefore, less accurate results for some elements.

Solid Test Metals Digestion Method:

1. Mix the sample to achieve homogeneity using a spatula.

2. Weigh out to the nearest 0.01 gram (g): 3 g of soils and sediments and 1 g of ashes and sludge cakes. Transfer to a 250-milliliter (ml) beaker.

3. Record the exact weight of the sample on the benchsheet as they are weighed out.

4. Add 10 ml of concentrated nitric acid, cover with a watch glass, and place the beaker on the hot plate and heat to 95° C. A setting of 2 should be sufficient. Reflux for 15 minutes.

5. Cool sample, add 10 ml of concentrated nitric acid, cover with a watch glass, and allow the sample to reflux for 30 minutes without boiling. If the sample bumps, turn down the heat and use a glass bump rod to prevent the spattering.

6. Remove the watch glass and allow the solution to evaporate to approximately 5 ml. Do not dry the sample.

7. After cooling the sample, add 2 ml of water and 3 ml of 30% hydrogen peroxide. Cover the sample with the watch glass and return it to the hot plate. Extreme care needs to be taken, as the peroxide will react violently in the beaker and may foam over.

8. Allow the sample to stop effervescing and cool the beaker. Continue to add the peroxide in small portions until the effervescence is minimal or the sample appearance is unchanged. Do not add more than a total of 10 ml of peroxide.

9. Add 5 ml of concentrated hydrochloric acid and 10 ml of deionized water and replace the watch glass and return the beaker to the hot plate. Allow the sample to reflux for 15 minutes; cool the sample.

10. Filter the sample through the Black Ribbon filter paper into a 100-ml volumetric flask. Be sure to thoroughly rinse the beaker, sample, filter paper, and funnel with deionized water.

11. Dilute to the mark with deionized water.

12. Mix the sample and pour it into a sample container labeled with the sample ID, date, and weight of sample digested.

Solid Sample Preparation - Total Recoverable Elements:

1. Thoroughly mix the sample to achieve homogeneity.

2. Accurately weigh 3.0 g of the sample and transfer it to a 250-ml beaker.

3. Add 6 ml concentrated nitric acid and 6 ml of concentrated hydrochloric acid.

4. Cover with a watch glass and heat on a hot plate for 30 minutes. A setting of 2 is generally hot enough to reflux the sample without boiling it.

5. Remove from hot plate and cool sample.

6. Filter the sample through Black Ribbon filter paper into a 100-ml volumetric. Be sure to thoroughly rinse the beaker, sample, filter paper, and funnel

7. Dilute to 100 ml using deionized water.

Quality Assurance/Quality Control (QA/QC):

1. A reagent blank needs to be prepared every time solid samples are digested. Treat a beaker with 10 ml of deionized water as you would a normal sample.

2. Duplicates need to be done at a frequency of 10%.

3. Matrix spike/matrix spike duplicates (MS/MSDs) are to be done at a frequency of 5%.

4. GFAAS spiking levels for Pb, Cu, Se, Tl, Cr, and As are 20.0 micrograms per liter (µg/L). Spiking concentrations for Cd and Ag are 2.00 µg/L. A spike mix may be prepared in advance to simplify the spiking procedure. The mix for the 20.0-µg/L metals is made by pipeting 1.0 ml of 1,000-milligrams per liter (mg/L) of each element into a 1-liter volumetric and diluting to the mark with deionized water. The mix for Cd and Ag is prepared by pipeting 1.0 ml of Standard into a 1,000-ml flask and diluting to the mark with deionized water. Prepare a second dilution by 10 ml of the working Standard into a 100-ml

volumetric flask and diluting to the mark with deionized water. From this point, 2.0 ml can be pipetted from each of the two Standards and added to the beaker containing the solid sample.

5. Spiking the ICP metals is done at much higher concentrations. Most metals can be spiked with the QC Standard 23, which can be purchased from different vendors. The Standard contains 100 mg/L of Sb, As, Be, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, Se, Tl, Ti, V, and Zn. The spike should be made by pipeting 5.0 ml of the Standard into the beakers holding the solid sample. The exact volume may vary depending on the expected concentration of the sample and the requests of the ICP analyst. A second Standard is used to spike for most of the remaining metals. QC Standard 7 is also purchased from Spex Industries. This Standard contains 1,000 mg/L K, 100 mg/L A1, Ba, B, Ag, Na, and 50 mg/L of Si. Use no more than 1 ml of this Standard as it contains Ag, which shows very poor spike recoveries at higher concentrations of spikes on the ICP. As a digestion note, use excess hydrochloric acid in silver spikes; it seems to improve recovery. An additional 5 ml should be sufficient. The Si, Na, K, Mg, and Fe spike concentrations are too low using these volumes. The concentration of these metals in many samples is normally quite high. Pipet 10 ml of 1,000-mg/L stock Standard into the beaker to insure an amount of spike that the ICP can detect apart from the samples concentration.

6. If a sample needs to be spiked with a metal not contained in either of the Standards normally used, they can be done separately at a concentration dictated by the ICP analyst.

7. Record all spike dilutions on the benchsheet and on the sample container.

Batches:

Samples are given a batch number in order to track the metal digestions in our LIMS computer system. These numbers are written on the upper right corner of the benchsheet and entered when the dilutions are entered into the computer. Batch numbers are changed at the first of the month, and that number is used throughout the month. There are two different batch numbers issued at the beginning of the month: one for liquid preps and one for solid preps.

Glassware Cleanup and Storage:

1. After use, wash glassware using hot water and a concentrated laboratory soap.

2. Immerse the washed glassware into a nitric solution and allow them to soak for 24 hours.

3. The acid solution should be 5A10% in strength in order to effectively clean all surfaces of the glass.

4. Rinse the glass with distilled water and allow to dry.

5. In order to prevent contamination, label separate glassware for use with solid samples. Keep this set separated from the liquid prep glassware throughout the entire prepping and cleaning process.

Special Note: Separate glassware for metals from glassware used for total sulfur or sulfate. High concentrations of barium used in those procedures would contaminate the metals glassware.

Reviewed by: _____

Approved by: _____

Wisconsin Department of Natural Resources
STS Project No. 4-27393E

Appendix C

Groundwater and Surface Water Sampling and Analysis Plan

Table of Contents

Wisconsin Department of Natural Resources
STS Project No. 4-27393E
October 22, 2004

Groundwater and Surface Water Sampling and Analysis Plan
CD Besadny Wildlife Area - Kewaunee Marsh Arsenic-Impacted Site
Town of Pierce, Kewaunee County, Wisconsin

1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) outlines the anticipated scope and procedures for the monitoring component of a site investigation that will be implemented to characterize the distribution of arsenic in soil, sediment, surface water, and groundwater in an arsenic-impacted site within the CD Besadny Fish and Wildlife Area in the Kewaunee Marsh. The purpose of the site investigation is to facilitate environmental remediation planning and further evaluate the feasibility of cleanup alternatives.

The site is located in the SW 1/4, Section 7, T23N, R25E, town of Pierce, Kewaunee County, Wisconsin, approximately 1 mile northwest of State Highway 42 (STH 42) and 1/4 mile east of County Trunk Highway E, along a Fox Valley & Western (FV&W) Railroad corridor formerly known as the "ferry yard lead." The site is approximately 1,000 feet northwest of the Kewaunee River.

The Wisconsin Department of Natural Resources (WDNR), with financial assistance through the Great Lakes National Program Office (GLNPO), authorized preparation of this SAP to support the environmental assessment of the Kewaunee Marsh project site. The scope of this SAP is limited to the groundwater monitoring procedures; the tasks partially funded by the GLNPO grant.

2.0 BACKGROUND INFORMATION

The CD Besadny Wildlife Area consists of over 22,000 acres of state-owned property including wetlands, farmland, forest, and stream habitats located in Pierce Township, Kewaunee County, Wisconsin. In August 1992, the WDNR was notified that a wetland area within the wildlife boundaries was either devoid of vegetation or showed signs of significant vegetative stress. Subsequent sampling indicated elevated concentrations of arsenic in soil and groundwater samples collected from within the wetland area.

The impacted area is located in the SW 1/4, Section 7, T23N, R25E, town of Pierce, Kewaunee County, and is approximately 1 mile northwest of STH 42 along a former railroad track once operated by FV&W Railroad. Investigation efforts by both the WDNR and the railroad suggest that subsurface degradation is a result of a surface release of sodium arsenate, which presumably occurred sometime between 1938 and 1950. During the 1930s and 1940s, sodium arsenate was used as an insecticide on cherry-producing orchards of Door County, and the surface release may have been the result of a train derailment, which reportedly occurred in this area. Since initial WDNR sampling in October 1992, the arsenic-impacted wetland area was characterized by collecting and analyzing numerous soil, groundwater, and surface water samples. Site assessment and corrective action efforts included the following:

- A preliminary assessment completed in 1994 by STS Consultants, Ltd. (STS) on behalf of FV&W Railroad. The preliminary assessment included completing shallow soil borings to a maximum depth of 1.5 feet. A subsequent investigation included collecting surface water samples, groundwater samples from the shallow monitoring wells, sediment samples, and soil pore water.
- Interim action performed in 1996 consisted of placing a textile/wood chip cover over visibly impacted areas of the marsh and enclosing the area with a security fence. The interim cover reduced direct contact risk over an area of about 3.25 acres within the 15-acre fenced area. Before the cover was constructed, shallow groundwater monitoring points were installed near the cover area, and a staff gauge was installed nearby in the Kewaunee River. Groundwater and river elevations were measured, and groundwater samples were collected from the monitoring points. Surface water samples were also collected from the Kewaunee River. Monitoring Points MP-1 through MP-4 were installed downgradient of the cover area within the security fence; and Monitoring Points MP-5 and MP-6 were installed outside the fence, sidegradient of the cover.
- Soil, surface water, and groundwater sampling completed by the WDNR in 1996 and 1997.

- February 1997: Groundwater and surface water modeling was done to estimate the environmental fate and migration potential of arsenic. The model indicated that the transport of arsenic in the groundwater at the site is very slow, with the model predicting that the maximum concentration of arsenic in the groundwater would reach the Kewaunee River in approximately 2,800 years. The groundwater model generally simulated dissolved arsenic migration based on estimated sorption through the saturated organic soil. A (HydroCAD) surface water model was utilized to evaluate transport of the arsenic in stormwater. Results indicated that the maximum stormwater arsenic concentration was 28.3 micrograms per liter (μg/L); the highest downstream arsenic concentration calculated was 15.6 μg/L. The surface water model predicted arsenic migration in surface water using the ratio of arsenic mass and total runoff from regional sub-basins.

- April 2000: A baseline risk assessment was completed by the WDNR for the Kewaunee Marsh site. The WDNR published its Baseline Ecological Risk Assessment (BERA) of the Arsenic Contaminated Wetland Associated with the CD Besadny Fish and Wildlife Area and the Kewaunee River. The BERA was conducted to determine the present and future risks to wildlife, birds, and aquatic resources from exposures to arsenic in the soil, sediment, groundwater, and surface water following implementation of the interim action at the site. The BERA also documented the degree of uncertainty and quality of data available in performing the risk assessment and indicated that further investigation of soil, sediment, groundwater, and surface water is warranted to determine if the interim action cover is sufficient to protect the environment and public health.

- In May and June 2001, the WDNR Bureau of Watershed Management conducted additional investigation at the site. Ten groundwater monitoring wells were installed at the site, and ten surface water samples and ten soil/sediment samples were collected. In addition, the WDNR installed a continuous water level tracker on the former railroad bridge located adjacent to the site.

- 2002 and 2003: Environmental monitoring of sediment, soil, groundwater, and surface water including installation of additional groundwater monitoring wells, and piezometers was completed. Results reported in November 2003 concluded the following:

  - Groundwater quality exceeds ES concentrations established under Chapter NR 140, Wisconsin Administrative Code.

  - Arsenic concentrations in surface water samples obtained from two primary drainage sloughs exceed toxic threshold criteria established under Chapter NR 105, Wisconsin Administrative Code.

  - Arsenic concentrations in surface and near-surface soils represent a direct contact risk to human health beyond the limits of the interim cover and security fence.

  - Arsenic-impacted soil represents a continuing threat to groundwater quality.

  - Transport of arsenic appears to be accelerated by the reductive dissolution of iron hydroxides.

3.0 GROUNDWATER SAMPLING

3.1 Program Summary

The scope of the groundwater investigation will include sampling existing monitoring wells for the parameters and frequency summarized below.

Program Summary

Sampling Point	Frequency	Parameter Analysis
MW02-3, 3i, 3d MW02-4, 4i, 4d MW02-5, 5i MW02-6, 6i MW02-7, 7i MW02-8 GW01-2 GW01-3 GW01-5 GW01-6 GW01-8 MW04-9 MW04-10 MW04-11 MW04-12 MW04-13	Quarterly	Arsenic (by GFAAS) Sulfate Sodium Iron Nitrate Dissolved Oxygen (DO) Field pH, Conductivity, Temperature, and Redox Potential
MW02-1, 1i, 1d MW02-2, 2i MW02-7d MW02-8i GW01-1 GW01-4 GW01-10	Annually	Arsenic (by GFAAS) Sulfate Sodium Iron Nitrate DO Field pH, Conductivity, Temperature, Redox Potential

3.2 Equipment

Equipment used for groundwater sampling includes:

- Electric water level indicator
- Disposable or dedicated Teflon or polyethylene bailer
- Closed Flow-thru Cell

- Down-hole water quality probe, which measures pH, temperature, conductivity, and redox potential (Eh)
- Chemetrics Ampoules L-7512 for dissolved oxygen.
- Pre-filtration jugs
- Peristaltic pump and tubing
- 0.4-um filters
- Field log book and field forms/logs
- Personal protective equipment
- Tap water, distilled water, and Alconox®
- 5-gallon pails
- Sample containers and preservatives
- Chain of Custody forms
- Sample labels
- Indelible marking pen
- Coolers and ice
- Nylon rope

3.3 Procedures

To prevent potential contamination during transportation to the site, sampling equipment will be stored in clean plastic containers or wrapped with aluminum foil. A new sheet of clean plastic sheeting will be used at each sampling location to provide a clean surface on which to place sampling equipment during sample collection activities.

Depending on recharge capabilities, each well will be purged of at least four well volumes. Water quality measurements will be taken using a closed flow-thru cell during low-flow well purging. Probes for pH, DO, conductivity, temperature, and redox potential inserted into the closed flow-thru cell will collect measurements simultaneously during low-flow purging. Groundwater samples will be collected after passing through a closed flow-thru cell. Groundwater samples may also be collected by dedicated disposable bailer in wells which purge dry and do not recharge at a rate sufficient to sustain low-flow sampling, or in the case of inclement winter weather conditions where a flow-through cell may freeze. Time between the completion of purging and sample collection will not exceed 24 hours, unless the rate of recovery in the well requires more time for groundwater to collect in the well.

The standard procedure for Low-flow purging, sampling, and monitoring of indicator parameters for stability in a closed flow-through cell are as follows:

Low Flow Purging

- The intake tubing of the flow-through cell is lowered to the middle of the well screened section. The discharge tube of the flow-through cell is connected to the pump.
- Closed flow-through cell
  - Confirm that probe censors are completely submerged in the water during use.
  - Check the tubing for bubbles and avoid taking readings if bubbles are present in the tube.
  - Avoid exposing the flow-through cell to extreme heat and sun in the summer and freezing temperatures in the winter.
- Set up and calibrate all indicator parameter instruments and place each probe into its respective port or the closed flow-through cell.
- Set the pump controller to the desired purging rate (<1 L/min. or 0.26 gpm). Note: Do not use a valve to reduce the flow from a pump; valves can cause an "orifice" effect that can cause sample agitation and alteration.
- Record the "purging time start," and start purging the well at a rate of 1 L/min or less. During purging, the water level in the well should not decrease significantly and stabilize after purging for a few minutes. If the water level continues to decline while purging, the pumping rate should be decreased. Record the "purging flow rate" as an average. Use a graduated beaker, cylinder, calibrated bucket, or other device to measure the flow rate while purging and sampling.
- Purge the well until the readings for indicator parameters listed above vary within ±10% over three or more consecutive readings, spaced approximately 2 minutes or approximately 0.5 well volume apart.
- Record the final three stable readings for each indicator parameter on monitoring documents.
- Record the "volume purged," "purging time stop," "purged dry (Y/N)," and any problems purging.

Sample Collection

- Open only one sample container or one set of sample containers immediately before filling. Preserve samples within 15 minutes of collection and immediately place on ice.
- Minimize the contact of extraneous contamination with sample containers and equipment (i.e., engine fumes, marking pen fumes, bug spray, etc.).
- Complete sample label information using a waterproof marker.
- Collect sample parameters in the following standard sampling order:
    1. Unfiltered samples for in-field water quality measurements (if not utilizing closed flow-through cell measurements)
    2. Non-filtered, non-preserved.
    3. Non-filtered, preserved.
    4. Filtered, non-preserved
    5. Filtered, preserved immediately
    6. Miscellaneous parameters
- Collect field equipment blank samples and duplicate groundwater samples per project specification.
- Tip the sample container at a slight angle and allow a slow steady stream of water to run down its inner wall. Hold the sample discharge tube close to the sample container but do not touch it.
- Immediately after filling a sample container, if not already done, add any required preservative, replace cap, add the label, and place the sample in a plastic bag on ice in a cooler.
- Record the sample time collected, barometric pressure, wind speed, and direction in the field notes.

3.4 Decontamination

All sampling devices will be decontaminated between wells. The closed flow-through cell will be decontaminated by rinsing with distilled water, and then passing additional distilled water through the system when connected to the pump. Decontamination of a non-disposable bailer, if utilized, will include an Alconox® and tap water wash, a tap water rinse, and distilled water rinse. The decontamination procedure for the down-hole water quality probe will not include the soap and water solution, but will merely be rinsed with tap water, then with distilled water. Peristaltic pump tubing will be decontaminated by running approximately 1 gallon of distilled water through the tubing between samples.

3.5 Quality Control

To evaluate the effectiveness of the decontamination process, field rinsate blanks will be collected during the sampling process. The closed flow-through cell or bailer will be cleaned with distilled water. Distilled water passed through the closed flow-through cell will be passed through the pump and filter and collected as a rinsate sample. A bailer, if utilized, will be rinsed and filled with distilled water, and the water subsequently transferred to a new filtration jug, passed through the peristaltic pump and a filter, and placed into laboratory supplied sample containers. Field blanks will be maintained with the other groundwater samples. Field blanks will be collected at a rate of one rinsate blank per day of groundwater sampling.

In addition to the field rinsate blanks, duplicate groundwater samples will be collected at a rate of 1 duplicate sample per 20 groundwater samples. The duplicate sample will be submitted for analysis to evaluate the precision of the laboratory analysis. The laboratory will also prepare and analyze quality control samples of samples per laboratory quality control procedures.

3.6 Documentation

Data collected and field observations made during groundwater sampling will be recorded on the following field documentation forms:

- Field Notes Form
- Groundwater Monitoring Form
- Calibration records for the water quality probe

4.0 SURFACE WATER SAMPLING

4.1 Program Summary

The scope of the surface water investigation will include sampling surface water for the parameters and frequency summarized below.

Sampling Point	Frequency	Parameter Analysis
Surface Water SW1 SW2 SW3 SW4 SW5 SW6	Semi-Annual	Arsenic (by GFAAS) Sulfate Sodium Iron Nitrate DO Field pH, Conductivity, Temperature, and Redox Potential

4.2 Equipment

Equipment used to collect surface water samples is as follows:

- Sample containers and preservatives
- Tube-type drum sampler, capable of collecting water from a column approximately 3 feet deep.
- Clean pre-filtration jugs
- Peristaltic pump and tubing
- 0.4-um filters
- Field notes form
- Personal protective equipment
- Down-hole water quality probe which measures pH, temperature, DO, conductivity, and redox potential (Eh)
- Decontamination supplies
- Stakes for marking sample locations
- Camera and film

4.3 Procedures

Surface water samples will be collected both from areas of standing water in the marsh and tributary sloughs in the study area. During collection activities, the buddy system will be employed for safety purposes.

Before samples are collected, water quality will be measured at the sampling location using a down-hole water quality probe. The probe will be inserted into the water at the area to be sampled. Readings for pH, temperature, conductivity, DO, and redox potential will be recorded.

Surface water samples collected from "casual" standing water in the marsh will be collected by dipping the pre-filtration jug directly into the surface water and allowing it to fill by gravity. Samples will be field-filtered using a peristaltic pump placed in the sample container and preserved. Samples collected from the drainage sloughs will be collected using a tube-type sampler ("drum thief coliwasa") if the depth of water in the sloughs allow. The advantage of the drum thief is it can collect a sample from a length of water column, not just from the surface or a particular discrete depth. These samples will also be transferred from the drum sampler into the pre-filtration jug to be filtered and preserved. If only shallow water (water less than 1.5 feet in depth) is present in a slough sampling location, collection of a water sample will follow the "casual" standing water sampling method.

4.4 Decontamination

Equipment expected to require decontamination for this task is the down-hole water quality probe and peristaltic pump tubing. The pre-filtration jugs, filters, and drum sampler tubes are designed to be disposable. The probe is decontaminated by rinsing it in tap water, then rinsing with distilled water. The tubing for the peristaltic pump will be decontaminated by running approximately 1 gallon of distilled water through the pump between sample locations. Personal protective equipment, namely gloves, will be changed between surface water sampling locations to avoid cross-contamination.

4.5 Quality Control

Sampling and analytical quality control (QC) will include duplicate samples in which two sample aliquots (in separate containers) are submitted to the laboratory for analysis. QC of both the field sampling procedures and analytical procedures will be measured by the degree of agreement of analytical results of the two samples. These analytical samples will be collected at a rate of one duplicate per sampling event. In addition to duplicate samples, a field blank will be collected using a clean unused tube and/or jug.

4.6 Documentation

Surface water sampling documentation will consist of the following:

- Field notes form
- Calibration records for the water quality probe

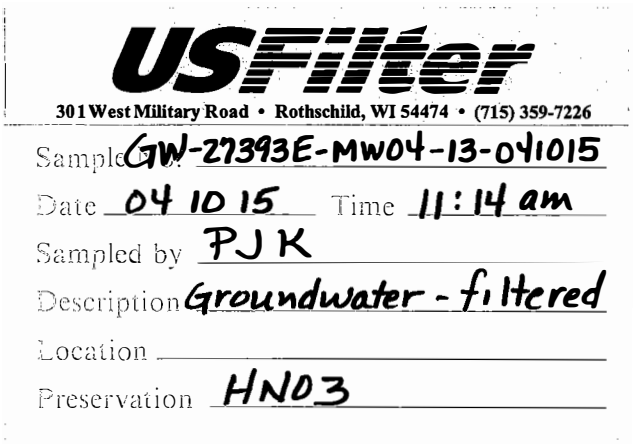
Surface water investigation field documentation will undergo a QC review during and after the completion of field activities. Upon completion of the field program, documentation will be relinquished to the Project Manager.

Wisconsin Department of Natural Resources
STS Project No. 4-27393E

Appendix D

Example Sample Label and Chain of Custody Form

Example of a Sample Label



CHAIN OF CUSTODY RECORD

№ 37349

RECORD NUMBER _____ THROUGH _____

Contact Person _____
Phone No. _____ Office _____
Project No. _____ PO No. _____
Project Name _____

Special Handling Request

- ☐ Rush
- ☐ Verbal
- ☐ Other

Laboratory _____
Contact Person _____
Phone No. _____
Results Due _____

Sample I.D.	Date	Time	Grab	Composite	No. of Containers	Sample Type (Water, soil, air, sludge, etc.)	Preservation		Field Data				Analysis Request	Comments on Sample (Include Major Contaminants)
							Y	N	PID/FID Ambient	PID/FID Sample	PH	Special Cond.		

Collected by:	Date	Time	Delivery by:	Date	Time
Received by:	Date	Time	Relinquished by:	Date	Time
Received by:	Date	Time	Relinquished by:	Date	Time
Received by:	Date	Time	Relinquished by:	Date	Time
Received for lab by:	Date	Time	Relinquished by:	Date	Time

Laboratory Comments Only: Seals Intact Upon Receipt? ☐ Yes ☐ No ☐ N/A

Final Disposition:	Comments (Weather Conditions, Precautions, Hazards):

Distribution: Original and Green - Laboratory Yellow - As needed Pink - Transporter Goldenrod - STS Project File
Instructions to Laboratory: Forward completed original to STS with analytical results. Retain green copy.

6/99cp10k

STS Consultants Ltd.
Consulting Engineers

CHAIN OF CUSTODY RECORD

№ 37349

RECORD NUMBER _____ THROUGH _____

Contact Person _____
Phone No. _____ Office _____
Project No. _____ PO No. _____
Project Name _____

Special Handling Request

- ☐ Rush
- ☐ Verbal
- ☐ Other

Laboratory _____
Contact Person _____
Phone No. _____
Results Due _____

Sample I.D.	Date	Time	Grab	Composite	No. of Containers	Sample Type (Water, soil, air, sludge, etc.)	Preservation Y	Preservation N	Field Data PID/FID Ambient	Field Data PID/FID Sample	Field Data PH	Field Data Special Cond.	Analysis Request	Comments on Sample (Include Major Contaminants)

Collected by:	Date	Time	Delivery by:	Date	Time
Received by:	Date	Time	Relinquished by:	Date	Time
Received by:	Date	Time	Relinquished by:	Date	Time
Received by:	Date	Time	Relinquished by:	Date	Time
Received for lab by:	Date	Time	Relinquished by:	Date	Time

Laboratory Comments Only: Seals Intact Upon Receipt? ☐ Yes ☐ No ☐ N/A

Final Disposition:	Comments (Weather Conditions, Precautions, Hazards):

Distribution: Original and Green - Laboratory Yellow - As needed Pink - Transporter Goldenrod - STS Project File
Instructions to Laboratory: Forward completed original to STS with analytical results. Retain green copy.

6/99cp10k

STS Consultants Ltd.
Consulting Engineers

SAMPLE LAB COPY

Wisconsin Department of Natural Resources
STS Project No. 4-27393E

Appendix E

WSLH Laboratory Quality Assurance Procedures

USFilter/Enviroscan Services Laboratory Quality Assurance Procedures

The State of Wisconsin

DEPARTMENT OF NATURAL RESOURCES

Hereby grants

Wisconsin Reciprocal Certification with EPA Region 5

under the provisions of ch. NR 149, Wisconsin Administrative Code to:

Wisconsin State Laboratory of Hygiene
2601 Agriculture Dr
Madison, WI 537077996

Lab ID Number: **113133790**

Issued Date: **August 24, 2004**

Expiration Date: **August 31, 2005**

for the following test categories:

* Safe Drinking Water
  - Asbestos
  - Arsenic
  - Barium
  - Beryllium
  - Cadmium
  - Cyanide
  - Chromium
  - Copper
  - Fluoride
  - Haloacetic Acids (five)
  - Mercury
  - Nitrate + Nitrite
  - N/P Pesticides by GC
  - Nickel
  - Nitrite
  - Nitrate
  - Lead
  - Antimony
  - Selenium
  - Thallium
  - Total Trihalomethanes
  - Volatile Organics

David Webb

Chief, Environmental Science Services

P. Scott Hassett

Secretary

Certification or registration by the State of Wisconsin is not an endorsement or guarantee of the validity of data generated by this laboratory. This certificate is valid unless revoked or suspended and supersedes all previous certificates.

The State of Wisconsin

DEPARTMENT OF NATURAL RESOURCES

Hereby grants

Wisconsin Certification under NR 149

under the provisions of ch. NR 149, Wisconsin Administrative Code to:

Wisconsin State Laboratory of Hygiene
2601 Agriculture Dr
Madison, WI 537077996

Lab ID Number: **113133790**

Issued Date: **August 24, 2004**

Expiration Date: **August 31, 2005**

for the following test categories:

* Oxygen Utilization
  - Biochemical Oxygen Demand
  - Carbonaceous BOD
* Nitrogen
  - Ammonia as N
  - Nitrite as N
  - Nitrate as N
  - Nitrate + Nitrite as N
  - Total Kjeldahl Nitrogen
* Phosphorus
  - Orthophosphate
  - Total Phosphorus
* Physical
  - Oil and Grease (HEM)
  - Oil and Grease (Freon)
  - Total Dissolved Solids
  - Total Solids
  - Total Suspended Solids
  - Total Vol. Suspend Solids
  - Total Volatile Solids
* General I
  - Alkalinity/Acidity
  - Bromide
  - Chlorophyll a
  - Color
  - Hardness
  - Silica
  - Silicate
  - Sulfite
  - Surfactants
* General II
* General II
  - Chloride
  - Cyanide
  - Chemical Oxygen Demand
  - Fluoride
  - Sulfide
  - Sulfate
* General III
  - Corrosivity
  - EP Toxicity
  - Waste Fingerprinting
  - Ignitability
  - Total Releasable Cyanide
  - Reactivity
  - Total Releasable Sulfide
  - TCLP
  - Total Organic Carbon
* Metals I
  - Silver
  - Aluminum
  - Arsenic
  - Boron
  - Barium
  - Beryllium
  - Calcium
  - Cadmium
  - Cobalt
  - Chromium (Total)
  - Copper
  - Iron
  - Chromium (Hexavalent)
* Metals I
  - Mercury
  - Potassium
  - Magnesium
  - Manganese
  - Molybdenum
  - Sodium
  - Nickel
  - Lead
  - Antimony
  - Selenium
  - Thallium
  - Vanadium
  - Zinc
* Organics; Purgeable
  - Purgeable Aromatics
  - Purgeable Halocarbons
  - Volatile Organics (VOCs)
* Semivolatiles by GC/MS
  - Base/Neutral/Acid Extract
* Liquid Chromatography
  - Aldehydes & Ketones by LC
  - PAHs by LC
* Pesticides
  - Triazines and Metabolites
* Organics; Organochlorine
  - PCBs
  - Organochlorine Pesticides
* Any Single Analyte
  - Sulfate in Drinking Water
* Any Single Analyte
  - Sodium in Drinking Water
* Effluent Toxicity
  - Acute Invertebrate Tox.
  - Acute Vertebrate Tox.
  - Chronic Invertebrate Tox.
  - Chronic Vertebrate Tox.

David Webb

Chief, Environmental Science Services

P. Scott Hassett

Secretary

Certification or registration by the State of Wisconsin is not an endorsement or guarantee of the validity of data generated by this laboratory. This certificate is valid unless revoked or suspended and supersedes all previous certificates.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 5
77 WEST JACKSON BOULEVARD
CHICAGO, IL 60604-3590

REPLY TO THE ATTENTION OF:

MAY 0 5 2004

WG-15J

Dr. Ronald Laessig
2601 Agriculture Drive
P.O. Box 7996
Madison, Wisconsin 53707-7996

Dear Dr. Laessig:

On April 13, 2004 we issued a certification to analyze drinking water for chemistry, radiochemistry and microbiology contaminants to your laboratory, pursuant to the National Primary Drinking Water Regulations as implemented by 40 CFR Parts 141 and 142. Two analytical procedures were inadvertently left off the list of certified methods that your laboratory is performing. This letter and the enclosed laboratory certification summary update your certification to include the Colilert method for total coliforms and E. coli and EPA method 552.2 for haloacetic acids. At this time, the certification for method 552.2 will remain provisional since the recent certification performed by the Florida Department of Health did not include this method.

Laboratory certification status (full certification, provisional certification and not certified) is granted based on the information provided in your application and the results of your participation in performance testing samples.

The United States Environmental Protection Agency grants to the Wisconsin State Laboratory of Hygiene, 2601 Agriculture Drive, Madison, Wisconsin 53707-7996, certification for the chemistry, microbiology and radiochemistry methods provided in Enclosure A.

If you have any questions or require clarification concerning this memo, please Mr. Patrick Churilla at (312)353-6175, by FAX at (312)886-6171 or by E-mail at churilla.patrick@epa.gov.

Sincerely yours,

Jo Lynn Traub
Director, Water Division

Enclosure

ENCLOSURE A

LABORATORY CERTIFICATION SUMMARY
Wisconsin State Laboratory of Hygiene
(April 29, 2004)

Parameters/Method	Certification Status
1. Volatile Organic Chemicals/ 524.2 * Benzene * Carbon tetrachloride * o-Dichlorobenzene * p-Dichlorobenzene * Dichloromethane * 1,1-Dichloroethene * 1,2-Dichloroethane * cis-1,2-Dichloroethene * trans-1,2-Dichloroethene * 1,2-Dichloropropane * Ethylbenzene * Chlorobenzene * Styrene * Toluene * Tetrachloroethylene * Trichloroethylene * 1,1,1-Trichloroethane * 1,1,2-Trichloroethane * 1,2,4-Trichlorobenzene * vinyl chloride * Xylene (total) * TTHM	Fully Certified
2. Atrazine / 507	Fully Certified
3. Metals-GFAA / SM3113 B * Antimony * Arsenic * Cadmium * Chromium * Copper * Selenium * Lead	Fully Certified
4. Metals-GFAA Platform / 200.9 * Thallium	Fully Certified
5. Metals-ICP/AES / 200.7 * Barium * Beryllium * Copper * Nickel	Fully Certified
6. Mercury / 245.1	Fully Certified
7. TotalCyanide /SM4500 CN-E	Fully Certified
8. Fluoride / 380-75WE	Fully Certified
9. Nitrate /353.2	Fully Certified
10. Nitrite /SM4500 NO2-B	Fully Certified
11. Nitrate/Nitrite / 353.2	Fully Certified
12. Total Coliforms /SM9221-B	Fully Certified
13. Total Coliforms / SM9221-D	Fully Certified
14. Total Coliforms / SM9222-B	Fully Certified
15. Fecal Coliforms / SM9221-E	Fully Certified
16. Total Coliforms + E. coli / Colisure	Fully Certified
17. Total Coliforms + E. coli / Colilert	Fully Certified

Parameters/Method	Certification Status
18. Total Coliforms + E. coli / E*Colite	Fully Certified
19. Total Coliforms + E. coli / Readycult	Fully Certified
20. Asbestos / 100.1	Fully Certified
21. Gross Alpha, Gross Beta / 900.0	Fully Certified
22. Gamma Emitters / 901.1	Fully Certified
23. Radium 226 / 903.1	Fully Certified
24. Radium 228 /904.0	Fully Certified
25. Uranium / 908.0	Fully Certified
26. Iodine-131 / 901.1	Fully Certified
27. Strontium 89, Strontium 90 / 905	Fully Certified
28. Tritium / 906.0	Fully Certified
29. Haloacetic acids / 552.2	Provisionally certified

Quality Assurance Procedures & Policies

Environmental Health Division

Environmental Sciences Section

Biomonitoring Department
Inorganic Chemistry Department
Organic Chemistry Department
Radiochemistry Department
Water Microbiology Department

Wisconsin State Laboratory of Hygiene
University of Wisconsin
2601 Agriculture Dr.
Madison, Wisconsin 53707-7996
Phone: (608)-224-6200
Fax: (608)-224-6213

Revision 3.3 – Oct., 2003

William C. Sonzogni, Ph.D. Date
Division Director
Environmental Health Division
Wisconsin State Laboratory of Hygiene

George T. Bowman Date
Chemistry Management Supervisor
ESS — Inorganic Chemistry Department

Steve Geis Date
Chemistry Supervisor
ESS — Organic Chemistry & Biomonitoring Department

Lynn West Date
Chemistry Supervisor
ESS — RadioChemistry Department

Sharon Kluender Date
Microbiology Supervisor
ESS — Water Microbiology Department

Susan D. Hill Date
Chemist Advanced / QA Officer
ESS — Inorganic Chemistry Department

Matthew J. Roach Date
Chemist Advanced / QA Officer
ESS — Organic Chemistry Department

Figure 1.1 — NELAC Concordance — Mandatory Elements

MANDATORY ELEMENTS & NELAC REFERENCE		QUALITY MANUAL REFERENCE
5.5.2	Title Page	Page 1
5.5.2(a)	Quality Policy Statement, Objectives, & Commitments by top management	Chapter 2 - All
5.5.2(b)	Organization & Management Structure, organizational charts, relationship to parent organization	Chapter 3 - All See Chapter 20 for Organizational Charts
5.5.2(c)	Relationship between Management, Technical Operations, Support Services, & Quality System	Chapter 3 - All
5.5.2(d)	Procedures for Control & Maintenance of Documentation; Document Control System	Chapter 8 (8.2.2, 8.3.2, 8.4.2, 8.5.6, 8.6.2)
5.5.2(e)	Job Descriptions of Key Staff, plus reference to job descriptions of other staff	Chapter 3 - All
5.5.2(f)	Identification of Approved Signatories for the Laboratory (e.g., for laboratory test reports)	Pages 1 & 3
5.5.2(g)	Procedures for Achieving Traceability of Measurements	Chapter 14
5.5.2(h)	List of All Test Methods, under which accredited testing is performed	Chapter 17
5.5.2(i)	Procedures for Reviewing New Work & Ascertaining Appropriateness of Facilities & Resources prior to commencing new work	Chapter 9 - All
5.5.2(j)	Reference to Calibration and/or Verification Test Procedures Used	Chapter 11 & 12
5.5.2(k)	Procedures for Handling Submitted Samples	Chapter 10 (10.1, 10.2.2, 10.3.2, 10.4.2, 10.5.2, 10.5.3) Also Chapter 4 for Building Security
5.5.2(l)	Reference to Major Equipment, Reference Standards, Facilities, & Services used in conducting tests	Chapter 11 - All

MANDATORY ELEMENTS & NELAC REFERENCE		QUALITY MANUAL REFERENCE
5.5.2(m)	Reference to Procedures for Calibration, Verification, & Maintenance of Equipment	Chapter 11 & 12 - All
5.5.2(n)	Reference to Verification Practices (e.g., proficiency testing, interlaboratory comparisons, use of reference materials)	Chapter 12 - All
5.5.2(o)	Procedures Followed for Feedback & Corrective Action when testing discrepancies are detected or when departures to documented policies & procedures occur	Chapter 12 - All
5.5.2(p)	Management Arrangements for Permitting Departures from Documented Procedures or Standard Specifications	Chapter 12 - All
5.5.2(q)	Procedures for Dealing with Complaints	Chapter 5 - All
5.5.2(r)	Procedures for Protecting Confidentiality & Proprietary Rights (including national security)	Chapter 7 7.2.3
5.5.2(s)	Procedures for Audits & Data Review	Chapter 12
5.5.2(t)	Procedures for Establishing that Personnel Are Adequately Experienced and/or Receive Any Needed Training	Chapter 6 for Hiring Chapter 7 for Training
5.5.2(u)	Procedures for Training Personnel in Their Ethical & Legal Responsibilities (including potential penalties & punishments)	Chapter 7 7.2.3
5.5.2(v)	Reference to Procedures for Reporting Analytical Results	Chapter 8 (8.2.1.2, 8.3.1.3, 8.4.1.3, 8.5.4, 8.6.1.2)
5.5.2(w)	Table of Contents and Applicable Lists of References, Glossaries, & Appendices	TOC - page 7 Chapter 19 - Glossary Chapter 15 & 16 for References.

Figure 1.2 — NELAC Concordance — Optional Elements

OPTIONAL ELEMENTS & NELAC REFERENCE		QUALITY MANUAL REFERENCE
5.5.1(c)	Policies, Objectives, & Commitment to Accepted Laboratory Practices & Quality of Testing Services	Chapter 2 - All
5.5.3.2	Procedures for Conducting the Annual Quality System Review by Management	Chapter 12 (12.1.1)
5.9.4.2.1(i)	Procedures for Determining the Number of Points for Establishing Initial Instrument Calibrations	Chapter 12
5.10.1.1	Procedures for Assessing Data Integrity, Corrective Actions, Handling Complaints. Test methods, & Other Phases of Current Laboratory Activities	Chapter 5 - Complaints Chapter 8 - Corrective Actions Chapter 10 & 12 - Data Integrity
5.10.3	Procedures for Obtaining Representative Subsamples	Chapter 10 (10.2.3, 10.3.3, 10.4.3, 10.5.3)
5.10.4(a)	Procedures to Check & Correct Data for Transcription and Calculation Errors	Chapter 8 (8.2.1.2, 8.3.1.2, 8.4.1.2, 8.5.3, 8.6.1.2)
5.10.4(b)	Procedures to Review & Evaluate All Quality Control Measures before data are reported	Chapter 8 & 12
5.10.5	Procedures for Purchasing, Receiving, & Storing Materials used in technical operations	Chapter 11 for Purchasing (11.1.4.1) Chapter 14 for Traceability
5.11.1(a)	System for Uniquely Identifying Items (i.e., samples) to be tested	Chapter 10 (10.1, 10.2.2, 10.3.2, 10.4.2, 10.5.2)
5.11.2	Sample Acceptance Policy	Chapter 10
5.11.2(f)	Procedures Followed When Samples Show Signs of Damage or Contamination	Chapter 10 (10.1, 10.2.2, 10.3.2, 10.4.2, 10.5.2)

OPTIONAL ELEMENTS & NELAC REFERENCE		QUALITY MANUAL REFERENCE
5.11.4	Procedures to Avoid Deterioration, Contamination, or Damage to Samples during storage, handling, preparation, & testing	Chapter 10
5.11.5	Procedures for Disposal of Samples, Digestates, Leachates, & Extracts	Chapter 10 (10.1, 10.2.4, 10.3.4, 10.4.4, 10.5.4)
5.12	Laboratory Record System	Chapter 8 - All
5.12.2(d)	Laboratory Record Management System	Chapter 8 - All
5.13(f)	Procedures for Preserving Confidentiality during Electronic or Electromagnetic Transmission of Test Results	Chapter 11 (11.1.2.1)
5.15(b)	Procedures to Ensure that Purchased Equipment, Materials, & Services Meet Specified Requirements	Chapter 11 (11.1.4)
D	Procedures for Development of Quality Control Acceptance/Rejection Criteria	Chapter 13 - All

Table of Contents

Figures and Tables

1. Manual Organization and Maintenance

1.1. Historical Perspective

The Departments that are covered under this manual have, over the years, maintained various documents that have served as de facto Quality Assurance Manuals. In November of 1998 an attempt was made to construct a NELAC compatible Quality Assurance Manual for the Inorganic and Organic Chemistry Departments (Revision 2.0). That manual was revised in May of 1999 (Revision 2.1).

(Revision 3.0). That revision was developed during the laboratory's NELAP application process. It was an attempt to cover all of the Departments in the laboratory that would be accredited under NELAP. Those departments are Biomonitoring (Whole Effluent Toxicity), Inorganic Chemistry, Organic Chemistry, Radiochemistry, and Water Microbiology.

The current document (Revision 3.3) is a result of the third annual review process required under the NELAC standards.

1.2. Structure

The manual is currently constructed using Microsoft Word 97. As software is updated the manual format may change as necessary.

1.3. Organization

The manual consists of a title page, list of signatories, NELAC concordance, table of contents, and the various sections. Chapters which are considered general in nature (i.e., apply to the laboratory as a whole), are intermingled with those which are more department specific. Where a chapter is department specific, it will be broken into five sections named for each department. See the example below:

Each department sub-chapter will contain the necessary information covering that department.

A copy of the manual will be kept by the Laboratory Director and will be available electronically to all staff. Other copies will be made available in hardcopy form to the various departments.

1.4. Maintenance

Annually each department will review their sections of the manual for any necessary changes. In addition, the Quality Assurance Officers will review the entire manual. Any necessary changes will be submitted to the QA officer, who will then update the manual and publish a new revision (if necessary).

Although the manual will be updated at a minimum on an annual basis, changes may occur at anytime. Major changes will result in a new revision of the Manual. Each new revision will have a new revision number, an effective date, and will list the Manual revision it is replacing. In general, if minor semantic or typographical changes are made a new revision number will not be used.

Old revisions will be labeled with the appropriate dates and archived. The electronic version of the manual will reside on the shared server used by the EHD. Specifically it will be located at M:\EHD\ESS(4900)\Admin\QA\QA Manual\<filename>

2. Quality Policy

It is the intention of the management and staff of the Wisconsin State Laboratory of Hygiene to be a world class public and environmental health laboratory. The Environmental Sciences Section embraces this goal whole-heartedly, with the further recognition that despite the numbers or the reports that are generated, it is quality that remains the principal product of the laboratory. As such, we are committed to providing superior quality laboratory testing, method development, and training services to our clients and to the citizens of the state of Wisconsin. We also recognize that such a commitment is a dynamic endeavor, to be pursued continually.

Through a process of continual improvement and in cooperation with the Environmental Protection Agency, the Wisconsin Department of Natural Resources, the National Environmental Laboratory Accreditation Conference (NELAC), the Occupational Health and Safety Administration, the Wisconsin Division of Health, Wisconsin Department of Agriculture, Trade and Consumer Protection, local public health agencies and others, the laboratory will strive not only to meet, but to exceed, the quality objectives of our customers. Such continued improvement will be achieved through the following means:

- Heavy emphasis will be placed on the technical accuracy of the work done. This applies not only to the actual analytical work but also to written or verbal reports, court testimony, and communications with outside clients.
- In order to maintain and develop staff capabilities, employees will undergo continuing training. Such training will include both technical (e.g., computer skills) and philosophical (e.g., quality management).
- In addition the laboratory may provide incentives for staff to further their education in more in-depth ways.
- Such improvements in skills and technology will be employed whenever and wherever possible to improve both the efficiency and quality of the work done.
- Recognizing the relationship between job satisfaction and employee commitment, the management and staff will work to maintain the Environmental Health Division as an enjoyable and fulfilling place to work.
- The laboratory will provide a work environment based on the integrity and professionalism of its staff. Such an environment will help ensure that the work is performed with a dedication to excellence and continuous improvement.
- The laboratory staff in general, and its Quality Assurance Officers in particular, will strive to develop and maintain a good working relationship with state and federal regulators. It is viewed as necessary to our quality objectives to work in partnership with these agencies.

The specific procedures, practices and policy objectives employed in the laboratory are detailed in the following pages.

3. Management Structure and Responsibility

The Environmental Health Division (EHD) is one of six divisions of the Wisconsin State Laboratory of Hygiene (WSLH). The WSLH was created by state statute in 1903 and is overseen by the WSLH Board. The Board serves to set policy and direction for the Laboratory, and its members are either designated by statute or appointed by the Governor.

Operational management of the WSLH is the responsibility of the Laboratory Director. Division Directors report directly to the Laboratory Director. Department Supervisors report to the Division Director. There also may be subordinate supervisory personnel within the department structure who would report to the Department Supervisor.

It must be emphasized that while there is a hierarchical management structure that defines the laboratory, the WSLH embraces the "team" concept. All individuals, regardless of position, are encouraged to work together toward the goal of continuous improvement and a quality product.

3.1. Division Director

The EHD Division Director reports directly to the Laboratory Director, and is responsible for managing the organizational operations of the division. In addition, the Division Director is ultimately responsible for data quality. The position of Division Director is equivalent to the NELAC designation of Technical Director.

3.2. Department Supervisor

Each Section of the EHD is organized into various Departments. Each Department has a Department Supervisor who is responsible for day-to-day operation of the laboratory. In addition to the Department Supervisor there may be subordinate supervisors within the Department structure. For example, there may be a supervisor for the metals group within the Inorganic Chemistry Department. As a group these supervisors oversee sample analysis, data entry, report generation and all other related areas. In addition, they are responsible for employee management and review. In most cases these Supervisors meet the qualifications of a NELAC technical director and serve that function for their department.

3.3. QA Officer (QA Coordinator)

The QA Officer reports directly to the Division Director and is responsible for instituting and maintaining quality control procedures throughout the laboratory. The QA Officer will oversee the laboratory's certification status and coordinate the various regulatory programs. At the same time the QA Officer will maintain a working relationship with those same regulatory agencies and closely monitor any program or statutory changes. In addition they will perform internal QC audits to check on the on-going performance of both the personnel and the analytical processes.

Though the current QA Officers are experienced analysts, it must be recognized that they may not possess expertise in every area of environmental chemistry. The QA

Officers will rely on the senior chemists and supervisors of all areas to assist in carrying out their quality assurance duties. It must also be remembered that despite the presence of QA Officers, quality control procedures are the responsibility of all analysts.

3.4. Laboratory Staff

It is the primary responsibility of the frontline laboratory staff (bench analysts and support/administrative staff) to produce quality data within the structure of each individual method and within the parameters of the laboratory's quality control guidelines. It is also the responsibility of the staff to identify existing problems or inefficiencies, and to improve the processes of the laboratory whenever possible.

3.5. Position Descriptions

General descriptions for all employment categories are available online (O:\Job Specifications). Specific position descriptions for all personnel are located in the main Human Resources office in Room 226 of the WSLH at 465 Henry Mall. In addition, each Department Supervisor or Section Chief has copies of the position descriptions for their staff. See Chapter 20 for organizational charts.

3.6. Special Note — Biomonitoring

The Wisconsin State Laboratory of Hygiene is committed to a team management philosophy based in the tenets of total quality management. One of those tenets calls for empowering team members to make data based decisions in a customer-focused environment.

The Biomonitoring Department (WET) has embraced this philosophy whole-heartedly. The department is a self-directed work group, consisting of all of the scientists working within a functional unit or on a specific project and managing all aspects of the unit or project. A team leader is designated as responsible for the overall success of the team, and will either act as or appoint a facilitator responsible for team meeting processes. All aspects of the day to day operations will be managed by this team using the tools of quality management. The team takes full responsibility for carrying out the quality assurance plan, and will include a quality assurance data check as part of each team meeting.

Due to the autonomous nature of the team, the entire Biomonitoring Department (as a group) functions as a NELAC Technical Director.

4. Security and Access

Access to the Wisconsin State Laboratory of Hygiene (WSLH) Agriculture Drive site will be restricted to authorized individuals to insure the safety of all staff members and to maintain the integrity of all samples. The exterior doors of the main entrance and the loading dock areas will be open to our customers during the hours of 7:45 a.m. to 4:30 p.m., Monday through Friday. Both of these areas will be secured from the rest of the laboratory by electronic locks. Only staff members, custodial staff, and authorized visitors will have access beyond these two secured areas. The following sections detail the specifics of the policy.

4.1. General Access

All outside doors except the main entrance and loading dock, will be kept locked at all times.

The Customer Service (CS) reception area is open to the general public from 7:45-4:30, Monday-Friday. However, the interior door from the reception area to the laboratories will remain locked at all times and is only accessible through the use of electronic access cards, hard keys or through a buzzer-activated lock behind the CS reception desk.

The exterior loading dock door is open during the above hours. The sliding door between rooms 103D and 103, and the door between rooms 102A and 102 is electronic card accessible. Customers or vendors must buzz Receiving or Mailroom staff when making deliveries. There will be a main buzzer and sign on the west wall of the loading dock that provides simple instructions for access by vendors and customers. A backup buzzer will be located on the West wall of room 102A.

4.2. Staff Access

All staff members are issued one electronic access card and a photo ID card. Staff are required to have both their access card and their photo ID available while in the building. Supervisors must make arrangements to obtain an access card for new staff members no later than their starting day, and the photo ID as soon as practical--usually within two days.

Staff members have access to side doors and the main entrance of the laboratory through electronic locks that are activated by their access cards. If a staff member forgets their access card, they must enter at the main entrance.

Staff must report any lost access cards and ID cards to their supervisor immediately. The supervisor or appropriate WSLH liaison must notify the Capital Police to have the access card deactivated and request that a new card be issued. Staff will be charged for replacement of lost access cards.

Areas and hours of access are determined by the department supervisor or jointly determined by the supervisor and a team of staff members. Access decisions are based on the needs of the department.

WDNR fish grinding staff and the WDNR Laboratory Liaison are issued access cards, and are considered part of the EHD team. The grinding room staff will have the same access rights and responsibilities as other WSLH staff.

4.3. Custodial and Service Staff Access

Three access cards have been issued to Environmental Control (EC), which is the current cleaning contractor employed by the Department of Administration (building landlord). The area manager for EC has access from 7:00 a.m. until midnight, Monday-Friday. Two other crew supervisors have access from 4:30-midnight, Monday-Friday. One of the EC supervisors will let 3-4 cleaning crew members in the lab each evening at about 5:00 p.m. Each crew member will enter the lab, call EC's time clock from a single, designated phone and check-in. The EC time-clock system uses caller ID to make sure the crew members are on-site. After completing their shift, each EC crew member calls back into the time clock and checks-out. One of the two crew supervisors or the area manager will then let the crew members out before leaving themselves. EC will, at our request, provide a detailed printout showing who was on-site on any given evening, when they arrived and when they left the building. That, combined with the Capital Police door activity log, can provide the lab with a detailed list of those present on-site.

The protocol for the personnel will be as follows:

- A supervisor will be on site at all times and will let the employees into the building. Each employee is required to call the office and report to work at the site. They are to use a designated phone in the building so that their location can be verified. This record is maintained by the company.
- Stripping and waxing of the floors will take place two times per year and we will get a tentative schedule so that we can plan our work.
- A log and request book will be kept in the front office area. This book will be looked at and initialed by the supervisor each day. Any special requests that you have that fit into cleaning can be put in the book and they will be addressed that night.

4.4. Visitor Access

All visitors must sign-in at the Customer Service (CS) reception desk, and are issued one of two types of visitor badges. These badges must be displayed at all times while in the laboratory.

It should be noted that laboratory staff members play an important role in the overall security of the facility. Through day-to-day contact they should be aware of the proper personnel associated with their area. If an individual who is not displaying proper credentials and/or is unknown to staff members is seen in an area where they may not belong, staff members should inquire whether they need assistance. If a staff member feels uncomfortable or threatened they should contact a supervisor or call 9-911 and request assistance from the proper authorities.

The Customer Service office maintains the visitor's log. The logs will be maintained for a length of time consistent with the laboratory's record retention policy (generally 10 years).

4.5. Chain-of-Evidence Security

All Chain-of-Evidence (COE) (law enforcement) samples are maintained in a secured (locked) area when not in use. Only authorized laboratory staff have access to the samples. See Chapter 10 for a more complete explanation.

4.6. Maintenance of the Security Processes

An electronic copy of the Security and Access SOP will be maintained on the shared EHD server. See EHD GENOP 011. All staff will have read access to the policy. Administrative staff can only make changes after approval by the EHD Director. All revisions will be tracked and it will be clear which revision is in force. The revision number and revision date will be documented in the SOP header field.

5. Complaints

Although the Laboratory strives to provide services in a timely and high quality fashion, it is expected that we will occasionally make mistakes or fail to please a customer. When complaints occur it is expected that the laboratory staff will handle them in a consistent, courteous, and prompt manner. To further this goal the laboratory has put a Customer Complaint Procedure in place, outlining the way in which complaints should be handled, documented and resolved.

A summary of this procedure is listed below. The complete procedure is outlined in SOP EHD GENOP 017. A copy of the complaint form can be found on the following page.

- When a complaint is received, relevant information is recorded on a Customer Feedback Report Form. The form is then forwarded to the supervisor of the applicable area.
- The supervisor will determine what resolution is required and direct appropriate staff if necessary.
- The follow up action taken will be recorded on the form, signed by the supervisor and filed in the appropriate location once the resolution is completed.

Figure 5.1 — Customer Feedback Report Form

(For Department Use Only)

Initiated by:_____ **Date:**_____ **Department:**_____

Customer Name:_____

Customer Company Name:_____

Date of Contact:_____

Feedback Classification:

o Compliment o Complaint o Information Only o Other:_____

Brief Description of Feedback:

Name(s) of Employee(s) Involved:

Immediate Action Taken (if any): (include dates and specifics)

Resolved? Yes o No o

Forwarded to:_____ **Date:**_____

Reference #_____

6. Hiring Process

When a position opens the relevant supervisor (or principal investigator), along with the Human Resources Department (HR), will determine whether it falls under the designation of classified staff or academic personnel. Once that designation has been made one of the following processes will be followed.

6.1. Classified Staff

The classified staff hiring process conforms to the requirements of Wisconsin's Civil Service system and the University of Wisconsin's policies for hiring classified staff. These requirements are designed to ensure the Laboratory's ability to hire highly qualified personnel.

If the position is determined to be classified staff the relevant supervisor will submit a request to hire and a position description to HR. The HR department will review the materials, finalize the position description and submit the materials to the University Classified Personnel Office (CPO) for review. A position number will be assigned and the position will be posted following the civil service guidelines. Please see SOP EHD GENOP 012 for a more detailed explanation of the recruitment options.

Once a list of candidates has been chosen, interviews can be scheduled. The supervisor is responsible for choosing the interview team and for devising the interview questions. At the time of the interview all applicants will be informed that a criminal background check will be done before hiring. Each applicant will be asked the same questions and the interviewers will score their answers. All candidates will be notified if they are not selected. Once a position is offered and accepted, an appointment letter will be sent to the successful candidate.

6.2. Academic Staff

If the position is determined to be academic staff, the principle investigator, with guidance from HR, will assign an appropriate title and determine the type and salary range of the appointment. A request to hire and a position description will be prepared and sent to HR. The HR department will review the materials, prepare a recruitment file and submit the materials to the University Academic Personnel office for review. A position number will be assigned and the position will be posted.

Upon receiving applications the principal investigator will determine the appropriate candidates, select an interview panel and conduct the interviews. All candidates will be notified if they are not selected. Once a position is offered and accepted, an appointment letter will be sent to the successful candidate.

All records associated with the hiring of staff for the Division will be turned over to the Human Resources Department. Human Resources will be responsible for the maintenance and final disposal of these records.

The procedures set forth in this document will not be changed without the consent or direction of the Human Resources Department.

7. Personnel

7.1. Safety

To insure the safety and well being of all Wisconsin State Laboratory of Hygiene personnel, new employees must become familiar with basic safety precautions before working in the laboratory. Before beginning any work, they will be given the University of Wisconsin System Classified Employee Work Rules and taken through the EHD - safety check list. They will also be required to familiarize themselves with the following items:

- The Safety Section from Standard Methods
- The Environmental Health Division Chemical Hygiene Plan
- The UW Chemical Safety and Disposal Guide
- The Quality Assurance Manual
- All applicable general operations SOPs of the specific department.

Depending on their position, some employees may require more specialized instruction such as HIPAA Privacy Rule training, review of the Bloodborne Pathogens Reference and Training Manual, or participation in the Radiation Safety Training offered by the University. In addition, members of the Shipping and Receiving area have received training in the "Handling and Shipping of Hazardous Materials", also offered by the University.

The laboratory maintains a number of safety videotapes that may be used at the discretion of the Department Manager. These include:

- "Working Together: Needlestick Prevention" (Clinical Staff)
- "Working Together: Needlestick Prevention" (Support Staff)
- Vented Balance Safety Enclosure 2000 Series
- For Your Protection: The OSHA Regulation on Bloodborne Pathogens
- A Discussion of the Bloodborne Pathogens Standard. 29 CFR 1910.1030
- As It should Be Done: Workplace Precautions Against Bloodborne Pathogens
- The Bloodborne Pathogens Standard: What It Means To You (BBP 01)
- Controlling Your Risks — HIV In The Research Laboratory
- Bloodborne Pathogens In The Laboratory: A New Way of Looking At Things (BBP 02)

All employees are made aware of the safety facilities available in the laboratory including the location of safety showers, eyewash, fire extinguishers and chemical spill clean-up materials. Emergency procedures and fire evacuation routes will also be explained. No employee will be allowed to work with strong acids, bases, or other

hazardous materials until they have become familiar with the appropriate safety precautions.

All current employees are required to reaffirm, on an annual basis, that they are familiar with basic safety concepts and the various safety equipment in the laboratory. If necessary, employees are expected to re-familiarize themselves with the Chemical Hygiene Plan and specific safety procedures as appropriate.

The Environmental Health Division operates a building safety committee that meets regularly and conducts safety inspections. Minutes of this committee are available on the shared network drive. Membership of this committee consists of a cross section of laboratory personnel. Safety training is encouraged, and it is the responsibility of department managers to approve safety training courses and workshops. Safety reminders are occasionally sent to employees via e-mail and a safety column appears regularly in the WSLH employee newsletter.

7.2. Training

7.2.1. Bench Training

7.2.1.1. General

New employees will become familiar with the general laboratory organization as well as the basic quality assurance practices before performing any analytical functions. This information is presented verbally during bench training and further reinforced by reviewing the laboratory Quality Assurance Manual.

After thoroughly reviewing the in-house SOP and/or the regulatory method, the new employee will work under the direct supervision of an experienced analyst until they become familiar with the analytical procedures.

If applicable, they will perform an Initial Demonstration of Capability (IDC). In addition, a senior analyst may randomly select and analyze samples that have already been analyzed by the new employee to verify the validity of the test results. The results of the IDC will be placed in the method MDL/IDC file, and the initials of the analyst doing the training will be placed on the training sheet. In addition, a "Demonstration of Capability Certification Statement" will be completed (see figure 7.1 for an example). Other DOC statements that contain the same required language, but that also have areas for data entry may be used by individual departments. A copy will be placed in the employee's personnel training file.

"Unknowns" or reference samples may be analyzed by the new employee at the discretion of the supervisor to further verify their understanding of the methods. When IDC criteria have been satisfied and the experienced analyst and supervisor are confident that the employee is thoroughly familiar with the test, that employee is allowed to work on their own with only routine supervision.

7.2.1.2. Instrumentation

The new employee will review the instrument manual, along with any other special instructions, before operating an instrument. They will observe an experienced analyst prepare and operate the instrument. They will then operate the instrument under the direct supervision of an experienced analyst. When the experienced analyst is confident that the new employee is thoroughly familiar with the instrument, they will be allowed to work on their own with only routine supervision.

7.2.2. Employee Improvement

7.2.2.1. Formal Outside Education

In addition to training offered by the organization, the laboratory supports continuing education that may include seminars, training offered by vendors, or formal higher education.

7.2.2.2. Technical Training

All employees are encouraged to keep up with changes or advances in analytical methods and instrumentation. This is done by circulating literature and other pertinent information as it becomes available.

Employees are also offered lab-wide training in new computer aided tools (e.g., PC-based software or new programs developed on LIMS).

7.2.3. Ethical and Legal Responsibility

All employees receive a copy of the Environmental Health Division Policy on Ethical Behavior. The policy includes the University of Wisconsin System Classified Employee Work Rules, and spells out the personal and professional conduct which is expected of all employees, and the possible consequences for not behaving in a professional manner.

In addition all employees have access to the Wisconsin State Laboratory of Hygiene Confidentiality Policy which outlines duties and responsibilities for dealing with client information.

7.2.4. Documentation

The training process is tailored according to the needs of each department. In all cases training will be documented for each new employee. Once the new analyst has successfully completed the list of items on the analyst training record, their supervisor will sign the form confirming that all training requirements have been met for that department. The training checklists are kept in a central file for quick reference. All outside training records and safety records are kept in the Human Resources Department, in the individual personnel records.

7.3. Education and Experience

7.3.1. Environmental Health Division — Administration

Name	Title	Degree	Yrs Exp
William C. Sonzogni	Professor / Division Director	BS Chemistry, Ph.D. Water Chemistry	30
Thomas Dunnick	IS Comprehensive Sr.	BS Chemistry, MS Computer Science	29
Karl Patzer	Chemist Advanced	BS Biology	16
Susan Hill	Chemist Advanced / QA Coordinator	BS Chemistry, MS Water Chemistry	18
Dave Schleis	IS Programmer Analyst Senior	BS Chemistry, + UW Computer Science Courses	20
Matt Roach	Chemist Advanced / QA Officer	BS Chemistry	16

7.3.2. Biomonitoring (WET)

Name	Title	Degree	Yrs Exp
Miel Barman	Environmental Toxicologist - Senior	BS Bacteriology	10
Steven W. Geis	Chemist Supervisor	BS Water Chemistry, MS Water Chemistry	14
Jocelyn Hemming	Assistant Scientist	BS Biology, Ph.D. Environmental Toxicology	6
Dawn Karner	Environmental Toxicologist - Senior	BS Zoology	8
Amy Mager	Environmental Toxicologist - Senior	BS Zoology, MS Environmental Toxicology	7
Robert Tocco	Environmental Lab Tech--Senior		1
Jonathan Standridge	Microbiologist Management Supervisor	BS Bacteriology	28

7.3.3. Inorganic Chemistry

Name	Title	Degree	Yrs Exp.
Graham Anderson	Chemist Advanced	BS Biology / Med. Tech	9
Michael Arndt	Assistant Scientist	BS Chemistry/Zoology, Ph.D. Analytical Chemistry	4
George Bowman	Chem. Mgmt. Supervisor	BS Fish Management	30
Kit Bruehl	Program Assistant 2	Associate Med. Lab Tech	17
Al Clary	Chemist Advanced	BS Chemistry	28
Jane Frawley	Chemist Senior	BS Biology	13
Dustan Helmer	Chem. Lab Tech - Senior	Associate Science	3
Kevin Kaufman	Chemist Senior	HS + 4 yrs University (Chemistry)	13
DeWayne Kennedy-Parker	Chemist Advanced	HS + 3 yrs University (Chemistry)	16
Andy Klisz	Chemist Senior	BS Physics	13
Royce Kreul	LTE Chemist Senior	BS Education, MS Education	29
Mike Manix	LTE Chemist Senior	BS Education, MS Education	22
Julie Maybee	MGMT INFO TECH 3	Associate Data Processing	14
Chris McSweeny	Chemist Supervisor	BS Chemistry / Business Administration	18
Joan Mitchell	Chemist Senior	BS Education	28
Anthony Plourde	Chemist Senior	BS Chemistry	3
Roger Schulz	Chemist Senior	BS Biology	15
Martin Shafer	Associate Scientist	Ph.D. Water Chemistry	15
Cristine Thielman	Chemist Senior	BS Chemistry	24
Lorrine Vingum	Chemist Senior	BS Biology, MS Biology	10
Gene Wick	LTE Chemist	BS Education, MS Education	5
Chris Worley	Chemist Supervisor	BS Biology, MS Biology	12

7.3.4. Organic Chemistry

Name	Title	Degree	Yrs Exp
Erin Bean	Chemist	BS Chemistry	2
Carol Buelow	Chemist Advanced	BS Chemistry / MS Water Chemistry	20
Steven W. Geis	Chemist Supervisor	BS Water Chemistry, MS Water Chemistry	14
Tom Gibson	Chemist Advanced	BS Chemistry	38
Bill Krick	Chemist Advanced	BS Chemistry	19
John Mathew	Chemist Supervisor	Ph.D. Analytical Chemistry	21
Jim O'Loughlin	Chemist Advanced	BS Chemistry / Biology Minor	19
Matt Roach	Chemist Advanced	BS Chemistry	15
Dave Rogers	Chemist Senior	BS Water Resources/ Chemistry Minor	13
Al Spallato	Chemist Senior	BS Life Science	17
Jim Tortorelli	Chemist Senior	BS Chemistry / Ph.D. Physical Chemistry	23

7.3.5. Radiochemistry

Name	Title	Degree	Yrs Exp
Doris Anderson	Chemical Lab Tech Objective	HS Diploma	10
Yuliya Henes	Chemist Entry	BS Chemistry (Russia), MS Chemistry (area of specialization – Radiochemistry)	1
Gary Krinke	Chemist Senior	HS + 3.5 yrs University	15
Lynn West	Chemist Supervisor	BS Chemistry, MS Water Chemistry	23

7.3.6. Water Microbiology

Name	Title	Degree	Yrs Exp
Barb Gaffney	Program Assistant 2	HS + 1 yr University	11
Sharon Kluender	Microbiologist Supervisor	BS Bacteriology	24
Rebecca Leidner	Micro Lab Tech - Objective	Associate Biotechnology	7
Veronica Mack	Program Assistant 1	HS Diploma	7
Jeremy Olstadt	Microbiologist	BS Biology	6
Linda Peterson	Microbiologist Senior	BS Medical Microbiology	9
Alan Degnan	Microbiologist Senior	MS Food Science	13
	Micro Lab Tech--Objective		
Jonathan Standridge	Microbiologist Management Supervisor	BS Bacteriology	28

Figure 7.1 — Demonstration of Capability Certification Statement

Demonstration of Capability Certification Statement

Date: _____

Laboratory Name: Wisconsin State Laboratory of Hygiene

Laboratory Address: 2601 Agriculture Dr. Madison, WI 53707-7996

Analyst(s) Name(s): _____

Matrix: _____

Method number, SOP#, Rev#, and Analyte, or Class of Analytes or Measured Parameters:

_____

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

_____	_____	_____
Analyst	Signature	Date
_____	_____	_____
Technical Director	Signature	Date
_____	_____	_____
Quality Assurance Officer	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

Figure 7.2 — New Employee Training List for Biomonitoring

Employee's Name:

Date started:

Task	Trained	Week 1	Week 2	Week 3	Week 5	Independent	Comments
Turn on meters							
Fish tasks (a.m.)							
Temperatures (a.m.)							
Clean pH probes							
Calibrate meters							
Prepare waters							
Update water chems							
QA							
Algal tasks							
Transfer broodboards							
Hyalella							
D. magna							
Fish tasks (p.m.)							
Temperatures (p.m.)							
Sanitize Milli-Q							
Acute Test							
Chronic Test							
Algae Test							
Acute RTT Test							
Chronic RTT Test							
Microtox							
Weigh Fish							
Chemistries							

Figure 7.3 — Inorganic Chemistry New Employee Training Checklist

1. Safety Training
    - 1.1. Chemical Safety
        - 1.1.1. UW Chemical Disposal Guide
            - 1.1.1.1.Location ☐
            - 1.1.1.2.Review ☐
        - 1.1.2. SLH Chemical Hygiene Plan
            - 1.1.2.1.Location ☐
            - 1.1.2.2.Review ☐
        - 1.1.3. Standard Methods Safety section ☐
    - 1.2. Safety Equipment Location and Use
        - 1.2.1. Safety Shower ☐
        - 1.2.2. Hoods ☐
        - 1.2.3. Eyewash ☐
        - 1.2.4. Fire Extinguisher ☐
        - 1.2.5. Spill Control Kits ☐
        - 1.2.6. First Aid Kit ☐
        - 1.2.7. Lab Coats ☐
        - 1.2.8. Safety Glasses ☐
        - 1.2.9. Other Personal Protective Equipment ☐
2. Ag Drive Lab Tour
    - 2.1. Key System & Access Card ☐
    - 2.2. Breakroom
        - 2.2.1. Refrigerators ☐
        - 2.2.2. Vending machines ☐
        - 2.2.3. Coffee fund ☐
        - 2.2.4. Supplies ☐
    - 2.3. Conference Rooms ☐
    - 2.4. Customer Service ☐
    - 2.5. Shipping ☐

2.6. First Floor ☐
2.7. Second Floor ☐
2.8. Basement ☐
2.9. Restrooms ☐
2.10. Personal Space ☐

3. Inorganic Lab Tour
   3.1. Lab Space ☐
   3.2. Supply Storage ☐
   3.3. Sample Check-in ☐
   3.4. Sample Storage ☐
   3.5. Chemical Storage ☐
4. Meet with Human Resources Rep
   4.1. Complete forms ☐
   4.2. Time Sheet ☐
5. Computer logon and training
   5.1. Get Logon ☐
   5.2. NT training ☐
   5.3. Outlook training ☐
   5.4. Computer policy ☐

6. SLH Policies
   6.1. Employee Handbook ☐
   6.2. Q.A. Manual, Inorganic Chemistry Dept. ☐

New Employee_____Date_____

Supervisor_____Date_____

Figure 7.4 — Analyst Method Training Form

(Inorganic Chemistry)

Name (Trainee):_____ Name (Trainer):_____

Title:_____ Title: _____

State Laboratory of Hygiene Method # and title:_____

Reference method:_____ ___

Analyte(s):_____

Training Criteria	v Complete	Date
1) Analyst has reviewed and understands the current SOP and reference method.	_____	_____
2) Analyst is familiar with sample handling, preservation techniques, and holding times associated with the method. This includes training on the sample tracking software.	_____	_____
3) Analyst is familiar with hazards specific to the method and with procedures for reducing any risks involved.	_____	_____
4) An Initial Demonstration of Capability has been successively completed by the analyst and documented using the IDC form.	_____	_____
5) Analyst knows how to make a worklist (ESS INO GENOP 108) for samples to be analyzed by this method.	_____	_____
6) Analyst has been trained on proper entries into logbooks, which may include logbooks for instruments, standards, reagents, and balances, and other documentation requirements.	_____	_____
7) Analyst has been trained on the calibration procedures detailed in the method.	_____	_____
8) Analyst understands the step-by-step instructions for processing samples as detailed in the method, including all calculations used to arrive at the final result.	_____	_____
9) Analyst demonstrates an understanding of the mechanics of the instrument, knows where the instrument manuals are located, and has some knowledge of trouble-shooting the instrument.	_____	_____
10) Analyst is familiar with all QC/QA issues and procedures associated with this method including; frequency and acceptance criteria of blanks, check samples, controls, and duplicated or spiked samples. Proficiency with calculations or software used to process QC data has been demonstrated.	_____	_____

	v Complete	Date
Training Criteria		
11) Analyst is familiar with any general operating methods and QA methods associated with this method (e.g. calibration of balances, calibration of pipettes, temperature checks, neutralizer operation).	_____	_____
12) Analyst is familiar with data management, including the process for reporting data (manual or direct transfer), and filing completed analytical runs.	_____	_____
13) Analyst has been trained on proper disposal procedures for samples analyzed by this method and any intermediate aliquots or by-products created.	_____	_____
14) Analyst has completed the above criteria and has gained enough experience to do QC Audits of other analysts' runs.		

_____ _____
Trainee signature Date

_____ _____
Trainer signature Date

Figure 7.5 — Analyst Training and SOP Review Record

(Organic Chemistry Department)

SOP Name _____

SOP Number _____

Revision Number _____

Revision Date _____

Analyst	Initial Training (X)	SOP Reviewed (X)	Verified / Trained by	Date Verified

Figure 7.6 — Analyst Training Record for Radiochemistry

Administrative, Analytical, and Instrument Methods

Analyst Name: _____ Employee ID No.: _____

Supervisor: _____ Supervisor ID No.: _____

SOP No:	SOP steps	Instructor ID No	Training Date	Completed

Figure 7.7 — Radiochemistry New Employee Training Checklist

7. Safety Training
   - 7.1. Radiochemical Safety
     - 7.1.1. UW Radiation Safety Course ☐
       - 7.1.1.1. Schedule Training ☐
     - 7.1.2. UW Radiation Safety Manual ☐
       - 7.1.2.1. Location ☐
       - 7.1.2.2. Review ☐
   - 7.2. Chemical Safety
     - 7.2.1. UW Chemical Disposal Guide
       - 7.2.1.1. Location ☐
       - 7.2.1.2. Review ☐
     - 7.2.2. SLH Chemical Hygiene Plan
       - 7.2.2.1. Location ☐
       - 7.2.2.2. Review ☐
   - 7.3. Safety Equipment Location and Use
     - 7.3.1. Safety Shower ☐
     - 7.3.2. Hoods ☐
     - 7.3.3. Eyewash ☐
     - 7.3.4. Fire Extinguisher ☐
     - 7.3.5. Spill Control Kits ☐
     - 7.3.6. First Aid Kit ☐
     - 7.3.7. Lab Coats ☐
     - 7.3.8. Safety Glasses ☐
     - 7.3.9. Other Personal Protective Equipment ☐
8. Ag Drive Lab Tour ☐
   - 8.1. Key System ☐
   - 8.2. Breakroom
     - 8.2.1. Refrigerators ☐

8.2.2. Vending machines ☐
8.2.3. Coffee fund ☐
8.2.4. Supplies ☐
8.2.5. Conference Rooms ☐
8.2.6. Customer Service ☐
8.2.7. Shipping ☐
8.2.8. First Floor ☐
8.2.9. Second Floor ☐
8.2.10. Basement ☐

8.3. Restrooms ☐
8.4. Personal Space ☐

9. Radchem Lab Tour ☐
9.1. Counting Room ☐
9.2. Lab Space ☐
9.2.1. Supply Storage ☐
9.3. Sample Check-in ☐
9.4. Sample Storage ☐
9.5. Chemical Storage ☐

10. Meet with Human Resources Rep
10.1. Complete forms ☐
10.2. Time card ☐

11. Computer logon and training
11.1. Get Logon ☐
11.2. NT training ☐
11.3. Outlook training ☐
11.4. Computer policy ☐

12. SLH Policies and Handbook
Location and review ☐

Figure 7.8 — Media MICRO Lab Tech Training List

Name:			
TASKS	**DATE TRAINED**	**TRAINER**	**DATE REVIEWED**
1. Record temperatures 2X daily of refrigerator, incubator, water baths			
2. Record date, volume used, conductivity of Type I water			
3. Sanitize Type I polisher weekly			
4. Operate steam sterilizer			
5. Record run date, contents, sterilized, max temp			
6. Run spore strips on steam sterilizers			
7. Clean trap on steam sterilizer weekly			
8. Clean inside of autoclave			
9. Maintain copies of maintenance agreements and maintenance log			
10. Inspect the seal on the autoclave daily			
11. Wipe down countertops daily			
12. Scrub media room floor weekly			
13. Prepare MFC according to recipe			
14. Prepare Kstrep according to recipe			
15. Prepare ME according to recipe			
16. Prepare EIA according to recipe			
17. Prepare urease according to recipe			
18. Prepare Standard Methods Agar plates			
19. Prepare Standard Methods Agar			
20. Prepare Nutrient Broth			
21. Prepare Trypticase Soy Broth			
22. Prepare Lauryl Tryptose Broth			
23. Prepare Double Strenght LTB			
24. Prepare Brilliant Green Bile Broth			
25. Prepare BHI broth			
26. Prepare BHI agar			

27. Prepare 40% bile broth			
28. Prepare nutrient agar slants			
29. Prepare buffer jugs			
30. Prepare bottles of dilution water			
31. Check timing device with stopwatch quarterly			
32. Standardize pH meter with each use			
33. Date Bottles of media when received			
34. Date Bottles of media when opened			
35. Date bottles of solutions with date made			
36. Fill out media sheets each batch			
37. Calibrate balances with certified weights			
38. Keep maintenance logs for balances			
39. Calibrate thermometers against NIST therm.			
40. Calibrate micropipettors			
41. New lots of containers validated for <2.5%			
42. Spot check glassware for pH reaction			
43. Maintain media preparation records			
44. Label each batch of media with name, date and expiration date			
45. Throw out expired media			
46. Clean biological safety hood after each use			
47. Proper use of biological safety hood			
48. Proper use of fume hood			
49. Calibration of dispensing equipment			
50. Maintain media supply			
51. Maintain chemical inventory			
52. Deliver media and reagents to units			
53. Clean carts			
54. Order supplies - Fill out Hy48			
55. Clean and maintain supply inventory of plate, petri dishes, etc			
56. Wrap and sterilize filter funnels			
57. Maintain MSDS			

Figure 7.9—Water Microbiology Training Verification

Employee _____

TASK	DATE/INITIAL VERIFIED	DATE REVIEWED	COMMENTS
CHECK IN PUBLIC SAMPLES			
-date and time			
-separate test methods			
-designate qualifiers (CS,30 hrs, old sample, etc)			
-record municipal times			
-verify times			
BACTI COMPUTER # SYSTEM -create #'s, record disposition, etc			
CHECK IN PRIVATE SAMPLES			
-dates			
-pour off chemistry			
-check pools for chlorine			
-separate 'tribe' samples			
CHECK IN SURFACE WATERS			
-date and time			
-test method (bacteria requested)			
-check that iced			

SET UP	DATE/INITIAL VERIFIED	DATE REVIEWED	COMMENTS
MUNICIPAL/PRIVATES			
-Colilert, Colisure, Qauntitray (CT, CS,ENT, C-18)			
-MPN 10 Tube			
-Membrane Filtration-Total Colif M ENDO MF			
-Heterotrophic Plate Count-HPC			
-Sulfate Reducing Bacteria			
-Iron Bacteria			
-Pseudomonas			
-E*colite			
-Coli blue			
SURFACE WATERS			
-Membrane Fitration			
Fecal Coliform			
Fecal Strep			
E. coli			
Enterococci			
COLIPHAGE (Male Specific)			
-Presence/Absence Enrich Meth.			
-Double Agar Layer Method			

READ RESULTS	DATE/INITIAL VERIFIED	DATE REVIEWED	COMMENTS
Colilert -Tot Colif			
-E. coli			
C-18 -Tot Colif			
-E. coli			
Colisure -Tot Colif			
-E. coli			
Enteroalert -Tot Colif			
-E. coli			
10 Tube MPN- Total Coliform			
- Fecal Colif/ E coli			
Membrane Filtration- Total Colif			
-Fecal Coliform-MF			
-Fecal Strep-MF			
-E. coli-MF			
-Enterococci-MF			
-HPC			
-Pseudomonas			
-Sulfate Reducing bacteria			
-Iron Bacteria			
-Coliphage -presence/absence			
-double agar layer			

VERIFY RESULTS	DATE/INITIAL VERIFIED	DATE REVIEWED	COMMENTS
-Total Coliform-BG and SS			
-Fecal coliform			
-Fecal strep			
-E. coli			
-Enterococci			
-MPN 10 Tube **QUALITY CONTROL**			
-Membrane Filtration QC (Equip check and Buffer jugs)			
-Daily temperature recording			
-Quantitray QC per box			
-ONPG-MUG quality per month			

8. Documentation Procedures

8.1. General

See Chapter 1, Manual Organization and Maintenance, for procedures used in the maintenance of the Quality Assurance Manual.

8.2. Biomonitoring

8.2.1. Information Flow, Data Retrieval and Storage

8.2.1.1. Sample Receipt

Samples received at the State Laboratory of Hygiene for Whole Effluent Toxicity testing are accompanied by a sample log sheet. These sheets contain biographical information about sample type, collection site, date, time, temperature and pH. Sample receiving and disposal information is also recorded on the sample sheets. Samples are given a unique laboratory test number, which is logged into the bound test logbook. Test sheets are made for each sample site. Data is recorded on the test sheet daily.

8.2.1.2. Data Review and Reporting

At the conclusion of each test, results are recorded by the analyst on the bench sheets along with any data qualifiers. The test results, QC data, and any data qualifiers are entered into an official report form. Each report is then reviewed, any errors are corrected, and the final report is mailed to the client.

8.2.1.3. Data Retention

All test sheets and final test reports are maintained in a manila folder for 5 years. Test results are recorded electronically in a Microsoft ACCESS database.

8.2.2. Manuals, Methods and SOPs

Current Manuals, SOPs, and other supporting documents are readily available to all personnel. Any archived items will also be available if required. Each item will be clearly labeled and dated, so that it is apparent when said document was in force. Hardcopies will be made available to Laboratory customers or to regulatory agencies if requested.

All SOPs are reviewed yearly and revised when necessary, due to instrument/technology changes, regulatory changes, etc. Major changes, such as those noted above, will result in a new revision of the SOP. Each new revision will have an effective date.

8.3. Inorganic Chemistry

8.3.1. Information Flow, Data Retrieval and Storage

8.3.1.1. Sample Receipt and Distribution

Samples are initially received at the Specimen Receiving Department located at the north end of the building (see ESS INO GENOP 106). The sample containers and test request forms (TRFs) are removed from their shipping containers. The TRFs are labeled with the laboratory ID number, and the sample containers are given a barcode label with the corresponding laboratory ID and a unique letter identifier. Information regarding date and time of receipt, preservation and/or thermal status (if required), sample condition, and type of sample container(s) received, is recorded in the sample tracking software (see EHD GENOP 024). The samples are then delivered to the Inorganic laboratory sample receipt area where they are checked in for processing (see ESS INO GENOP 104). Once check-in is complete the samples are placed in the appropriate location for storage until analysts remove them for analysis.

All pertinent sample information is entered into the Laboratory Information Management System (LIMS) database. The LIMS database automatically tracks sample status, entry, and modification times on a per result basis. The general information management scheme used by the Inorganic Chemistry Department is described in Figure 8.1.

8.3.1.2. Data Review

Each analyst will search the LIMS database and generate a worklist based on test codes assigned to the analyses (see ESS INO GENOP 108). All sample data along with the appropriate QC data are linked to that worklist. When the analyses are completed the results are audited at the bench level by a chemist familiar with the analysis (see ESS INO QA 103 & 107). Both the data package and the QA worksheet are initialed and dated. The approved results are either transferred directly from the instrument into LIMS or entered manually by data management staff. Once entered, the results are checked by a variety of programs (e.g. RELATE). These programs look at relationships among analytes and check for calculation errors. For example, dissolved phosphorus cannot be greater than total phosphorus. If no errors are found and the results look reasonable the Department Supervisor or their designee will review the data and authorize the release of final results to the customer.

8.3.1.3. Data Reporting

Results are available in hard copy reports, which may be sent to the applicable parties, and/or filed. For Safe Drinking Water Act (SDWA) and law enforcement samples the original TRF, chain-of-custody form (if applicable), and final report are mailed directly to the customer. In

addition, results are available to the Wisconsin Department of Natural Resources (WDNR) in electronic format.

8.3.1.4. Data Retention

Hardcopies of all bench sheets (including calibrations and raw data) are currently kept on site for at least ten years. A listing of the record boxes stored is available on the shared EHD server (Record Storage Boxes.doc). Electronic copies of the sample biographical data and their associated results are automatically backed-up daily on magnetic tape. Each month one of the tapes is archived. Electronic data is available on-line for at least three years. Electronic versions of raw data will be backed up onto CD-R on a routine basis.

8.3.2. Manuals, Methods and SOPs

Current Manuals, SOPs, and other supporting documents, such as IDC/MDL studies and program specific Quality Assurance Project Plans (QAPPs) are readily available to all staff. Any archived items will also be available if required. Each item will be clearly labeled and dated so that it is apparent when the document was in force.

Although printed hard-copy versions of Department SOPs are available. All "official" and current SOPs are kept in an electronic format (MSWord or HTML) and will be available to all staff members as "read-only" documents. Hardcopies will be made available to customers or to regulatory agencies if requested.

Analytical SOPs (i.e. analytical methods) are revised when necessary due to instrument/technology changes, regulatory changes, etc. Major changes, such as those noted above, will result in a new revision of the SOP. Each new revision will have a new revision number, an effective date, and will list the SOP it is replacing. In general, if minor semantic or typographical changes are made a new revision number will not be used.

IDC and MDL studies are stored directly in the laboratory in the relevant area. The MDL data is also listed in the appropriate analytical SOP in the form of LOD and LOQ tables.

Occasionally, at the request of a client, the laboratory will prepare a Quality Assurance Project Plan. These documents are kept with the actual contract and other historical information associated with the project.

8.3.3. Logbooks

The laboratory utilizes logbooks for various purposes including: instrument maintenance, instrument injection or run logs, cataloging of standards and their preparation, monitoring sample movement for chain of custody or enforcement purposes, and temperature record-keeping for ovens, refrigerators, freezers and walk-in coolers. A master logbook tracks locations and effective dates for all logbooks.

8.4. Organic Chemistry

8.4.1. Information Flow, Data Retrieval and Storage

8.4.1.1. Sample Receipt and Distribution

All samples received at the Laboratory are accompanied by a test request form (TRF). Each TRF is dated, and given a unique number. In addition samples which have temperature specific requirements will have their temperatures taken and recorded along with the initials of the individual who checked in the sample. All pertinent sample information is then entered into the Laboratory Information Management System (LIMS) database. The LIMS database automatically tracks sample status, entry, and modification times on a per result (and per sample) basis. The general information management scheme used by the Organic Chemistry Department is described in Figure 8.2.

For the Organic Chemistry Department, worksheets are generated and attached to the TRF. These sheets are placed in a manila folder identified with pertinent sample information and distributed to the appropriate analysts for testing. The analyst will initial the paperwork, record dates and other identifiers (batch #s), and list any necessary comments about the analysis.

8.4.1.2. Data Review

Upon completion of analysis (including the checking of QC data and the possible electronic transfer of data to LIMS), the sample is completed by the analyst. Sample completion consists of entering all results, (including dates of analysis, temperature, analyst ID, all qualifiers and comments, and analytical results), into the LIMS system, generating a logbook for the purposes of auditing the completed results, and then forwarding the samples to peers and/or supervisors for review. Once reviewed the data is stamped, dated and initialed before being forwarded to the Department Supervisor where it is checked again. During the review process, all aspects of the sample are checked (see ESS ORG QA 0008 for more detail). Any corrections are made in consultation with the analyst and then final results are released.

8.4.1.3. Data Reporting

Results are available in hard copy reports, which may be sent to the applicable parties, and/or filed. For SDWA and enforcement samples the original TRF and final report are mailed to the customer. In addition results are available to various external agencies (primarily the WDNR) in an electronic format.

8.4.1.4. Data Retention

Hardcopies of all worksheets, calibrations, and raw data are currently kept on site for ten years. Electronic copies of the sample biographical data

and their results are backed-up daily, and each month one of those tapes is archived. Electronic results and biographical data are available from the LIMS system for at least ten years. Electronic data, including methods, data files and sequence logs is backed up at least every night, archived on suitable media and retained indefinitely.

8.4.2. Manuals, Methods and SOPs

Current Manuals, SOPs, and other supporting documents, such as IDC/MDL studies and program specific QAPPs are readily available to all personnel. Any archived items will also be available if required. Each item will be clearly labeled and dated, so that it is apparent when the document was in force.

Although printed hard-copy versions of Department SOPs are available, all "official" and current SOPs are kept in an electronic format (MSWord or HTML) and will be available to all staff members as "read-only" documents. Hardcopies will be made available to Laboratory customers or to regulatory agencies if requested.

SOPs (analytical and administrative) are revised when necessary, due to instrument/technology changes, regulatory changes, etc. Major changes, such as those noted above, will result in a new revision of the SOP. Each new revision will have a new revision number, an effective date, and will list the SOP it is replacing. In general if minor semantic or typographical changes are made a new revision number will not be used.

IDC and MDL studies may be found in the Department file cabinets and in archive (generally for records older than two years) file cabinet in the basement. Each set of MDL data is also included in the appropriate analytical SOP in the form of LOD and LOQ tables.

Occasionally, at the request of a client, the laboratory will prepare a Quality Assurance Project Plan (QAPP). These documents are generally kept with the actual contract and other historical information associated with the project.

8.4.3. Logbooks

The laboratory utilizes logbooks for various purposes including: instrument maintenance, instrument injection or run logs, cataloging of standards and their preparation, monitoring sample movement for chain of custody or enforcement purposes, and temperature record-keeping for ovens, refrigerators, freezers and walk-in coolers. These logbooks are clearly and uniquely labeled as to be easily associated with their purpose.

8.5. Radiochemistry

8.5.1. Sample Receipt and Distribution

Samples are received from the WDNR, DHFS, local public health officials, private citizens, and other sources. Samples are initially received at the Specimen Receiving Department located at the north end of the building (see ESS INO GENOP 106). The sample containers and test request forms (TRFs) are removed from their shipping containers. The TRFs are labeled with the laboratory ID number, and the sample containers are given a barcode label with the corresponding laboratory ID and a unique letter identifier. Information regarding date and time of receipt, preservation and/or thermal status (if required), sample condition, and type of sample container(s) received, is recorded in the sample tracking software (see EHD GENOP 024). The samples are then delivered to the Radiochemistry laboratory sample receipt area where they are checked in for processing. Once check-in is complete the samples are placed in the appropriate location for storage until analysts remove them for analysis.

A test request form is normally submitted with each sample. This form contains the sample identification, collection information, and indicates which analyses are required. If a form is not provided by the client, this information is recorded on a sheet of paper by Radiochemistry Staff. The samples are held for a minimum of 4 weeks from the date which the results were reported.

8.5.2. Sample Tracking

Detailed sample information is then entered into the WSLH main frame Data General (DG) computer using the "SAMPLE CHECKIN" option of the menu-driven "WORK" program on DG. No samples are rejected without first contacting the client and advising them of the situation.

All sample information is stored in the */slh/grp/radprot/vir* directory on DG. The information can be accessed using the "VIR" and "LOOKAT" options of the "WORK" program

8.5.3. Data Review

Worksheets are reviewed by the analyst and approved by the department supervisor. The approval process includes matching worksheet data with data transferred to the calculation program manually or through electronic input. The QC results are reviewed to determine if protocols for reporting a run have been met. After all of the analyses have been completed and approved, the hard copy report is printed and reviewed by the analyst and approved by the department supervisor. This approval process includes checking collection dates and times, reporting and billing addresses, proper departments, PWSID number, sample description information, comments, and reasonableness of the results. If a PWSID number is provided with the sample, the current analytical results are compared to the history of that system. Samples which do not compare with previous history are repeated or the client is contacted to determine if the sample was treated differently (i.e., soft, rather than hard, water

collected). As a final check, the department supervisor matches the reported results against the data in the VIR records. Spot checks are made at this point to make sure that the data from the calculations is going into the VIR records correctly.

Yearly, a complete hand calculation of programs is performed to verify results. If the program is altered during the year another verification is performed. Spot checks of different parts of the program are performed throughout the year. Documentation of program verification is kept in the QC lab notebook and in the computer verification file in the file cabinet.

8.5.4. Data Reporting

8.5.4.1. The analyses required for a particular sample are indicated by the absence of a "0" in the "DATE REPORTED" field in formats 2, 3 and 4.

8.5.4.2. When the results of a chemical analysis are calculated, the results are automatically entered in the proper fields in formats 2 and 3 by the various calculation programs.

8.5.4.3. After the completion of the gamma analysis, the results must be transferred from the Aptec Gamma Counting System to DG by running the "APTEC"option under "INSTRUMENTATION" using the "WORK" program. The gamma results are entered into format 4.

8.5.4.4. The identification numbers of the associated QC samples for each analysis are entered into format 5.

8.5.4.5. Hard copies of testing results are provided to the submitter of the sample.

8.5.5. Data Retention

8.5.5.1. Paper

All hard copies of records will be placed in State Records Center storage boxes, and will be kept for a minimum of two years in the hall storage area. After two years the records can be moved to the basement storage area where they will be held indefinitely.

8.5.5.2. Electronic

The Radiochemistry Information Management System (RIMS) is used to support the laboratory tracking, testing and reporting processes for this department.

System Description

The Radiochemistry Information Management System uses the WSLH Data General computer and software developed by WSLH personnel.

Primary Software

VIR and its related programs are used to store and retrieve sample information

Results

Laboratory results are electronically transferred from instrument data systems utilizing interactive programs for calculating, monitoring and editing result information before it enters the RIMS database. Instruments use data buffers and PC based data systems to transfer count information.

Magnetic Backup

Daily backups of the entire contents of all system disks (except the temporary volume) are created and verified between 1:00 and 6:00 AM daily, Monday through Saturday, and saved for six weeks. The first back up of the month for January and July are saved for the life of the tape.

Outputs

Bench sheets (worklists) are generated for analyst use and subsequent results and quality control entry. Bench sheets remain with the paper archive of each sample.

8.5.6. Manuals, Methods and SOPs

Current Manuals, SOPs, and other supporting documents are available to all Radiochemistry staff in both hard copy and electronic versions. Any archived items will also be available if required. Each item will be clearly labeled and dated, so that it is apparent when said document was in force.

Currently manuals are reviewed on an annual basis and updated if necessary. Changes which occur in-between time are documented and promulgated to the necessary staff members. In addition, a hard copy of the new SOP is placed in the "official" Department SOP binder.

In the future, printed hard-copy versions of Department SOPs will no longer be available. All "official" and current SOPs will be kept in an electronic format (MSWord or HTML) and will be available to all staff members as "read-only" documents. Hardcopies will be made available to Laboratory customers or to regulatory agencies if requested in writing.

SOPs (analytical or administrative) are revised when necessary, due to instrument/technology changes, regulatory changes, etc. Major changes, such as those noted above, will result in a new revision of the SOP. Each new revision will have a new revision number, an effective date, and will list the SOP it is replacing. In general if minor semantic or typographical changes are made a new revision number will not be used.

8.5.7. Logbooks

The laboratory utilizes logbooks for various purposes including instrument maintenance, instrument run logs, cataloging of standards and their preparation, and temperature record keeping for ovens and refrigerators. Currently most logbooks are still the hardbound/binder type. However, an effort will be made toward establishing a uniform electronic logbook where practical.

8.6. Water Microbiology

8.6.1. Information Flow, Data Retrieval and Storage

8.6.1.1. Sample Receipt and Distribution

All samples received at the Laboratory are accompanied by a test request form (TRF). Each TRF is date stamped, and given a unique number. The date, time received, unique number, temperature, comments, type of sample container, number of bottles for TRF, preservation, and initials are logged into a sample tracking database specifically for Environmental Sciences. In addition, samples that have temperature specific requirements will have their temperatures taken and recorded. All pertinent sample information is then entered into the Laboratory Information Management System (LIMS) database. The LIMS database automatically tracks sample status, entry, and modification times on a per result basis.

Once samples are received in the Water Microbiology Department the test request form is checked by the microbiologists and laboratory technicians for sample validity, holding times and tests. Qualifiers will be placed on the TRF if necessary. Sample information (PWS ID, collection date and time of each sample, date received, collectors name, location of samples and lab number) is then entered into the Laboratory Information Management System (LIMS) database. Samples are analyzed. After inoculation the sample numbers, the time the samples were put into the incubator and the test method are recorded in a permanent logbook.

8.6.1.2. Data Review and Reporting

Results are recorded by the microbiologists and laboratory technicians on the test request form along with any data qualifiers. The support staff enter the results into the Laboratory Information Management System (LIMS) database. The results, biographical information, and qualifiers are doubled checked by support staff for accuracy. If no errors are found results are released.

Results are available in hard copy reports, which may be sent to the applicable parties (for SDWA and private water samples) or filed (enforcement samples) or both. In addition results are available to various external agencies (primarily the WDNR) in electronic format. Hardcopies

of all worklists, worksheets, calibrations, and raw data are currently kept on site for at least five years.

8.6.1.3. Data Retention

The TRFs that are submitted with samples are sent to the WDNR for SWDA samples. The TRFs for private customers are sent back to the customer along with the report. The lab retains a copy of the field sheet for enforcement samples. All records are maintained for a minimum of five years. Samples, which have the potential of going to litigation, should be held indefinitely. Electronic copies of the sample biographical data and their results are backed-up daily and each month one of those tapes is archived. Electronic data is available on-line for one year.

8.6.2. Manuals, Methods and SOPs

Current Manuals, SOPs and other supporting documents are available to all personnel. Both a hard copy and an electronic copy are kept. All department SOPs are reviewed each year and any changes which occur in-between time are documented and promulgated to the staff. Each new revision will have an effective date indicating when the document was in force. In addition a hard copy of the new SOP is placed in the official department SOP manual. The official copy of an SOP is the electronic copy.

8.6.3. Logbooks

The laboratory utilizes logbooks for various purposes including positive and negative controls for media and reagents, bottle sterility, instrument maintenance, instrument run logs, cataloging of standards and their preparation, monitoring sample movement for chain of custody or enforcement purposes, and temperature record-keeping for ovens, refrigerators, freezers and walk-in coolers. The logbooks are kept for a minimum of 5 years.

Figure 8.1 — Information and Data Flow for the Inorganic Chemistry Department

1. Samples are received, data is recorded (i.e., temperature), and ID #s assigned

2. Samples are logged into the sample tracking system

3. Samples are logged into LIMS: ID #s, acct. #s, and analytical & prep tests

4. Biographical data is verified by a data management staff member

5. Requested tests and analytical testing data are verified by a supervisor

6. A worklist is generated

7. Analytical testing is performed

8. QC Limits are checked - QA worksheets are created and completed

- a) If limits are OK proceed to next step.
- b) If QC fails:
  - i) Re-analyze the samples (go back to step 6) or
  - ii) Qualify results and proceed to next step

9. Senior analyst or supervisor review of data

10. Results are transferred to LIMS database

- a) Electronically from the instrument or
- b) Hand entered by data management staff

11. Data is reviewed by Supervisor

12. Results are verified

13. Data is evaluated by a number of LIMS programs

14. Is the data acceptable?

- a) If Yes proceed to next step.
- b) If No check for errors and possible re-analysis

15. Results are made available to clients via telecommunications and mailed hardcopy

Figure 8.2 — Information and Data Flow for the Organic Chemistry Department

1. Samples are received, data is recorded (i.e., temperature), and ID #s assigned

2. Samples are logged into LIMS: ID #s, acct. #s, and analytical & prep tests

3. Samples are logged into the sample tracking system

4. A worksheet is generated

5. Analytical testing is performed

6. QC Limits are checked - QA worksheets are created and completed

- **a.** If limits are OK proceed to next step.
- **b.** If QC fails:
  - **i.** Re-analyze the samples (go back to step 5)
  - **ii.** Qualify results and proceed to next step

7. Results are transferred to LIMS database

- **a.** Electronically from the instrument
- **b.** Hand entered by the analyst

8. Senior analyst or supervisor review of data

9. Data is reviewed by Supervisor

10. Results are verified

11. Data is evaluated by a number of LIMS programs

12. Is the data acceptable?

- **a.** If Yes proceed to next step.
- **b.** If No check for errors and possible re-analysis

13. Results are released and made available to clients via telecommunications and mailed hardcopy

Figure 8.3 — Information and Data Flow for the Water Microbiology Department

1. Samples are received, data is recorded (i.e., temperature), and ID #s are assigned in the Sample Tracking System.

2. Samples are checked for sample validity and tests.

3. Analytical testing is performed simultaneously with samples being logged into LIMS: ID #s, Acct #, and analytical tests.

4. Results and biographical information are entered into LIMS and double-checked for accuracy.

5. Results are available to clients via telecommunications and mailed hardcopy

Figure 8.4 — Information and Data Flow for the Radiochemistry Department

1. Samples are Received, Data Recorded, and ID #s assigned
2. Samples are logged into RIMS: sample #s, acct. #s, and analytical & prep tests
3. A worklist is generated
4. Analytical testing is performed
5. Results are calculated and automatically entered into RIMS
6. QC Limits are checked
    a. If limits are OK proceed to next step.
    b. If QC fails:
        i. Re-analyze the samples (go back to step 4)
        ii. Qualify results and proceed to next step
7. Analyst and supervisor review the data
8. Results in RIMS database may be reported.
9. Report is reviewed by the supervisor
10. Is the data acceptable?
    a. If Yes proceed to next step.
    b. If No check for cause and possible re-analysis
11. Results are made available to clients via telecommunications and mailed hardcopy

9. Procedures for Accepting New Work

The Strategic Leadership Team has written a lab-wide policy for offering new tests to the public that can be accessed via the State Laboratory of Hygiene internal website at: http://intranet.slh.wisc.edu/policy/contract/newTestProcess.html

A draft SOP, EHD GENOP 027 SOP Policy on Board Approval for accepting work.doc, is available which is specific for the Division of Environmental Health.

9.1. Biomonitoring

New work is discussed at the biweekly meeting of the Biomonitoring/DNR team. Laboratory capabilities, workload and personnel are discussed and the proposed new work is either accepted or rejected.

9.2. Inorganic Chemistry

When considering new work for the Inorganic Chemistry Department, the management group (Department supervisor, supervisory staff, and in some cases the Director) determines if the work is within the laboratory's mission and statutory authority. The work should come from another state agency (e.g., WDNR, DHSS), a municipality, the University of Wisconsin system, or Federal government agencies. It must also be determined if the laboratory has the desired methodology, experience, and facilities to complete the work.

If the work is to be accepted, an assessment is made of existing instrumentation. For example, will existing instrumentation meet the detection limits specified in the data quality objectives, and does ample capacity exist? Management will then assess whether there is sufficient staffing to accommodate the new workload by consulting with key analysts from the affected area. If more staff or instrumentation is needed to process the new work, request forms must be filled out and approved by the Director. The affected department must also meet with key support staff in other departments that may be impacted by the new work. Finally, the management staff must determine the revenues and expenses anticipated from the new work and make appropriate budget adjustments. A contract for services is completed, stating expected costs and expected deadlines. Once the contract is signed by the appropriate officials from both the laboratory and the client, the work may commence.

9.3. Organic Chemistry

All requests for new work are reviewed by the Department Supervisor and occasionally by the Division Director. In most cases the request will come from a governmental agency (WDNR, DHHS, USGS, etc.) and will ask for analyses that are currently done by the laboratory. Depending on projected sample load, the work will be accepted and a contract will be written (or an account number assigned). In some cases, however, an agency will request that a new test be done, or that a new analyte be added to an old test. In such a case there are a number of criteria that will be considered before the acceptance of samples.

First, the Department Supervisor will decide if the laboratory can accept the work within the constraints of a state institution. Because of the governmental/public service nature of the WSLH, decisions to accept or reject work are often more complex than they would be in the private sector. On one hand the WSLH must fulfill its role as the state's public health laboratory, accepting those samples which are necessary to ensure the public health. On the other hand we must still function in a fiscally responsible manner, in which real costs for analysis must be recovered.

With the above restrictions in mind the Department Supervisor will assess the nature of the proposed analyses. If the work is of a research nature, or is not being performed by any of the private environmental laboratories in the state, it can be considered. In addition if another governmental agency specifically requests analysis at the WSLH (e.g., in cases of environmental enforcement) the work will be taken into consideration. Lastly, in cases of immediate environmental hazard (e.g., a spill), the laboratory will make every effort to conduct the necessary analyses.

Once a determination of acceptability has been made, the Department Supervisor will consult with the analysts in the affected area. Together they will decide whether such analysis is possible within the bounds of current workload and analytical equipment. If the current equipment is inadequate, and the work being requested is considered a priority (e.g., a matter of public health), it is possible that such equipment would be purchased by the laboratory. It should be noted that some requests also involve a fiscal penalty for the WSLH (i.e., costs of analyses are not fully recoverable). The decision to accept "money losing" work or to purchase required equipment will be made primarily by the Division Director.

Once laboratory capability has been established, method development will commence. If a recognized (i.e. validated) method already exists then it will be pursued for use in the laboratory. It must be noted, however, that requested analysis will often fall into the unregulated category, where no "standard" method exists (e.g., analyzing for pesticides in human serum). In such cases, the analyst will look to modify existing methods, survey research journals, consult with peers and generally rely on their experience to develop a method. If the method succeeds (see validation below) then a standard contract will be written and samples will be accepted. Any costs associated with developing the method will generally be covered by the requesting agency.

Each new method that is developed (including new methods that are created and existing methods that the laboratory is using for the first time) will be validated in a number of ways. Consideration will be given to the limits of detection (LOD), limits of quantification (LOQ), method biases (including instrumental and matrix factors), method accuracy, precision, and equivalency.

The statistical LOD (also referred to as the method detection limit or MDL) and the LOQ are determined using the procedure outlined in 40CFR part 136 (appendix A & B) and Analytical Chemistry (volume 55, 1983) respectively. The method LOD and LOQ may be identical to the generated statistics or alternatively, a common sense approach may be employed (see ESS INO QA 116). If common sense is used the statistical measurements will serve as a floor (i.e., no common sense LOD can be set

at a lower concentration than the statistical LOD). The common sense approach will take into account such factors as "real" instrument capability, signal-noise ratio, spectrum quality, quantitation accuracy at or near the LOQ, etc. If a common sense approach is used it will be noted in the method SOP. The reasoning will be recorded with the LOD study data.

In addition to LODs/LOQs an Initial Demonstration of Capability (IDC) will be performed to gauge method accuracy (see ESS INO QA 115). IDC acceptance criteria for a number of various analytes can be found in the "Wisconsin Laboratory Certification & Registration Program" document (WDNR PUBL-TS-007-98). When developing an unregulated method these criteria will be used as a guideline for method acceptability.

If another "approved" method is available, or if approval is being sought for an alternate technique, equivalency testing will be performed. This testing is done following the guidelines outlined in "Proposed System for Determining NPDES and NIPDWR Method Equivalency" (USEPA, May 1978). Equivalency testing generally involves analyzing a variety of sample matrices in replicate (minimum of four) by each method. The data are then subjected to a variety of statistical tests including the F-test, the students t-test, standard deviation, etc. These tests are used to determine whether the two methods are significantly different. The new test method must be equivalent or better than the approved method before an application for approval can be submitted. Although not required, equivalency testing is also useful when comparing two approved methods.

In general, routine quality control guidelines will be established during the method development process. In some cases however, the client may request the development of a separate quality assurance project plan (QAPP). If such a request is made, the analyst, along with the Department Supervisor, and the client, will develop a QAPP that meets the needs of the client. In general the QAPP itself, or references to it, will be included in the contract.

9.4. Radiochemistry

9.4.1. Sediment dating

The WDNR will determine the sample testing priorities for this analysis. No new work will be accepted from another client without approval from WDNR.

9.4.2. Nuclear Surveillance Program

The DHFS Radiation Protection Department determines the sample load for this program. Since the workload from DHFS is on the decline, work from other agencies may be accepted provided all testing requirements for DHFS samples can be maintained. There is an agreement in place between neighboring states to help analyze excess samples in the event of a nuclear disaster.

9.4.3. Other

Requests for non-standard work will be reviewed on a case-by-case basis by the staff and department supervisor.

Radiochemistry accepts all work on a fee for service basis provided the samples would be analyzed by our standard procedures. Clients from out of state are advised of our certification status and it is left to the client to determine if that meets their needs.

The Radiochemistry Department maintains a list of states in which we are allowed to perform radon testing in air. If kits are to be sent to an out of state client, the client is provided with this list.

9.5. Water Microbiology

All requests for analysis are reviewed by the Department Supervisor and occasionally by the Division Director. In most cases the request will come from a governmental agency (WDNR, DHHS, USGS, etc.) and will ask for analyses that are currently done by the laboratory. Depending on projected sample load, the work will be accepted and a contract will be written. In some cases, however, an agency will request that a new test be done, or that a new analyte be added to an old test. In such a case there are a number of criteria that will be considered before the acceptance of samples.

First, the Department Supervisor will decide if the laboratory can accept the work within the constraints of a state institution. Because of the governmental/public service nature of the WSLH, decisions to accept or reject work are often more complex than they would be in the private sector. On one hand the WSLH must fulfill its role as the state's public health laboratory, accepting those samples which are necessary to ensure the public health. On the other hand we must still function in a fiscally responsible manor, in which real costs for analysis must be recovered. The decision to accept "money losing" work or to purchase required equipment will be made primarily by the Division Director.

Once a determination of acceptability has been made, the Department as a whole will decide whether such analysis is possible within the bounds of current workload and analytical equipment. If the current equipment is inadequate, and the work being requested is considered a priority (e.g., a matter of public health), it is possible that such equipment would be purchased by the laboratory.

10. Sample Handling and Submission Procedures

10.1. Biomonitoring

Procedures for sample collection are contained in ESS BIO GENOP 15 ("Sample Collection for Bioassay Toxicity Tests"). Procedures for sample submission, receipt, check-in (including Chain-of Custody procedures), sample rejection, and sample disposal are included in ESS BIO GENOP 20 (Sample Receiving/Check-in/Disposal for Bioassay Toxicity Tests"). All SOPs are contained in a bound manual, stored in the laboratory and are available to all employees.

10.2. Inorganic Chemistry

10.2.1. Record Keeping

All submission bottles sent out by the laboratory undergo routine quality control checks. Records and data for all bottle checks are kept in the laboratory. For a detailed description of the bottle check procedure see ESS INO QA 101.

10.2.2. Sample Receipt

Samples arrive at the laboratory in several ways. They may be mailed in, brought to the laboratory by client field representatives, or brought to the front desk of the laboratory by the general public. Each sample received is accompanied by a test request form (TRF). Please see ESS INO GENOP 106 for sample receipt details.

Samples pass through the sample receiving area where they are assessed for acceptability (e.g., proper temperature, proper preservation). If a sample arrives showing definitive signs of contamination or if the container type nullifies any meaningful analysis (e.g., oil and grease in a plastic bottle), the sample will not be analyzed. In general, however, samples will not be rejected. If a sample is compromised the collector will be consulted and any data generated from that sample will be appropriately qualified. Any SDWA sample that does not meet the necessary requirements will be rejected and re-sampled. If the second sample is also compromised it will be analyzed (per State of Wisconsin program requirements) and reported with a qualifier. Please see ESS INO GENOP 103 for the Sample Acceptance Policy for the Inorganic Chemistry Dept.

All samples are entered into the ESS Sample Tracking database (EHD GENOP 024) along with the following information: temperature, date received, initials of the receipt analyst, the number of containers received, the type of containers received, field acidification or preservation (if any), lab acidification or preservation (if any), the location for storing the sample, and any particular pertinent comments about the sample. Labels, with unique lab numbers and barcodes, are printed for each sample container and test request form. All sample containers making up one sample are assigned the same root lab number along with individual letter identifiers.

Once assessed, and labeled, the samples are taken to their proper storage areas in the appropriate section of the laboratory to await analysis or may be analyzed immediately. All pertinent paperwork is given to data management staff and entered into the Laboratory Information Management System (See Chapter 8).

10.2.3. Sample Handling

Once a sample has been received it undergoes the process described in Chapter 8 — Documentation Procedures. The sample is procured from storage by the analyst and undergoes the testing procedure. Each testing procedure is described in detail in the individual method SOP.

When taking a sub-sample for analysis the sample is homogenized and the aliquot is removed employing appropriate laboratory techniques. Liquid samples are mixed well and volumetric pipettes (wide bore blowout type for samples with particulate) are used to remove an aliquot. Solid samples are dried and ground if allowed by method. If a sample is multi-phasic and requires TCLP it will be handled as prescribed in the EPA method. For all other analyses of multi-phasic samples the collector should be contacted for direction on how to proceed. Some test procedures will consume the entire sample or compromise the remaining sample by removing an aliquot. If the sample is not compromised it will be returned to the proper storage area as soon as possible. When a sample is used up, broken, disposed or its status is otherwise altered, this is noted in the Sample Tracking database, along with the date and the analyst's initials.

10.2.4. Sample Storage and Disposal

Samples are stored in a variety of places depending on the requirements of the method and the regulatory program. They may be kept in refrigerators, walk-in coolers, freezers, or in other designated areas of the laboratory.

Routine non-enforcement samples are kept until results for those samples are released and may be kept longer if necessary. Samples used for environmental enforcement action are to be kept until the proper enforcement entity authorizes their disposal (see section 10.2.5.4).

Samples are disposed of in a manner which is consistent with the nature of the sample and the applicable rules and regulations (see ESS INO GENOP 110). Waste disposal guidelines are described in the University of Wisconsin Chemical Safety and Disposal Guide.

10.2.5. Law Enforcement/Chain-of-Custody Procedures

Chain-of-custody for law enforcement samples is a means of demonstrating the traceability of a sample from the time of collection through its introduction as evidence in a law enforcement case. This is accomplished by maintaining an accurate written record that may be used to physically trace a sample from the

moment of collection through transportation, storage, analysis, and disposal. In some cases, partial chain-of-custody procedures (through sample receipt at the laboratory) are used for non-enforcement samples at the request of individual study coordinators. For more details please consult ESS INO GENOP 106, section 4.

Complete chain-of-custody is only possible through the cooperative effort of both laboratory and field personnel. The procedures used by the laboratory are described in the following section. The responsibilities of the sample collector and chain of custody procedures used by field personnel are described in the Wisconsin Department of Natural Resources Field Procedures Manual.

10.2.5.1. Transfer of Law Enforcement/Chain-of-Custody Samples to the Laboratory

These samples may be transported to the laboratory by two means:

The sample collector may keep the samples in his/her physical possession and deliver them directly to the laboratory.

The collector may seal the samples in a field pack (shipping container) and ship the samples to the laboratory by U.S. mail.

10.2.5.2. Physical Transfer

Laboratory personnel will receive chain-of-custody samples, identify each sample container with a unique laboratory number and the date received, and complete a Chain-of-Custody record. The record (Figure 10.1) will include the following information: 1) field identification number, 2) laboratory number; 3) description of the sample; 4) the name, title, work station and telephone number of the person delivering the samples; 4) the time (hour and minute) and date the samples are received in the laboratory; 6) the disposition of the unused portion of the samples remaining after the analyses; and 7) the signature of the laboratory employee receiving the samples. The original receipt will be given to the person that delivered the samples and a copy will be retained by the laboratory.

10.2.5.3. U.S. Mail Transfer

The collector will place the samples in a Laboratory of Hygiene Styrofoam field pack (shipping container) and seal with reinforced nylon tape. The ends of the tape are kept straight so they overlap slightly. Using a waterproof pen, the collector will write "Enforcement Case", his/her name, date, time and sample identification on the tape where the ends overlap. The collector will also write, "To be opened by Inorganic Chemistry personnel only" on the tape. When the field pack is received at the Laboratory of Hygiene, Inorganic Chemistry personnel will examine the seal to make sure that it has not been tampered with. He/she will then open the field pack, remove the samples and laboratory sheets, and write his/her initials, the time and date on the bottom of the laboratory sheets

and on the accompanying Chain of Custody record sheet. The samples are then assigned a unique laboratory number and processed according to Chapter 8.

10.2.5.4. Handling of Law Enforcement (and other Chain-of-Custody) Samples in the Laboratory

After chain-of-custody samples have been properly transferred to the laboratory's possession, the laboratory data sheets and sample bottles are labeled with a red "COC" ink stamp. This indicates that the samples should have special handling. The employee that receives the samples will distribute them to the appropriate laboratory personnel to perform the analyses, or place them in one of the secured law enforcement coldrooms (rooms 119C, 217A, 217B, 219C and the 217E freezer). Only authorized personnel, through the use of key-card activated electronic locks, have access to the enforcement coldrooms. In addition, key-cards will enable Capitol Police and Security to maintain an electronic record of all entries to enforcement coldrooms

Inorganic Chemistry personnel will open the COC samples, acidify samples as required, and verify the pH. In addition, Inorganic Chemistry personnel will determine if a COC sample is also a law enforcement sample (see ESS INO GENOP 106, section 4.9). If the sample is an enforcement sample, Inorganic personnel will label the labslip and bottles with an "ENF" stamp. The enforcement sample bottles will be placed in line in the enforcement section of the coldroom (room 119C). The enforcement samples will receive special handling as noted below. If the COC sample is not an enforcement sample, it will be placed in line along with routine samples.

Special handling for enforcement samples includes using the ESS Sample Tracking database to "track" the samples in and out of the locked enforcement coldroom (room 119C) when they are being analyzed. After the sample aliquot is removed from the container or at the end of a normal workday, the unused portions of the samples are returned to the law enforcement coldroom (rm. 119C). It is the responsibility of the analyst to maintain possession of law enforcement samples during times that the samples are out of the coldroom for analysis.

Upon completion of all tests, a sample disposition form (Figure 10.2) is mailed to the sample collector along with a computer-generated report. Unless otherwise specified on the Chain of Custody record sheet, or in a telephone conversation, the sample collector should return the disposition form to the laboratory within 90 days. If the disposition form is not returned within the 90-day period an ACCESS program is run to generate a list of all completed enforcement samples that are stored in the walk-in. A memo is sent to all collectors on the ACCESS list asking for confirmation of disposition and another 90-day deadline is given. If the memo is not answered within that time period, a second memo is sent with

a two-week deadline. If the second memo is not responded to after two weeks, the collector is contacted by phone by the WDNR liaison. At this time the collector must approve disposal, otherwise the laboratory will retain the samples in their secured enforcement coldroom until the case is settled or the sample collector authorizes disposal of the sample. After another 90 day time period the process is repeated. Once permission is given to dispose, the date of disposal is recorded in the ACCESS database and the sample is disposed in an appropriate manner.

10.3. Organic Chemistry

10.3.1. Record Keeping

All submission bottles sent out by the laboratory undergo routine quality control checks. These checks may be performed by the manufacturer (for new bottles) or by the laboratory (for re-used bottles). Records and data for all sample bottle quality control checks are kept in the laboratory. The Organic Chemistry Department keeps the data, including sample and standard chromatograms, in specific folders. (See ESS ORG QA 0010)

10.3.2. Sample Submission

Samples arrive at the laboratory in several ways. They may be mailed in, brought to the laboratory by client field representatives, or brought to the front desk of the laboratory by the general public. Currently all samples pass through the sample receiving area where they are assessed for acceptability (e.g., proper temperature) and assigned a unique laboratory number. If receiving personnel have any questions about acceptability of a sample, they will consult with Department personnel before proceeding.

If a sample arrives showing definitive signs of contamination or if the container type nullifies any meaningful analysis (e.g., semi-volatiles in a plastic bottle), the sample will not be analyzed. In general, however, samples will not be immediately rejected. If a sample is compromised the collector will be consulted and any data generated from that sample will be appropriately qualified. However, any sample used for SDWA compliance that does not meet the necessary regulatory requirements will be rejected and re-sampled. If the second sample is also compromised it will be analyzed (per State of Wisconsin program requirements) and reported with a qualifier. See ESS ORG GENOP 0028.

Each sample received is accompanied by an informational sheet (an Organic Chemistry test request form, WDNR field sheet, WSLH labslip, or other project specific form) which is date stamped, and labeled with a unique laboratory number assigned to that sample. In the case of samples comprised of multiple containers, each container will receive a unique number, which is tied directly to the parent sample number. Also at this time samples with temperature specific requirements have their temperatures taken, recorded, and initialed. Once assessed, and labeled, the samples are taken to their proper storage areas in the appropriate section of the laboratory to await analysis. All pertinent

paperwork is given to Department personnel and entered into the Laboratory Information Management System (See Chapter 8).

All samples, as well as being entered in the LIMS database, are entered into the ESS Sample Tracking database using the following information: Unique sample ID number, temperature, date received, initials of the receipt analyst, the number of containers received, the type of containers received, field acidification or preservation (if any), lab acidification or preservation (if any), the location for storing the sample and any particular pertinent comments about the sample. A unique laboratory number and associated barcode is assigned to each sample container (in conformance with current NELAC guidelines).

When a sample is removed from storage (for analysis or any other reason) for any length of time, its unique sample ID label is scanned into the ESS Sample Tracking database and recorded as being removed, noting the date and the initials of the analyst. This also occurs when the sample container is returned. If it is used up, broken, disposed or its status is otherwise altered, this is noted in the database, again including the date and the analyst's initials.

10.3.3. Sample Handling

Once a sample has been received it undergoes the process described in Chapter 8 — Documentation Procedures. The sample is procured from storage by the analyst and undergoes the testing procedure. Each testing procedure is described in detail in the individual method SOP.

When taking a sub-sample for analysis the sample is homogenized and the aliquot is removed employing appropriate laboratory techniques. Liquid samples are mixed well and volumetric pipettes (wide bore blowout type for samples with particulate) are used to remove an aliquot. Solid samples are dried and ground if allowed by method. If a sample is multi-phasic and requires TCLP it will be handled as prescribed in the EPA method. For all other analyses of multi-phasic samples the collector should be contacted for direction on how to proceed. Some test procedures will consume the entire sample or compromise the remaining sample by removing an aliquot. If the sample is not compromised it will be returned to the proper storage area as soon as possible.

10.3.4. Sample Storage and Disposal

Samples are stored in a variety of places depending on the requirements of the method and the regulatory program. They may be kept in refrigerators, walk-in coolers, freezers, or other designated areas of the laboratory.

Routine non-enforcement samples are kept until results for those samples are released and may be kept longer if necessary. Samples used for environmental enforcement action will be kept until the proper enforcement entity authorizes their disposal. The only exception to this is those samples whose nature makes it meaningless to keep them (e.g., VOCs in water). These samples will be kept for two weeks past their holding times and then disposed.

Samples are disposed of in a manner which is consistent with the nature of the sample and the applicable rules and regulations. Waste disposal guidelines are described in the University of Wisconsin Chemical Safety and Disposal Guide and the Department Sample Disposal SOP ESS ORG GENOP 0029.

10.3.5. Chain-of-Custody Procedures

Chain-of-Custody (chain-of-possession) is a means of demonstrating the traceability of a sample from the time of collection through its introduction as evidence in a law enforcement case. This is accomplished by maintaining an accurate written record that may be used to physically trace a sample from the moment of collection through transportation, storage, analysis, and disposal.

Complete chain-of-custody is only possible through the cooperative effort of both laboratory and field personnel. The procedures used by the laboratory are described in the following section. The responsibility of sample collector and chain of custody procedures used by field personnel are described in the Wisconsin Department of Natural Resources Field Procedures Manual.

10.3.5.1. Transfer of Law Enforcement Samples to the Laboratory

Enforcement samples may be transported to the laboratory by two means:

The sample collector may keep the samples in his/her physical possession and deliver them directly to the laboratory.

The collector may seal the samples in a field pack (shipping container) and ship the samples to the laboratory by U.S. mail.

10.3.5.2. Physical Transfer

Department personnel will receive law enforcement samples, identify each sample container with a unique laboratory number and the date received, and complete a chain of custody record that was initiated by the sample collector. The COC record (Figure 10.1) includes the following information: 1) field identification number, 2) laboratory number; 3) description of the sample; 4) the name, title, work station and telephone number of the person delivering the samples; 4) the time (hour and minute) and date the samples are received in the laboratory; 6) the disposition of the unused portion of the samples remaining after the analyses; and 7) the signature of the laboratory employee receiving the samples. The original COC record will be given to the person that delivered the samples at the time of delivery, or it will be sent with the test report when the analysis is complete. In either case a copy will be retained by the laboratory.

10.3.5.3. U.S. Mail Transfer

The collector will place the samples in a Wisconsin State Laboratory of Hygiene Styrofoam field pack (shipping container) and seal with reinforced nylon tape. The ends of the tape are kept straight so they overlap slightly. Using a waterproof pen, the collector will write

"Enforcement Case", his/her name, date, time and sample identification on the tape where the ends overlap. The collector will also write, "To be opened by Organic Chemistry personnel only" on the tape. When the field pack is received at the Laboratory of Hygiene, Organic Chemistry personnel will examine the seal to make sure that it has not been tampered with. He/she will then open the field pack, remove the samples and laboratory sheets and write his/her initials, the time and date on the bottom of the laboratory sheets and on the accompanying Chain of Custody record sheet. The samples are then assigned a unique laboratory number and processed according to Chapter 8.

10.3.5.4. Handling of Law Enforcement Samples in the Laboratory

After law enforcement samples have been properly transferred to the laboratory's possession, the test request form and sample bottles are labeled with a red "ENF" ink stamp. This indicates that the samples are for law enforcement activities and should have special handling. The employee that receives the samples will distribute them to the appropriate laboratory personnel to perform the analyses, or place them in one of the secured law enforcement coldrooms (rooms 119C, 217A, 217B, 219C and the 217E freezer).

It is the responsibility of the analyst to maintain possession of the samples at all times from the moment he/she receives the samples until the analyses are completed and the samples returned. The analyst will perform the requested analyses and record all pertinent information on the laboratory worksheet before returning the samples to the person that distributed them or to the designated secured area. After the sample aliquot is removed from the container or at the end of a normal workday, the unused portion of the samples is locked in the appropriate coldroom.

Currently only authorized personnel, through the use of key-card activated electronic locks, have access to the enforcement coldrooms. In addition, key-cards will enable Capitol Police and Security to maintain an electronic record of all entries to enforcement coldrooms.

Upon completion of all tests, a sample disposition form (Figure 10.2) is mailed to the sample collector along with a computer-generated report, the original test request form, and the original COC record form. Copies of these forms are retained on file at the laboratory. Unless otherwise specified on the Chain of Custody record sheet, or in a telephone conversation, the sample collector must return the disposition form to the laboratory within 90 days or the sample will be discarded. Otherwise the laboratory will retain the samples in their secured enforcement coldroom until the sample collector authorizes disposal of the sample.

It must be remembered that the above disposal process applies only when sample integrity is not compromised by the passage of time. Samples for which holding times are analytically critical will be kept for two weeks past the holding time and then disposed. These samples are not kept

because once the holding time passes they no longer have any analytical validity.

In either case, the laboratory supervisor will indicate the name of the official that authorized disposal of the sample, and the date on the sample disposition form before discarding the sample.

10.4. Radiochemistry

10.4.1. Record Keeping

All submission bottles sent out by the laboratory undergo routine quality control checks. These checks are performed by the manufacturer. Records and data for all sample bottle quality control checks are kept in the laboratory. The Radiochemistry Department keeps the data, including sample and standard data in specific folders.

Procedures for sample collection are described in individual analytical method SOPs where applicable. Often other State agencies have jurisdiction over the sample collection process and these procedures are not covered by our documentation. Additionally, sample collection techniques are provided to each client requesting a sampling kit. These instructions now include photos of the proper sampling technique. The collection procedures are also available on the Radiochemistry Web site http://www.slh.wisc.edu/radiochem/kits.html.

The process of receiving samples and entering the samples into the computer system is described in the following SOPs: ESS RAD GENOP 020 SOP - Sample Checkin.doc, and ESS RAD GENOP 021 SOP - Radon Air VB App.doc

Record management and sample disposal are described in the following SOPs: ESS RAD GENOP 014 SOP - Rad Records.doc, and ESS RAD GENOP 011 SOP - Sample Disposal.doc

All SOPs are available on-line to all Radiochemistry staff. A hardcopy version of the methods manual and instrument manual is kept in the laboratory for convenience.

10.4.2. Sample Submission

Samples arrive at the laboratory in several ways. They may be mailed in, brought to the laboratory by client field representatives, or brought to the front desk of the laboratory by the general public. Currently samples pass through the sample receiving area and are passed on to the Department sample custodian to be assessed for acceptability and assigned a unique laboratory number. Samples may be rejected if they show definitive signs of contamination. In general, however, no samples are rejected without first contacting the client and advising them of the situation. See the Department SOP for sample check-in (ESS RAD GENOP 020).

Each sample received is accompanied by an informational sheet (usually a WDNR field sheet or WSLH labslip) which is date stamped, and labeled with a unique laboratory number assigned to that sample. Samples requiring preservation are preserved at this time. For analyses of multi-phasic samples the client should be contacted for direction on how to proceed. Once assessed, and labeled, the samples are taken to their proper storage areas in the appropriate section of the laboratory to await analysis. All pertinent information on the paperwork is entered into the Radiochemistry Information Management System (RIMS)(See Chapter 8).

All samples, as well as being entered in the RIMS database, are entered into the ESS Sample Tracking database using the following information: unique sample ID number, temperature, date received, initials of the receipt analyst, the number of containers received, the type of containers received, field acidification or preservation (if any), lab acidification or preservation (if any), the location for storing the sample and any particular pertinent comments about the sample. A unique laboratory number and associated barcode is assigned to each sample container (in conformance with current NELAC guidelines).

If a sample is used up, broken, disposed or its status is otherwise altered, this is noted in the database, again including the date and the analyst's initials.

10.4.3. Sample Handling

Once a sample has been received it undergoes the process described in Chapter 8 — Documentation Procedures. The sample is procured from storage by the analyst and undergoes the testing procedure. Each testing procedure is described in detail in the individual method SOP. If it is necessary to take a sub sample for analysis, the sample is homogenized and an aliquot is removed employing appropriate laboratory techniques. Some test procedures will consume the entire sample or compromise the remaining sample by removing an aliquot. However, for those that do not, the sample will be returned to the proper storage area as soon as possible.

10.4.4. Sample Storage and Disposal

Samples are stored in the sample storage room. Perishable samples, such as milk, are stored in the cooler. The radon air samples are kept in the lab since they are analyzed the same day they arrive (the analysis is done in the sample container). All samples or the original empty container are kept at least four weeks after the sample was reported.

Samples are disposed of in a manner which is consistent with the nature of the sample and the applicable rules and regulations. Waste disposal guidelines are described in the University of Wisconsin Chemical Safety and Disposal Guide.

10.4.5. Chain-of-Custody Procedures

The Radiochemistry department does not process enforcement samples at this time. The WDNR submission form or the SLH lab slip functions as the COC. Occasionally other laboratories will submit samples using their own COC. In

this case, the staff receiving the sample will sign and date the COC in the appropriate places and return a copy of the COC to the other laboratory.

10.5. Water Microbiology

10.5.1. Record Keeping

The Water Microbiology Department does a sterility check, fluorescence check and volume check on one bottle from each box of bottles received from the vendor or prepared internally. The results are recorded in the QC logbook in the Water Microbiology Department. See ESS MIC QA 212 for more details. The bottles are then sent to the client for sample submission.

10.5.2. Sample Submission

Drinking water, pool, surface, and water samples for *Cryptosporidium* and *Giardia* are collected by public water supply operators, private citizens, private business personnel, local government personnel and state agency personnel and returned to the Wisconsin State Laboratory of Hygiene for analysis. The laboratory does sampling only for research projects. All sampling instructions are per EPA manual or WSLH instructions.

In addition, research samples may be collected by laboratory personnel, public water supply operators, private citizens, private business personnel, local government personnel and state agency personnel.

Samples arrive at the laboratory in several ways. They may be mailed, delivered by private carriers, delivered by a client's field representative or hand delivered by the general public to the customer service area. Samples are then passed through the sample receiving area where they are assigned a unique laboratory number.

If a sample arrives showing definitive signs of contamination or in a condition that nullifies any meaningful analysis (e.g., frozen), the sample will not be analyzed. If a sample is damaged (i.e., leaking) it may be analyzed. If a positive result occurs the client will be contacted and another sample will be submitted. In some cases a compromised sample may be analyzed after consultation with the appropriate regulator. Any data generated from that sample will be appropriately qualified. See ESS MIC GENOP 102 and 104.

All samples, as well as being entered in the LIMS database, are entered into the ESS Sample Tracking database using the following information: unique sample ID number, temperature, date received, initials of the receipt analyst, the number of containers received, the type of containers received, field acidification or preservation (if any), lab acidification or preservation (if any), the location for storing the sample and any particular pertinent comments about the sample. A unique laboratory number and associated barcode is assigned to each sample container (in conformance with current NELAC guidelines).

When a sample is removed from storage (for analysis or any other reason) for any length of time, its unique sample ID label is scanned into the ESS Sample

Tracking database and recorded as being removed, noting the date and the initials of the analyst. This also occurs when the sample container is returned. If it is used up, broken, disposed or its status is otherwise altered, this is noted in the database, again including the date and the analyst's initials.

10.5.3. Sample Handling

Once a sample has been received it undergoes the process described in Chapter 8 - Documentation Procedures. The sample is analyzed immediately. If it is necessary to take a sub sample for analysis, the sample is homogenized and an aliquot is removed employing appropriate laboratory techniques. Please see the individual method SOP for a detailed description of the procedure.

10.5.4. Preservation, Storage and Disposal

Samples are shipped in Styrofoam boxes to hold temperature constant. Holding/transit time between sampling and analysis should not exceed 30 hours for public drinking water samples. For samples received after the 30 hour holding time and up to 48 hours, the laboratory is to indicate that the data may be invalid. Samples arriving after 48 hours shall not examined. Date and time of sample analysis is recorded. Samples for private wells are tested up to 2 days.

Cryptosporidium and *Giardia* samples are shipped in Styrofoam boxes with a freezer pak. Samples received after 96 hours are not tested. Date and time of sample collection and analysis is recorded.

Surface waters are shipped in Coleman coolers with ice to keep transit temperatures below 10°C. Temperature of samples is taken when ice is completely melted. Holding/transit time between sampling and analysis should not exceed 6 hours for enforcement and 24 hours for monitoring. Samples arriving after 24 hours shall not be examined unless requested by the collector and marked as being received after holding time. (Most of our clients request sampling after the 6-hour and 24 hour holding time.) Dates of sample collection and analysis are recorded.

Samples are disposed as outlined in the method SOP. Cultures and sewage samples are placed in the red MERI (Madison Energy Recovery Institute) barrels to be treated and landfilled. Alternatively sewage samples may be autoclaved.

10.5.5. Chain of Custody Procedures

Chain-of-Custody (chain-of-possession) is a means of demonstrating the traceability of a sample from the time of collection through its introduction as evidence in a law enforcement case. This is accomplished by maintaining an accurate written record that may be used to physically trace a sample from the moment of collection through transportation, storage and analysis.

Complete chain-of-custody is only possible through the cooperative effort of both laboratory and field personnel. The procedures used by the laboratory are

described in the following section. The responsibility of sample collector and chain of custody procedures used by field personnel are described in the Wisconsin Department of Natural Resources Quality Assurance Manual.

10.5.5.1. Transfer of Law Enforcement Samples to the Laboratory

Enforcement samples may be transported to the laboratory by two means:

The sample collector may keep the samples in his/her physical possession and deliver them directly to the laboratory.

The collector may seal the samples in a field pack (shipping container) and ship the samples to the laboratory by U.S. mail or other transport service.

10.5.5.2. Physical Transfer

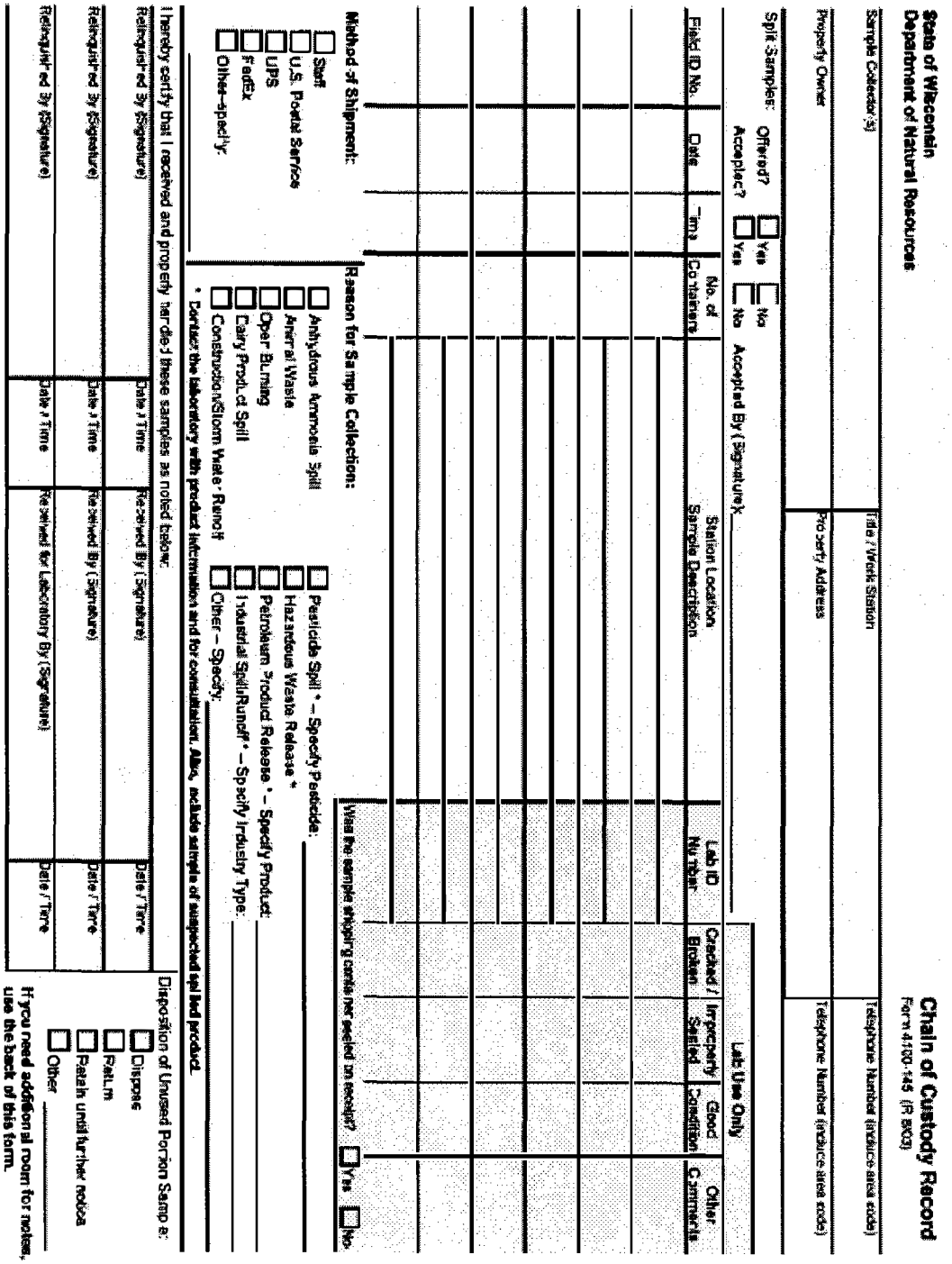
Department personnel will receive law enforcement samples, identify each sample container with a unique laboratory number and the date received, and complete a law enforcement receipt. The receipt (Figure 10.1) will include the following information: 1) field identification number, 2) laboratory number; 3) description of the sample; 4) the name, title, work station and telephone number of the person delivering the samples; 4) the time (hour and minute) and date the samples are received in the laboratory; 6) the disposition of the unused portion of the samples remaining after the analyses; and 7) the signature of the laboratory employee receiving the samples. The original receipt will be given to the person that delivered the samples and a copy will be retained by the laboratory.

10.5.5.3. U.S. Mail Transfer and Transport Carriers

The collector will place the samples in a Laboratory of Hygiene Styrofoam field pack (shipping container) and seal with reinforced nylon tape. The ends of the tape are kept straight so they overlap slightly. Using a waterproof pen, the collector will write "Enforcement Case", his/her name, date, time and sample identification on the tape where the ends overlap. When the field pack is received at the Laboratory of Hygiene, the sample receiving personnel will examine the seal to make sure that it has not been tampered with. He/she will then open the field pack, remove the samples and laboratory sheets and write his/her initials, the time and date on the bottom of the laboratory sheets and on the accompanying Chain of Custody record sheet. The samples are then assigned a unique laboratory number and processed according to Chapter 8.

10.5.5.4. Because of holding times for microbiological samples, they are processed immediately. After the sample has been processed, the samples are discarded.

Figure 10.1 — WDNR Chain of Custody Record



Chain of Custody Record 08/20/03

Figure 10.2 — Enforcement Disposition Form

COLLECTOR NAME - WDNR - NER
ADDRESS
CITY, STATE ZIP

ENFORCEMENT

Sample(s) will be disposed of ninety
days from the date the sample is reported,
unless this form is completed
and returned to:

Organic Chemistry Department
Wis. State Lab. of Hygiene
465 Henry Mall
Madison, WI 53706

Collector:_____.

Region:_____.

Phone Number:_____.

Sample Number(s):_____.

Report date:__/__/__.

___Retain sample(s) for ___ days.
___Retain sample(s) until further notice.

11. Instrumentation and Equipment

11.1. General

The Environmental Sciences Section relies heavily on instrumentation. It is imperative that all instruments are calibrated, operated, and maintained in a proper manner in order to obtain reliable data. Even the best, state-of-the-art instruments may produce unreliable data if they are not adequately calibrated, maintained and operated by experienced professionals.

All equipment and instrumentation is operated and maintained according to the manufacturer's recommendations. Although there are summaries listed below, if detailed performance, troubleshooting, and maintenance criteria are desired, then the appropriate instrument operational manual or instrument operating procedure (IOP) should be consulted. If an instrument fails to operate within manufacturer defined limits (or specifications), or self-diagnostic indicators become active, operation is immediately stopped, the problem is identified, and steps are taken to resolve the situation. It is the responsibility of the analyst to notify the department supervisor if non-routine maintenance is required.

Preventive maintenance is considered to be the general maintenance performed at the frequency recommended by the manufacturer to circumvent instrument failure. Most instruments and equipment are on some form of preventive maintenance schedule. Some instruments (i.e., atomic absorption spectrometer, ICP, UV/Vis spectrophotometer) are covered by maintenance contracts directly with the instrument manufacturer. In many cases maintenance schedules are posted near the instrument for easy access. Preventive maintenance is always recorded in the instrument logbook.

11.1.1. Facilities

The Wisconsin State Laboratory of Hygiene (WSLH) is currently housed in two separate facilities, 465 Henry Mall on the UW Madison Campus, and 2601 Agriculture Drive on the southeast side of the city of Madison. The Departments covered under this manual are only part of the 22 laboratory departments of the Environmental Health Division (EHD). The EHD is housed entirely in the 70,000 sq. ft Agriculture Drive facility, built in 1999.

The WSLH is a part of the University of Wisconsin System. As part of the University, the WSLH benefits from the services and expertise offered by the University such as the Biological and Chemical Safety Department, Radiation Safety Department, collaborations with faculty, and access to the library system. Although the Environmental Health Division laboratory is not physically located on the University of Wisconsin campus, the staff has full University privileges.

The activities of the various laboratories are supported by a fully automated glassware washing room, a shipping and mailing department, a stores department, a human resources department, an information systems department,

and a fiscal department. Some of these support departments are located in the main Henry Mall facility. However, once a day, a shuttle run is made between the two buildings to make drop-offs and pick-ups.

11.1.1.1. Air Handling

The Agriculture Drive facility is equipped with an air handling system supplied and maintained by an independent contractor. The system consists of two intake fans and two exhaust fans, with a third exhaust fan that serves as a back up. The back-up fan assures that in the event of a failure there is always negative pressure in all fume hoods and snorkels.

Except for the trace metal clean lab (rm. 120), there is a 100% exchange of air, (i.e., what comes in, goes out; there is no recirculation) and the volume of air in the building is changed approximately once per minute. In addition, the building is designed so that the laboratories will generally be under a negative pressure. The air will flow from the office/cubicle space, into the lab, and then be exhausted.

11.1.1.2. Laboratory Reagent Grade Water

Reverse Osmosis (RO) Water

RO water is plumbed to all the laboratories and is maintained by a service contract with a water treatment vendor. The RO system consists of a prefilter of graded density non-woven polypropylene for the carbon bed, a filter cartridge of non-woven polypropylene for the 3 - RO/DI 5µm resin traps (replaced every 6 months), a UV Sterilizer in which the Aqua Fine replacement lamp and the Aqua Fine Quartz Sleeve will be replaced annually, a submicron Absolute Rated 0.2 µm bacteria eliminating filter (replaced every 6 months), a recalculating tank vent filter (replaced annually), and cation, anion and mixed bed tanks that will be replaced as needed.

The water is Type II and is used for the glasswashing activities, filling water baths, and as a precursor for other "polished" water throughout the building.

Pure Lab Plus UV/UF ASTM Type I Polisher

These polishers are located throughout the laboratory and provide Type I water used in the preparation of media and reagents. There is a service contract with a water treatment vendor to maintain the polishers. Every six months they will exchange the carbon filter, the mixed bed cartridges and the organic scavenging Type II ultra pure anion resin. The UV filter will be changed based on the number of hours of use.

11.1.1.3. Ovens, Incubator, Cold Rooms, Refrigerators, Freezers, and Muffle Furnaces

Each Department maintains a logbook for its temperature sensitive equipment. The temperatures of the walk-in refrigerators are monitored continuously, and an alarm will be activated if limits are exceeded. Temperatures of all equipment are checked daily (see ESS INO GENOP 200), except the muffle furnace which is on a monthly schedule (when it is being used). The temperature and required adjustments are recorded in the logbook. The temperature of each item must be maintained within the tolerances specified (See Figure 11.4). If the temperature exceeds the allowable tolerance, the supervisor is notified and the problem corrected before the equipment is used

11.1.2. Computers

11.1.2.1. LIMS

The Laboratory Information Management System (LIMS) of the Environmental Sciences Section (ESS) is a mini-computer based data service used to acquire, organize, and report analytical data. The original system was comprised of a Concurrent Computer Corporation (CCC) 3200 series mini-computer, CCC's proprietary operating system (OS), utilities, and database management software (DMS), and Perkin Elmer's DMS application suite known as LIMS 2000.

Since that time, the entire application suite has been replaced with in-house developed software to better meet the changing needs of the laboratory. The hardware has gone through two major revisions and the supporting OS has been replaced with a UNIX based system. For more detailed information see the LIMS online manual.

The system is secured by the use of multiple accounts, user IDs and several levels of password protection. To ensure proper procedure, all electronic data transfers to clients are performed by the LIMS administrator or their fully trained designee.

11.1.2.2. Windows NT Network

The organizational structure of the WSLH is supported by a Windows NT network, which is spread across two building locations. NT servers in the two main locations share the workload. The Environmental Health Division (EHD) site (Agriculture Dr.) has an NT file server as well as its own mail, archival, and Oracle database servers. The EHD site is connected to our campus site by a T1 line with an ISDN line as a backup connection. The file server stores data files, user profiles, and software. The file server has a capacity of over 26 gigabytes of disk space in a hardware RAID array and is backed up daily.

The Agriculture Drive facility was built with a fiber optic cable backbone and wired throughout the building with Category-5 network cables. The majority of the desktop PCs (whether office or instrument) are Windows

NT workstations, however a slow migration to Windows 2000 and Windows XP is occurring due to instrument and security issues. There are also still a few PCs that use a mix of different versions of Windows, DOS and OS/2 operating systems. These are used for specific instrument needs, and may or may not be connected to the network.

11.1.3. Purchasing

11.1.3.1. Supplies and Standards

Within the bounds of the State of Wisconsin procurement system, laboratory staff will strive to purchase the highest quality products possible, from known vendors. However, if a change of supplier is called for, any new product will be thoroughly evaluated for appropriateness and quality before being put to use.

Chemicals and other laboratory supplies are ordered from the UW Materials Distribution System or from individual vendors. Supplies are ordered from the state primary contract vendor where possible. If, however, a particular item is not available from that vendor it may be ordered from any vendor.

11.1.3.2. Capital Equipment

Capital equipment purchases (i.e., > $5000) are generally sent out for bid. Laboratory staff work closely with the purchasing department to assure that all necessary specifications are included in the bid.

All items are purchased through the central purchasing office at 465 Henry Mall.

11.2. Biomonitoring

11.2.1. Laboratory Facilities

The Biomonitoring laboratory is composed of three separate laboratory spaces. All three rooms are separated from one another by doors with each room having their own air handling systems. Room 204 (825 sq. ft) is the WET testing room, and is comprised of bench space used for test set up and renewal, two walk-in environmental chambers, and a walk-in 4° cooler. Room 205 (785 sq. ft.) is comprised of the fathead minnow aquaculture and bench space used for culturing daphnia. Room 205A (105 sq. ft.) is used for special projects. The office area for Biomonitoring is located across the hallway from the laboratory space.

Separate culturing and toxicity testing areas are important in order to avoid possible loss of cultures due to cross contamination. Ventilation systems are designed and operated to prevent recirculation or leakage of air from chemical analysis laboratories or sample storage and preparation areas into organism

culturing or toxicity testing areas, and from toxicity test laboratories and sample preparation areas into culture rooms.

11.2.2. Laboratory Instrumentation and Equipment

11.2.2.1. Balances

Mettler PJ4000 Balance

Preventative maintenance, cleaning and calibration performed yearly. Weekly, the balance is checked with at least two type S standard weights (1000g and 0.2g). Observed mass of both standards must not deviate more than 0.1 grams from the standard. See ESS BIO GENOP 50.5.

Mettler AE240 Balance

Preventative maintenance, cleaning and calibration performed annually. Prior to each use, the balance is checked with at least two type S standard weights bracketing the mass range of samples to be weighed. Observed mass of both standards must not deviate more than 0.00010 grams from the standard. See ESS BIO GENOP 50.5.

11.2.2.2. Ovens, Environmental Chambers, Incubators and Freezers

Blue M-drying oven

Temperatures are monitored on every day of use. Temperatures must be 105-110°C, to be in control. If temperature varies from this, the dial is adjusted to reach proper temperature.

Walk-in Environmental Chambers (Norlake Scientific)

Chambers (2) need to be $\pm$ 1°C of their set temperature (temp. will depend on the test being run in the chamber at a given time). Temperatures are monitored twice daily (once on weekends and holidays) in at least two locations within each incubator and recorded in bound logbooks. Minimum and maximum temperatures are recorded daily. Temperature is monitored on a continuous basis with an external flywheel chart.

Percival I30BL Incubator

Temperature is monitored twice daily (once on weekends and holidays) in at least one position within the incubator and recorded on log sheets. The temperature must remain within $\pm$ 1°C of the desired temperature to be in control. Temperatures outside of this range will be adjusted using the external temperature control dials on the incubator.

Percival I37L Incubator

Temperature is monitored twice daily (once on weekends and holidays) in at least one position within the incubator and recorded on

log sheets. The temperature must remain within ± 1°C of the desired temperature to be in control. Temperatures outside of this range will be adjusted using the external temperature control dials on the incubator.

Hotpack Refrigerated Incubator

Temperature is monitored twice daily (once on weekends and holidays) in at least one position within the incubator and recorded on log sheets. The temperature must remain within ± 1°C of the desired temperature to be in control. Temperatures outside of this range will be adjusted using the external temperature control dials on the incubator.

Westinghouse Refrigerator

Temperature of refrigerator is monitored daily. Temperatures are recorded on log sheets. Temperature must be between 1 and 4°C. If not, controls are adjusted until proper temperature is obtained.

11.2.2.3. Laboratory Reagent Grade Water

See General Section

Milli-Q polisher

This polisher provides Type I water used in the preparation of reagents and synthetic laboratory water. There is a service contract with a water treatment vendor to maintain the department. Maintenance will be performed every six months. The polisher is sanitized weekly as described in the manual. See ESS BIO GENOP 30

11.2.2.4. Meters and probes

Orion 520 pH meter

Calibrations of the instrument are made using fresh buffers on a daily basis. Slope of the calibration must be within 98-102%. If slope is outside these limits, corrective measures are taken. The pH standard is replaced with fresh solution, the probe is soaked in acid, rinsed, and new fill solution is added to the probe. The calibration is rechecked. Daily, the calibration is checked with a second source pH 7 standard and a blank is checked using Milli-Q water. Weekly, the probe is soaked in 1N HCl and new probe solution is added. See ESS BIO GENOP 102

11.2.2.5. YSI 35 conductivity meter

On a daily basis the conductivity of a 0.01M KCl solution is determined. Conductivity must be 1413 ± 141 microhoms/cm to be in control. Values are recorded in the bound meter calibration logbook. The meter is standardized daily using the 0.01 M KCl standard. The value of the standard and the cell constant are determined. The cell constant must be

between 0.85 - 1.15. If the cell constant is not in control the probe is cleaned with chlorine bleach, re-platinized or replaced. A second source standard is checked daily, along with a Milli-Q blank. See ESS BIO GENOP 103

Dissolved oxygen meter

The meter is calibrated daily. The red line and zero line are checked, and if not lined up accurately, the respective knobs are adjusted to align the lines. The temperature is checked and the DO of the meter is determined by the saturation table for that temperature. Immerse the probe in Synthetic Hard Water and adjust the calibrate dial to match the correct calibration value. If the meter will not calibrate correctly, clean electrode, replace the membrane cap on the probe, and recalibrate. Daily, dechlorinated tap water is checked as a second source and along with a Milli-Q blank. See ESS BIO GENOP 104

Ammonia probe

The probe is calibrated prior to each use, using standards that bracket the expected sample range. The slope of the meter must fall between –54 and –60 mV/decade. If the probe falls outside of this range, the electrode operation must be checked. The probe is checked after calibration with a standard of 4mV. If this standard is not within $\pm$ 10%, then the probe must be re-calibrated. All measurements are recorded in the ammonia chemistry file. See ESS BIO GENOP 105

Chlorine probe

The probe is calibrated before every use. Calibration is done using bleach as a standard to achieve 100ppm Cl_2 (total residual chlorine). Reading should be about between 26-33mV. If the reading is below 26, then refer to the troubleshooting section of the probe manual. The meter must be standardized using a 1ppm standardizing solution. The calibration control is adjusted to read 000.0. Both the calibration reading and the standardization reading are plotted on scale 4-cycle semi-logarithmic graph paper. See ESS BIO GENOP 106

11.2.2.6. Digital thermometers and probes

Digital thermometers

The calibration data for digital thermometers are NIST traceable and come with a calibration certificate that states "once measured and calibrated your thermometer should maintain its accuracy."

Thermometer probes

The calibration data for thermometer probes are NIST traceable and come with a calibration certificate that states "once measured and calibrated your thermometer should maintain its accuracy."

11.2.2.7. Hirayama HA-240M/300M autoclave

Autoclave temperature is monitored with each use by autoclave tape stripes turning from white to black.

11.2.3. Supplies

11.2.3.1. Pipettes

A calibration check is done quarterly for all pipettes. Pipette calibration is checked by using the Artel PCS pipette calibration system (See ESS INO GENOP 200). Calibration measurements must fall within the range stated for each individual pipette. If pipettes fall out of this range for calibration they must be re-calibrated per the instruction manual for that pipette.

11.3. Inorganic Chemistry

11.3.1. Laboratory Facilities

The Inorganic Department of the Environmental Science Section is composed of three main areas: Metals (room 117--instrument, and room 118--prep), Wet Chemistry (room 119), and the Trace Metal Clean Lab (TMCL) (room 120).

The Metals area is shared with the Wisconsin Occupational Health Laboratory (WOHL) metals department. The area consists of an instrument room (1280 sq. ft.), a balance / chemical storage room (200 sq. ft.), and the prep room (600 sq. ft.) containing the hoods used for metal digestions and TCLP extractions. The balance room and prep room are separated from other areas by doors.

The Wet Chemistry area (2920 sq. ft.) contains the ovens, incubators, instrumentation, glassware, and hoods used for general chemistry (BOD, TSS, etc.) and nutrients analysis. This area is open to the hallway and also contains the Inorganic sample check-in area, wash room pick-up and delivery area, and a locked sample storage cold room (170 sq. ft.).

The TCML area (770 sq. ft.), which is used for ultra low metals analysis, contains a positive pressure entry vestibule, and a HEPA filtered instrument room with laminar flow hoods and a Milli Q water system.

All laboratory areas are separated from the cubicle office area (1125 sq. ft.) by a hallway.

11.3.2. Laboratory Instrumentation and Equipment

11.3.2.1. Balances (Instrument numbers 54, 80, 81, 82, 83)

All balances are on a preventive maintenance schedule. A Mettler service technician comes to the lab annually to clean, check, and adjust all the balances and to certify that they are in proper working order. The balances are calibrated at least once daily using an internal calibration feature. An accuracy verification using Class 1 weights is done daily. For details on these procedures, please see ESS INO GENOP 202, "Calibration, Maintenance, and Accuracy Verification Procedure for

Balances", and ESS INO GENOP 203, "Handling, Maintenance, and Calibration of Weights". Appendix 1 of ESS INO GENOP 202 has a separate section for each balance that lists acceptable mass ranges (based on certified masses) for the accuracy verification. Each balance has a logbook where calibration and accuracy verification data are recorded.

11.3.2.2. Color Wheel (Instrument number 58)

The color wheel is verified with potassium chloroplatinate standard solutions at least annually. Verification is recorded in the logbook. For details of these procedures see ESS INO METHOD 170.1.

11.3.2.3. Atomic Absorption Spectrophotometers: Perkin Elmer 5100Z Graphite Furnace (Instrument number 32), Perkin Elmer 4100ZL Graphite Furnace (Instrument number 33), Perkin Elmer Aanalyst 100 Flame (Instrument number 93)

These instruments are used for metals and elemental analyses. A logbook is maintained for each Spectrophotometer to document the date, operator, element, characteristic mass, lamp energy, hours of use, absorbance values of standard solutions, correlation coefficient, worklist, general maintenance performed and any problems encountered. Standard curves are verified with control samples, and check standards are analyzed at least every 10 samples. For more details of these procedures see ESS INO METHOD'S 400.3, 400.5, 560.1.

11.3.2.4. Autotitrator System: Radiometer (Instrument number 56)

The autotitrator is an automated instrument used to perform pH, conductivity and alkalinity analyses sequentially on a single sample. The instrument is calibrated daily according to the manufacturer's recommendations with standards for each constituent. Calibrations are verified with a quality control standard. All maintenance information is recorded in the instrument logbook. For more details of these procedures see ESS INO METHOD 115.1.

11.3.2.5. Continuous Flow Analyzers: Lachat Flow Injection Analyzers (Instrument numbers 11, 12, 13, 18)

All Continuous Flow Analyzers (CFA) are on a preventive maintenance schedule. A loose-leaf logbook is maintained for each CFA module. Items such as standard calibration control settings, baseline measurements, and general maintenance are recorded in the logbook. A logbook is also maintained for each Lachat CFA. CFAs are operated and maintained according to the manufacturer's recommendation. Detailed operating instructions are provided in the following SOP's: ESS INO METHOD'S 220.9 (Nitrate/Fluoride), 310.2 (Total Phosphorus), 370.3a (Sulfates), 141.0 (Chloride), 230.3 (TKN), 360.2 (Silica), 220.3 (Ammonia/Nitrate).

11.3.2.6. Dissolved Oxygen Analyzers (Instrument numbers 50, 51, 52, 53)

All probes and meters are identified by a unique number. The daily use, maintenance requirements and comments on the general performance of each item are recorded in the logbook.

Each morning the dissolved oxygen (DO) analyzer is calibrated with at least two Winkler titrations. The calibration is verified several times daily using a water sample with a known DO concentration. If the analyzer fails to maintain proper calibration, no further measurements will be made until the problem is identified and resolved. For details of these procedures see ESS INO METHOD 260.1.

11.3.2.7. pH Meter (Instrument number 61)

An instrument logbook is maintained for the pH meter. The meter is calibrated each day using two standard buffer solutions, either pH 7 and pH 4, or pH 7 and pH 10. The linearity of the instrument is checked monthly. The calibration and slope are recorded in the logbook along with any other pertinent information. Detailed instructions for checking the pH meter performance may be found in the instrument logbook. Also see ESS INO METHOD 300.0.

11.3.2.8. Ion Selective Electrode (Instrument number 19)

This instrument is used for ammonia analyses. An instrument logbook is maintained for the ion selective electrode. Maintenance, performance problems, analyst initials, and other pertinent information are documented in the logbook. Calibration is done for each run using a blank and four standards. A quality control standard is analyzed for each day of analysis. An instrument performance check and a calibration check blank are analyzed after every 10 readings. For more details see ESS INO METHOD 221.0.

11.3.2.9. UV-Visible Spectrophotometer: Beckman DU-650 (Instrument number 55)

This spectrophotometer is routinely used for dissolved phosphorus, COD, cyanide, nitrite, and hexavalent chromium analyses performed in the department. This instrument has a number of on-board performance test functions that include wavelength accuracy, repeatability, resolution, baseline flatness, noise and stability. All of these performance tests (except stability) are run each time that the instrument is used. Stability, which takes several hours to run, is checked yearly. If any of the checks exceed their established tolerances (as documented on the instrument printout), the department supervisor must be notified and the instrument repaired before any further analyses are performed.

The instrument is on a service contract and is checked yearly by a Beckman Service Engineer. The service engineer performs a preventative

maintenance check and runs tests to evaluate all of the above functions (in addition to photometric accuracy using NIST filters). The results of the performance checks are printed and placed in a binder logbook. A maintenance logbook and instrument usage log are also maintained. For details on the operation of the spectrophotometer see ESS INO IOP 400.

11.3.2.10. Turbidimeter: Nephelometer - HACH 2100N (Instrument number 57)

An instrument logbook is maintained for the nephelometer. Quarterly, the nephelometer linearity is verified using formazin standards. Sealed secondary standards are used for daily calibration for routine surface water analyses. These standards are verified quarterly using formazin standards. When drinking water compliance measurements are made, the nephelometer is calibrated using HACH primary standards. The nephelometer is maintained according to the manufacturer's instructions. The results of the calibration verifications and all maintenance are recorded in the logbook. For more details on these procedures see ESS INO METHOD 380.3.

11.3.2.11. Inductively Coupled Plasma Emission Spectrometer: Thermal Jarrell Ash 61E (Instrument number 30)

The ICP is used for metals and elemental analyses. A logbook is maintained for the ICP to document the date, operator, sample matrix, the Cu/Mn emission ratio, Cu profile offset, worklists, general maintenance performed and any problems encountered. Standard curves are verified with control sample checks including an interference check. A continuing calibration verification standard and a blank are analyzed after every 10 samples. For details on these procedures see ESS INO METHOD 400.2.

11.3.2.12. Ion Chromatographs: Dionex Model 600 (Instrument number WOHL 600), Dionex DX-100 (Instrument number WOHL 201)

These instruments are operated and maintained by the Wis. Occupational Health Department. They are used for hexavalent chromium and anion analyses. A calibration curve is analyzed and verified each day before proceeding with analysis. A midrange check standard is also analyzed at the beginning of each analytical run, every 10 samples thereafter and at the end of the run. Daily monitoring and maintenance of the separator column, etc. are documented in a bound logbook. If the standard curve proves to be unacceptable, or the precision and/or accuracy control limits are exceeded, the analyses are considered invalid. The problem must be identified and corrected before analyses are resumed. For more detailed procedures see ESS INO METHOD 470.5 (Hex Cr).

11.3.2.13. Micropipets

A logbook is maintained for all micropipets. Each pipette is identified by either a serial number or a unique ID number. The accuracy of

micropipets is verified quarterly using the procedure that is outlined in ESS INO GENOP 200. If a pipette does not perform within the accuracy tolerance (listed in the SOP), the pipette is cleaned, lubricated and checked again before it may be used. The adjustable column pipettes are the only exception to this; however, they should never be lubricated. If they do not meet accuracy tolerances they should be serviced by replacing the defective parts. All verification information and required maintenance are recorded in the logbook.

11.3.2.14. Inductively Coupled Plasma/Mass Spectrometers: FISONS VG Plasma Quad (Instrument number 34), VG Plasma Quad ExCell (Instrument number 94)

These instruments are used for metals, elemental, and possibly some isotopic analyses. Two logbooks are maintained for the ICP/MS's. One logbook documents the date, operator, time of use, vacuum levels, gas flows, sample uptake, lens' settings, ICP power, nebulizer type, sensitivity performance and any problems encountered with the analysis. The other logbook documents instrument problems, maintenance and service/repairs performed.

A mass calibration and a response calibration are performed each day the instrument is operated. A detector cross-calibration is performed at least once a week and whenever the multiplier settings and/or nebulizer are changed. An isotope peak resolution for a low mass element and a high mass element is performed every day of operation. Standard curves are verified with a second source control sample and continuing calibration checks and blanks are analyzed at least every 10 samples. For more details see ESS INO METHOD 400.4.

11.3.2.15. Flow Injection Mercury System: Perkin-Elmer FIAS 100 (Instrument number 31)

A logbook is maintained for the FIMS to document the date, operator, sample matrix, absorbance values of standard solutions, correlation coefficient, worklists, general maintenance performed, and any problems encountered. Standard curves are verified with control samples and check standards are analyzed at least every 10 samples. If these checks are not within 10% of their known values, the previous 10 samples are rerun after the problem is corrected. For more details on these procedures see ESS INO METHOD'S 540.2 (solids), 540.3 (waters), and 540.4 (tissues).

11.3.2.16. Atomic Fluorescence Systems: Brooks-Rand Model III Detector (Instrument number 35), Leeman Labs Hydra AF (Instrument number 95)

These instruments are used for low-level mercury analyses. A logbook is maintained for the AFS system to document the date, operator, sample matrix, sensitivity setting, calibration slope, analysis trap, sample traps, LIMS sample numbers, general maintenance performed and any problems

encountered. Standard curves are verified with second source control samples and continuing calibration check standards and blanks are analyzed at least every 10 samples. If the control or check standards are not within 10% of their known values or the blanks exceed the LOD, the previous 10 samples are rerun after the problem is corrected. For more details on these procedures see ESS INO METHOD'S 541.1 and 541.2.

11.3.2.17. Luminescence Spectrometer (fluorimeter) (Instrument number 96)

This instrument is used for chlorophyll analyses. A logbook for instrument operating conditions and maintenance is maintained. The calibration curve is constructed using a blank and six standards. The calibration is verified by analyzing a quality control standard. A continuing calibration blank and a continuing calibration verification standard are run every 10 samples and at the end of the run. For more details see ESS INO METHOD 151.1.

11.3.3. Laboratory Supplies

11.3.3.1. Sample Bottles

When the bottles arrive, the QA Coordinator randomly selects bottle(s) to check for contamination by the parameters of interest. Those bottles are selected on a per lot, per delivery (up to ten cases) basis. If the bottles do not meet required criteria, they are not used for sample collection until corrective action is taken. For further information refer to ESS INO QA 101. Bottle types are listed below:

Plastic Quart

Glass Quart

Plastic 1 liter

Plastic 500 mL

Plastic 250 mL

Plastic 60 mL

Plastic 125 mL

Table 11.1 — Analytical[1] Instrument Summary for Inorganic Chemistry

Instrument	Serial #	Type	Analysis	Instr #	Location	Dt in use
LACHAT 8000	A83000-1108	flow inj.	Fluoride/NO3	11	119	11/3/97
LACHAT 8000	A83000-303	flow inj.	Tot. Phos / Sulfate / Chloride	12	119	12/15/95
LACHAT 8000	A83000-1106	flow inj.	TKN/ silica / micro silica	13	119	12/3/97
LACHAT 8000	A83000-1433	flow inj.	Ammonia / NO3	18	119	
Orion 720A	059726	electrode	NH3	19	119	9/17/01
Thermo Jarrel Ash 61E	69290	ICP	metals	30	117	8/26/92
Perkin Elmer FIAS-100	1091	FIMS	Hg only	31	117	6/15/95
Perkin Elmer 5100Z	6743	AA FURN	metals	32	117	5/09/01
Perkin Elmer 4100ZL	6544	AA FURN	metals	33	117	5/10/94
Perkin Elmer Aanalyst 100	40S0040101	AA Flame	Metals (potassium)	93	117	10/10/00
Fisons	PQS-957	ICP/MS	low level metals	34	120	7/1/93
VG Elemental	ExCell EX141	ICP/MS	low level metals	94	120	12/01/00
Brooks Rand Model III	N/A	Atomic fluorescence	low level Hg	35	120	7/27/95
Leeman Labs Hydra AF	AFG-1021	Atomic fluorescence	Low level Hg	95	120	04/01/02
YSI Model 59	90F016027	DO Meter	BOD	50	119	N/A
YSI 5905	LNN6L0268	DO probe	BOD	51	119	3/14/97
YSI 5905	94C79999	DO probe	BOD	52	119	12/2/96
YSI 5905	LN96M0332	DO probe	BOD	53	119	5/15/95
Orion SA520	QV14A	pH meter	pH	61	119	1/4/93
Mettler AT200	N08554	Balance	solids	54	119	12/14/92
Beckman DU650	4318104	Spectrophoto meter	Cr+6, diss P, COD, Nitrites, CN	55	119	1/1/95
Radiometer ABU91 Autoburette TIM900 Titration Manager CDM210 Conductivity Meter	102R39N05 649R024N009 653R039N027	Autotitrator	alkalinity / pH / conductance	56	119	3/01/99
Hach 2100N	980300004381	Turbidimeter	turbidity	57	119	4/2/98
Perkin Elmer LS55	59401	Luminescence spectrometer (fluorimeter)	chlorophyll	96	119	04/01/02

Instrument	Serial #	Type	Analysis	Instr #	Location	Dt in use
Hellige	N/A	Color meter	color	58	119	N/A
Dionex 600	02020438	Ion Chromatograph	Hex Cr	WOHL 600	116	05/01/02
Dionex DX-100	940308	Ion Chromatograph	anions	WOHL 201	116	11/10/94

[1]For a complete list that also includes support equipment, see \\slheagle\grp\EHD\ESS(4900)\ESS Inorg(4910)\SOP\Drafts\Instrument number listing.xls

11.4. Organic Chemistry

11.4.1. Laboratory Facilities

The Organic Chemistry laboratory comprises four separate laboratory spaces. Due to the low level nature of the analyses, the GC/MS volatiles lab (750 sq. ft.), the air analysis lab (990 sq. ft.) and the PCB and pesticide clean room (660 sq. ft.) are separated from the rest of the facility by doors. The general organic chemistry lab (1650 sq. ft.) contains space for pesticide, herbicide, PCB, TOC and other analysis and is open to the hallway. Each laboratory is equipped with ample bench top space and multiple fume hoods. In addition, snorkel hoods are used to capture exhaust gases from the GCs and MS vacuum pumps.

The laboratory space is complimented by numerous storage rooms, a fish grinding room, walk-in coolers, cylinder closets and a weighing room. The analyst desk area is separated from the lab proper by a hallway.

11.4.2. Laboratory Instrumentation and Equipment

11.4.2.1. Entech Summa Canister Cleaning System

The Entech cleaning system is used to clean both six, and one and one half liter, Summa canisters used for ambient air analysis. When in use the cleaning system is checked routinely for leaks (at least daily). The Alcatel Molecular Sieve vacuum pumps are greased annually or when needed and the butterfly valves on the roughing pump are replaced as necessary. All maintenance is recorded in the appropriate logbook.

11.4.2.2. Entech Cryogenic Concentrator

The laboratory utilizes an Entech cryogenic concentrator in the analysis of ambient air samples for volatile organic compounds following modified versions of EPA TO-14A. The concentrators use liquid nitrogen to trap compounds down to C2 (e.g., acetylene). Care must be taken that proper temperatures are attained, and that moisture control systems are functioning properly. Consult the instrument handbook for details.

11.4.2.3. Nutech Cryogenic Concentrator

The laboratory utilizes a Nutech cryogenic concentrator in the analysis of ambient air samples for volatile organic compounds (PAMS analysis). The concentrators use liquid nitrogen to trap compounds down to C2 (i.e., ethylene, acetylene, and ethane). Care must be taken that proper temperatures are attained, and that moisture control systems are functioning properly. Consult the instrument handbook for details.

11.4.2.4. Gel-Permeation Chromatograph

The gel-permeation chromatograph uses size exclusion chromatography to separate lipid from analytes of interest. A logbook is maintained recording samples, dates, analyst, collect and dump times and recoveries

of the associated quality control sample. In addition all maintenance is recorded in the same logbook.

11.4.2.5. Total Organic Carbon Analyzer

A Tekmar/Dohrman Phoenix 8000 with a model 183 Boat Sampler is used to measure total and dissolved organic carbon in water and sediment samples. A maintenance/sample logbook is maintained for recording all routine and non-routine maintenance and tracking sample load. Pump tubing is changed routinely.

11.4.2.6. Purge and Trap Autosamplers and Concentrators

The laboratory utilizes Tekmar 3000 concentrators and Precept II autosamplers to perform volatile organic compound (VOC) analysis in a variety of matrices. In most cases system gas flows are set to method specifications and will only be checked if a problem develops. In addition the performance of the trap will be monitored by closely watching the performance of particular VOCs whose dwindling response can act as an early warning sign of trap degradation.

11.4.2.7. Gas Chromatographs (GC)

Gas chromatographs (GC) have improved to the point where there is no longer the need or possibility to accurately measure or adjust various components by external means. In fact instruments are now controlled by computer software which not only provides instrument control but also diagnostics. However several systems still need to be monitored in order to ensure good performance. For a summary list of GCs and other equipment see Table 11.2

Flow Control and System Gases

All gases used in the laboratory's GC systems are ultra high purity (UHP) or better. All pressure regulators are checked for leaks and appropriate pressures on a yearly basis. Defective regulators are immediately taken out of service and returned to the manufacturer for repair or disposal.

Gas flows are monitored with an electronic soap film bubble meter whenever needed (depending on the detector outlet arrangement). Many instruments now feature continuous electronic monitoring of flows, however manual checking is sometimes still required. Each flow system fitting that is changed or adjusted is checked before the instrument is returned to use. In addition, gas line dryers and filters are changed at one-year intervals and regenerated according to manufacturer's directions. (These may not be used on every instrument). Any maintenance is recorded in the instrument logbook.

Detectors

Gas chromatographic detectors used for analysis of organic compounds must be sensitive to minute amounts (nanogram and picogram) of the materials of interest. Therefore they must be maintained and operated for optimal performance.

Mass Selective Detectors and Mass Spectrometers — Each MS or MSD undergoes various tuning procedures before use. These include BFB and DFTTP tunes. Each tune must be within method specific parameters before analysis can begin. Copies of all tunes and tune checks are kept in a log near the instrument.

Electron Capture Detectors (ECD) — Each ECD has a beta particle source (63Ni) and all are registered with the University of Wisconsin Safety Department. The detectors are leak tested two times per year and the results are recorded by the Safety Department. In addition, any maintenance of ECD detectors must be performed by the manufacturer and detectors must be shipped and received by the UW Safety Department.

Flame ionization Detector (FID) — Modern FID instruments are factory or installer preset and require no further maintenance. Some older models (rarely) require flow adjustments or cleaning. Changes in sensitivity or fluctuations in response are normally associated with other causes (columns, oven, etc.).

Hall Electrolytic Conductivity Detector (Hall or HECD) — The HECD is a specific detector for gas chromatography. It is comprised of five basic detector components. These are the reactor, the conductivity cell, the signal processing module, the pump/reactor control module, and the solvent module. The HECD is capable of very high sensitivity. Peak tailing and loss of response at or near the detection/report limit may indicate the need for detector adjustment or repair as described in the instrument handbook.

Photoionization Detector (PID) — The PID is a specific detector for gas chromatography. It consists of the detector assembly proper and power supply for the vacuum ultraviolet (UV) lamp. The primary maintenance that is indicated by peak tailing and/or decreased response is to frequently clean the window of the UV lamp. More extensive cleaning of the detector will require complete disassembly as described in the instrument handbook.

Nitrogen Phosphorus Detector (NPD) — The NPD is monitored for decreased signal/noise ratio, erratic behavior, and increasing voltage requirement. Possible causes for these problems include improper bead height, a dirty detector, and/or an overused collector. A dirty detector can be cleaned by blowing out the detector compartment with a stream of compressed air. The jet can also be removed and cleaned. The jet should be removed and then scrubbed with a cleaning wire, followed by rinses with methanol/acetone. The interior of the collector can be cleaned by gently blowing out the loose material with low-pressure compressed air or nitrogen. All collectors should be washed off with hexane before reinstalling in the instrument to remove any fingerprints, or other contaminants. Collector replacement occurs when the above mentioned remedies fail to solve the problem. A new collector may

take as long as 3 days to condition before it is useable. Average lifetime of a collector is approximately 14 months.

11.4.2.8. High Performance Liquid Chromatography(HPLC)

An HPLC consists of the following modules: 1) autosampler, 2) autoinjector, 3) solvent delivery system, and 4) separator system. Because of the large quantity of movable parts involved with an HPLC including pumps, rotating valves, autosamplers, autoinjectors, and problems associated with a semi-closed system operating under high pressure, only the most frequented maintenance requirements will be discussed here. It is extremely important to maintain a well-documented logbook when operating an HPLC.

Troubleshooting

Because of the many moveable parts and extensive amount of tubing, problems are often difficult to locate. To describe the symptoms in detail will not only help solve the currently existing problem but will also save an inherent amount of time troulbleshooting in the future. A list of the most frequently encountered problems and there solutions, follows:

High backpressure (generally above 330 BAR)

Replace pre-column frit or guard column.

Check the tubing backtracking from the column for a clogged line-

Change the column

Stuck or sticking autoinjector

Clean autoinjector rods with small amounts of methanol

Leaky fitting

Replace nut and/or ferrule.

Bubbles in mobile phase (Waters 600 primarily)

Ensure solvents are properly pre-filtered and degassed. However with the Waters 600 pump, mixing 100% methanol or acetonitrile with 100% water appears impossible to perform.

Leaky pumps

Clean or replace seals

HPLC Detectors

Liquid Chromatograph Photo Diode Array Detector (DAD)

The DAD is monitored for decreased signal/noise ratio and erratic behavior. Causes of decreased signal/noise include a low lamp current setting, a dirty flow cell, and/or an aging lamp. The lamp current setting can be increased and flow cells can be cleaned which may alleviate the problem. If the lamp fails to ignite and/or the decreased

signal /noise continues, the lamp probably needs replacement. Average lifetime of a DAD lamp is approximately 1 year.

Fluorescence Detector

The fluorescence detector is monitored for increased signal to noise ratio and erratic behavior. Routine maintenance includes flushing the flow cell after each use and monitoring the time clock on the lamp. Possible causes for poor performance include a dirty flow cell, bubbles in the flow cell, and a weak lamp. Average lifetime of an FLD lamp is approximately 10 months.

11.4.3. Laboratory Supplies

11.4.3.1. Sample Containers

Virtually all samples received by the laboratory arrive in sample containers provided by the laboratory. For those exceptional cases where a non-standard container is used analysis may still be performed but the data produced will be qualified appropriately. All sample containers undergo quality control checks before shipment. The various containers and specific procedures are listed below.

Field Preservation Ampules

Most sample preservation ampules are obtained from a private provider, currently Eagle Picher. Ampules containing 12.5% sulfuric acid are used in the field preservation of total and dissolved organic carbon. In addition, in house ampules containing 50% HCl are used for field preservation of samples collected for the Wisconsin Modified DRO method.

Quart Mason Jars

ONLY brand new jars are used and they are sent through the washing/sterilization process and are usually set aside for warden kits (emergency sampling kits for WDNR wardens). They are used primarily for soil, sediments and waste. After years of experience and consultation with USEPA, bottle checking of brand new quart mason jars was discontinued.

One Liter Amber Bottles

Brand new one-liter amber bottles with Teflon lined septa are sent through the washing/sterilization process and receive no further checks. After years of experience and consultation with USEPA, bottle checking of brand new amber bottles was discontinued.

Bottles may also be re-washed and reused. After washing, one jar for every 60 (2 cases) is checked by rinsing the jar with hexane (20 mL), concentrating the rinse down to 2 mL and injecting onto a gas chromatograph equipped with an electron capture detector. If a response is seen the bottles are re-washed and rechecked. If no

compounds or interferences are detected the jars are deemed clean. Once they are clean the bottles and the boxes are labeled and prepared for shipment.

Forty mL and Sixty mL Vials

Brand new glass VOC vials with Teflon septa are obtained from Industrial Glassware and come certified as clean. They are used right out of the box and are not bottle checked. It should be noted that each 40 mL sampling kit that is mailed includes a trip blank. The water used to fill the trip blank is analyzed to ensure that it is VOC free.

Summa Canisters

The six-liter and one and one half liter Summa canisters used for VOCs in air analysis are recycled and re-used. After analysis is complete the containers are "cleaned" by the repeated application of humid air and vacuum. For each batch of eight cans that is cleaned one can is filled with humidified ultra-zero air and analyzed as a sample. All results must be below the method report limit and the total of identified and unidentified peaks must fall below ten parts per billion carbon. If the results are good the cans are tagged and ready for shipment to the field. If the QC fails the cans are continually cleaned until they pass. It should be noted that an attempt will be made to choose the "dirtiest" can in the batch for checking.

Resin Columns

The XAD-2 resin is cleaned in the lab by a series of solvent extractions in a large soxhlet apparatus. Approximately 2.5 kg of resin is extracted sequentially for 24 hrs each in methanol, acetone, hexane, and methylene chloride. This is followed by sequential 6-hour extractions in acetone, hexane, and acetone. This sequence cycles the resin back to a water-miscible solvent, which is displaced from the resin by rinsing with several volumes of organic free water. Cleaned resin is stored under organic free water in amber bottles for up to three months until column preparation.

Each batch of resin is checked by taking an aliquot (500 mL) of the final hexane rinse that is approximately equal to the volume contained in a packed resin column. The volume is reduced to one mL and an aliquot is injected onto the same analytical system as the samples. If all results are less than the analytical detection limit the resin is considered clean. If the resin is not clean it will be re-extracted and reanalyzed.

A log is kept of resin batches as they are being cleaned, and of columns as they are prepared and sent to the field so that traceability of samples to individual columns and to batches of cleaned resin is maintained.

Miscellaneous

There are a variety of "unregulated" tests performed at the lab that use various containers. These include small glass jars for fish and tissue analysis, screw-cap test tube vials for serum analysis, small plastic bottles for urine analysis, poly-urethane foam puffs for pesticides in air, and aluminum foil wrapped filters used for PCB and organo-chlorine pesticide analysis of the water column. It should be noted that most of these containers are provided by the client and are out of the control of the laboratory.

Table 11.2 — Instrument Summary for Organic Chemistry

ITEM:	MANF.	MODEL	Date Acq.	DESCRIPTION
GC/MSD	Agilent	HP6980 / 5973N	2003	Used for semi-volatiles. EPA 8270/625
GC	Tracor	540	1989	FID used for Gasoline Range Organics (GRO)
Purge & Trap Autosampler / Concentrator	Tekmar	LSC 2000 / ALS 2016	1989 (LSC) 1999 (ALS)	Used with above GC
GC/MSD	Hewlett Packard	HP6980 / 5973	1997	We have two of these. One for EPA 524.2 and one for EPA 8260/624.
Purge & Trap Autosampler / Concentrator	Tekmar	LSC 3000 / Precept II	1997	We have two of these. One each used with the above GCs
GC/MS	Varian / Finnigan	Finnigan Magnum	1994	Ion Trap used for unregulated semi-volatiles and research related work.
GC with cryofocusing	Hewlett Packard	5890 Series II/FID	1994	Equipped with a cryofocuser this is used for ozone precursors.
Autosampler / Cryogenic Concentrator	Nutech	3600 / 3550A	1994	We have two of these, used with the above GCs
GC/MSD	Agilent	HP6980 / 5973N	2003	Used for air analysis for air toxics (TO-14A)
Autosampler w/ Cryogenic Concentrator	Entech	7100	2003	Used with above GC for regular toxics analysis
Autosampler w/ Cryogenic Concentrator	Entech	8000	2003	An autosampler with a heated oven, used with above GC for analysis of semi-volatiles
GC/ECD with dual tower autosampler	Hewlett Packard	5890 series II plus.	1995	Used for low-level PCB and pesticide analysis

ITEM:	MANF.	MODEL	Date Acq.	DESCRIPTION
GC/ECD	Siemens	SiCHROMAT 2-8	1989	A "heart-cutting" GC used for analysis of co-planar PCBs
GC/NPD	Hewlett Packard	5890 series II	1992	Used for analysis of NP pesticides (EPA 507, 541, etc.)
GC/ECD	Hewlett Packard	6890	1997	Used for TO-4 air analysis and other pesticides.
HPLC / DAD / Fluorescence	Hewlett Packard	HP1090 / HP1046A	1985	Used for PAHs in water / soil / and fish. EPA SW846 8310
HPLC / DAD / Fluorescence	Agilent	Series 1100	2003	Used for PAHs in water / soil / and fish. EPA SW846 8310. Also may be used for EPA 547, 549, 531, and SW846 8315A
GC/ECD	Hewlett Packard	5890 series II	1989	Packed column GC for Aroclor and pesticide screening
GC/ECD	Agilent	6890N	2003	Two mega-bore columns used for PCB aroclor and pesticide analysis.
GC/ECD	Agilent	6890N	2003	Two capillary columns used for PCB and pesticide analysis.
GC/ECD/FID	Hewlett Packard	5890 series II	1987 & 1992	Two capillary column GCs used for pesticides, glycols, diesel range organics, and PCB congener analysis
TOC analyzer	Tekmar Dohrman	Phoenix 8000 & Model 183 Boat Sampler	2003	Used for TOC in sediment and TOC/DOC/DIC in water.
Gel Permeation Chromatograph	OI Analytical	Autoprep 1000	2003	Used to remove lipid from tissue samples
Gel Permeation Chromatograph	Analytical Bio-Chem Labs	Autoprep 1002A	1986	Used to remove lipid from tissue samples
Hydrogen Generator	Whatman	H21200	2000	Two of these used for carrier and FID gas.

11.5. RadioChemistry

11.5.1. Laboratory Facilities

11.5.1.1. Physical Space

The Radiochemistry Department occupies about 1600 square feet of the State Laboratory of Hygiene's Agriculture Drive facility. The sample preparation area is adjacent to, but separate from the counting room. Radioactive standards are kept in a locked storage cabinet. The lab is equipped with four hoods that are six feet in length, ample bench top space, a drying oven, digital analytical and top loading balances, standard laboratory glassware, and chemical reagents for routine work. The Department also has space for both cold storage of samples and room temperature storage adjacent to the sample preparation area. Additionally, the lab has a six-foot hood and work space near the loading dock for soil grinding. The entire building is security locked.

The counting room is equipped with state-of-the-art equipment that includes: a twelve detector low background alpha-beta counter, a single detector low background alpha-beta counter with an automatic sample changer, three intrinsic germanium detectors using Aptec gamma analysis software, a liquid scintillation analyzer, EG &G alpha spectrometer (four detectors), and ten Ludlum alpha scintillation cell counting systems. A complete list of laboratory equipment is summarized in the Table 11.3.

11.5.1.2. Computer Facilities

The primary computer used by the Department is the Data General housed at 465 Henry Mall. This is a Unix based system maintained by the Information Systems (IS) section of the Laboratory. The Department also has 5 PC's running Windows NT and 2 running Windows 98. All of the PC's are connected via an NT network administrated by the IS Section. All of the data entry, analysis and reporting software is custom written, and is therefore adaptable to the changing needs of our customers.

11.5.2. Laboratory Equipment and Instrumentation

11.5.2.1. Ortec Four Channel Alpha Spectroscopy System. (Instrument System ID #3)

This is a recent acquisition. The method manuals and instrument manual are under development. The analyses performed on this piece of equipment were for research purposes only.

11.5.2.2. Protean - Low Background 12 detector α-β counter. (Instrument System ID #1)

A Sr90 sealed un-calibrated source and a Po210 sealed un-calibrated source are counted each day that the instrument is used for both alpha and beta monitoring. The daily control check software informs the user if the

instrument has passed the performance check. Plots of the data are also available to the user for inspection. The plots indicate the mean of data used to determine the control limits as well as the warning limit (2 standard deviations) and the upper control limit (3 standard deviations). If the count rate is outside of the upper control limit, the source may re-counted up to three times. If the count rate for that detector remains outside of the upper control that detector is not used that day and the reason is investigated. Important information concerning the instrument performance is stored in the electronic log book in the equipment database.

A background count of at least 50 minutes is taken each day of use. The daily performance check software informs the user if the instrument has passed the background check. Plots of the data are also available to the user for inspection. The plots indicate the mean of data as well as the upper control limit set at 1.5 cpm for the beta channel and at 0.2 cpm for the alpha channel. A lower control limit for the beta channel has been set at 0.2 cpm. These values were chosen based on a review of the accumulated data. If the count rate is outside of the control limit, the detector is not used until the problem is resolved. See ESS RAD IOP 009 Protean Alpha Beta for detailed instructions on decontamination procedures and other trouble shooting techniques. Important information concerning the instrument performance is stored in the electronic log book in the equipment database.

11.5.2.3. Packard 1500 Liquid Scintillation Analyzer. (Instrument System ID #4)

Carbon-14 and tritium standards are counted each day of use to monitor instrument stability. Efficiencies are compared to values recommended by the manufacturer. A summary of the control information, with unacceptable results flagged, is mailed to the user and the QC manager when the information is transferred to the mainframe computer. Plots of the data are available for inspection by the user.

If any of the control parameters are unacceptable, the instrument is calibrated using the automatic *self-normalization and calibration* procedure.

The instrument is under a service agreement. Therefore, when ever the system does not perform according to manufacturer's specifications, call for service. Do not use the instrument until the service has been completed.

11.5.2.4. Aptec/Canberra/PGT Gamma Spectroscopy System (Instrument System ID #5, 6, & 7)

A Linearity standard consisting of radium-226 in a salt matrix sealed in a 500 mL merinelli beaker along with an americium-241 disk is counted for 10-20 minutes at the beginning of each working day for (systems 5 & 6). The calculated centroid of each of these peaks is automatically recorded, and checked against the known value to see if it is within allowable control limits. If the results are outside of control limits, the analyst is informed. This provides a check on the energy calibration and linearity of the Multi-Channel Analyzer (MCA) system.

Efficiency and Resolution Check. — The Co-60 standard is counted at a prescribed distance (25 cm) to determine the resolutions and counting efficiencies. Any fluctuation in the resolution would be an indication of detector deterioration or system malfunction. Detector efficiency, particular to each counting geometry and gamma energy, is a factor in all calculations. This parameter must be carefully monitored to assure that no change has occurred.

If the daily checks are out of control specific instructions are given in Appendix C of the *State Laboratory of Hygiene Gamma Spectroscopy Standard Operating Procedure.* Sample analysis will be discontinued until the problem is remedied.

A Linearity standard consisting of K40, Pb210 and Cs137 is counted until 1000 net counts are obtained in each peak (systems 7). The calculated centroid of each of these peaks is automatically recorded, and checked against the known value to see if it is within allowable control limits. If the results are outside of control limits, the analyst is informed. This provides a check on the energy calibration and linearity of the Multi-Channel Analyzer (MCA) system.

Efficiency and Resolution Check. — The Pb210 standard is counted on top of the appropriate sized ointment can filled with sodium chloride. Any fluctuation in the resolution would be an indication of detector deterioration or system malfunction. Detector efficiency, particular to each counting geometry and gamma energy, is a factor in all calculations. This parameter must be carefully monitored to assure that no change has occurred.

If the daily checks are out of control specific instructions are given in Appendix C of the *State Laboratory of Hygiene Gamma Spectroscopy Standard Operating Procedure.* Sample analysis will be discontinued until the problem is remedied.

Once a month a background spectrum of empty detectors is collected. To be acceptable all of the ROIs should have less than detectable activities. Note: detector #1 has measurable K-40 which is inherent in the shield or pre-amp materials which can not be removed. Detector #2 has measurable uranium progeny inherent in the shield or pre-amp materials which can not be removed.

If greater than detectable activities are found other than described above thoroughly clean the detector surfaces and recount the background. Contact instrument manufacturer for advice. If the contamination is not removable, use this new background when background subtraction is required.

11.5.2.5. Gamma Products - low background single detector α-β counter (Instrument System ID #2)

Strontium-90, Cesium-137, and thorium-230 standards are counted on each work day to monitor instrument stability. This data is stored on the instrument PC. A control limit plot is available to the user.

The limits for the control charts are dynamically determined by previous history.

If the daily count is outside the average or range control limit, repeat the count. If the controls are still out, corrective action is taken and the counting of samples on the instrument is discontinued until the problem has been identified and corrected.

The system is also monitored for the appearance of trends that may signal instrument failure and are treated the same as when the standards are out of the control limits. Any adjustments or maintenance of the system is recorded in the instrument's maintenance log.

Background samples appropriate to the sample type are interspersed amongst the samples as they are counted. An upper control limit of 0.15 cpm has been set for the alpha background for an empty planchet. An upper control limit of 2.0 has been set for the beta background and a lower control of 0.2 has been set for the beta background for an empty planchet. These values were chosen based on a review of the accumulated data. If the count rate is outside of the control limit, the detector is not used until the problem is resolved. See ESS RAD IOP 006 for detailed instructions on decontamination procedures and other trouble shooting techniques. Important information concerning the instrument performance is stored in the electronic log book in the equipment database.

11.5.2.6. Ludlum Scintillation Cell Counters (Instrument System ID #8,10-28)

A thorium-230 standard are counted on each work day to monitor instrument stability. The plots indicate the mean of data used to determine the control limits as well as the warning limit (2 standard deviations) and the upper control limit (3 standard deviations).

Backgrounds are determined on these systems by placing a clean scintillation cell on the detector and repeatedly counting the cell for either 100 or 990 minute cycles.

If the background rate is higher than is acceptable (less than 1 cpm), do not use that cell to perform a sample analysis. Clean the cell as described in the procedure and replace it on the counter for additional background counts.

11.5.3. Efficiency Determination

- Each instrument's efficiency is determined yearly for each nuclide in each procedure or counting geometry.
- Efficiencies are also confirmed in the event of major repair or instrument modification.
- The specific instructions for preparing standards for efficiency determinations are found in the standard operating procedures for the various analytical procedures.

11.5.4. Laboratory Supplies

11.5.4.1. Pipettes

The pipettes used for radon in water are calibrated when the cocktail is changed. The procedure is described in the Radon in Water SOP.

The instructions in the SMI manuals state that gravimetric calibrations are not required if the technique to adjust the pipette is followed correctly. Check the adjustment quarterly and record the results in a log. If a confirmation of an SMI is needed by a gravimetric technique, follow the instructions in ESS RAD GENOP 016

11.5.4.2. Balances

All analytical balances are on a preventive maintenance schedule. The balances are inspected and cleaned annually. The calibration of the analytical balance is checked daily with three internal class "s" weights. If the analytical balance exceeds the accuracy tolerances, the supervisor is notified and the problem corrected before the balance is used. All pertinent information is documented in the appropriate logbook.

The calibrations of top loader general purpose balances are checked the day of use using three internal class "s" weights. If the balance exceeds the accuracy tolerances listed in the logbook, the problem will be corrected before the balance is used. All pertinent information is documented in the appropriate logbook. (See ESS RAD IOP 004 for a more detailed account).

11.5.4.3. Ovens, and Coolers

See General Section 11.1

11.5.4.4. Centrifuge

The motor is cleaned and oiled as needed.

11.5.4.5. Laboratory Reagent Grade Water

Reverse Osmosis (RO) Water--See General Section 11.1

Pure Lab Plus UV/UF ASTM Type I Polisher--See General Section 11.1

Table 11.3 — Laboratory and Instruments Summary for Radiochemistry

ITEM:	MANF.	MODEL	ACQ.	DESCRIPTION
analytical balance	Ohaus	Explorer Series	Feb 2000	Max 210g, 0.1mg
top loader balance	Ohaus	Explorer Series	Feb 2000	Max 2100g 10mg
Weight sets	Troemner	1g-50g and 100g –1kg	12/99	Calibration weight sets
Desiccator	Labconco	55300	11/01/80	Glass desiccator cabinet, 12 shelves
Hot Plates	Corning Thermolyne	PC-100 Cimerac 3	1981-2001	10"x10" surface
Cooler	Jordon Scientific Products	1-SPST-5GS/S	03/99	50 cu ft sliding glass door
Oven	Fisher	IsoTemp 500 series	Used/refurbished	Gravity convection., to 200°C
IR Heat Lamps	Fisher	11-504-10V4	1980-	Two heaters per department
Freeze Drier	Labconco	Freezone Plus 6	10/99	16 port drying chamber
Centrifuge	IEC	model k	1978	12 x 50mL, floor model
Alpha beta low background proportional counter	Gamma Products	G500 automatic systems	1998	gross alpha beta counter automatic sample changer 50 sample capacity P-10 gas
Alpha beta low background proportional counter	Protean	PIC	2001	Simultaneous on 10 detectors, P-10 gas
Liquid scintillation	Packard	TC-1500	09/02/87	adj. discriminator channels
Radon gas counters	Ludlum	2000	1983 & 2001	2 cells per system
Gamma spectroscopy	Aptec analysis software		1992, 1996, 1997	3 intrinsic germanium 2-Canberra crystals, 1 PGT
Alpha Spectroscopy	EG &G	OTETE Plus	8/99	4 PIPS, Ultra AS 600

11.6. Water Microbiology

This section describes the checks and monitoring procedures that should be performed on materials, supplies, instrumentation and the physical facility. These quality control checks should be documented completely and recorded as performed.

11.6.1. Laboratory Facilities

The Laboratory is approximately 1100 square ft for total coliform testing, 430 square ft for pathogen testing, and separate glassware washing and media preparation facilities. The Total Coliform incubation is done in Room 203. The Pathogen Testing, where crypto and giardia and other pathogens are performed, is located in Room 202. The Dark Room is Room 202B for the fluorescent microscope. Glassware washing is located in Room 206 and Media Preparation is located in Room 206C. The heating and air conditioning system is equipped to provide a continuous supply of fresh air when laboratory work is in progress. Ambient temperature is controlled to be within 65 °F to 80 °F range. Workbenches are cleaned with disinfectant.

11.6.2. Laboratory Equipment and Instruments

11.6.2.1. pH meter

Orion Model 520A and Orion Model SA520 digital pH meters are used for the determination of pH values of media and have a precision and relative accuracy of ± 0.1 pH.

Model 520A - serial number: 29347 located in media room

Model 520A- serial number: 019506 located in pathogen lab

Model SA520 - serial number: S030A located in media room

With each use, the probe is rinsed with distilled water before and after each measurement. The meter is standardized against at least two standard buffers (pH 4.0, pH 7.0 pH 10.0) before each use and the results recorded.

Slope must be between 95% and 105%. If not within 95% and 105%, the probe and pH meter are checked and replaced if needed. Buffer solutions are not reused.

A third lot of standard is read after the slope has been calibrated and recorded.

11.6.2.2. Balances — 3 - Meter Toledo, 1-Model PG503-S - Serial Number: 1117441699, 1-Model AG245- Serial Number: 117441766, Scout - Model SC2020 - Serial Number: BJ457615

The balance is cleaned after each use, and a service contract is maintained to provide annual service. Balances are calibrated monthly with ASTM Class 1 weights that are re-calibrated each year and the results recorded.

METTLER TOLEDO PG503-S	
ASTM CLASS 1 WEIGHTS	TOLELRANCE
100 G	0.25 mg
50 G	0.12 mg
10 G	0.05 mg
0.1 G	0.01 mg

METTLER TOLEDO AG245	
ASTM CLASS 1 WEIGHTS	TOLERANCE
1.0 G	0.0340 mg
0.1 G	0.01 mg
0.01 G	0.01 mg
0.001 G	0.01 mg

11.6.2.3. Walk-in Incubator- Room 203 C

A Johnson Control system capable of maintaining a constant temperature of 35 ± 0.5°C is used. A temperature-recording chart is used and changed weekly to monitor temperature stability. Temperatures are recorded in the morning and afternoon (with at least four hours between readings) for days in use, except weekends and holidays when temperature is recorded only once.

Temperature must be within 35 ± 0.5 °C to be in control. Thermometers graduated in 0.1 °C increments are placed on top and bottom shelves of the use area. The thermometer bulbs are immersed in liquid. All shelves are cleaned periodically.

Incubator

Innova 4230 Refrigerated Incubator Shaker, Serial # 891220419, is a forced air incubator capable of maintaining a constant temperature of 36° C± 1°C is used for coliphage testing. Temperatures are recorded in the morning and afternoon for days in use. Temperatures must be 36° C± 1°C to be in control. Incubator is cleaned periodically. (This incubator is also used for special projects when coliphage testing is not being performed. The temperature settings could be changed for the project.)

Incubator/Shaker

Brinkimann Orbimix 1010 DT, model 543.12310.08 0, serial # 109700640, Brinkman Incubator 1000, Heating Model 549.90020.08 0, serial # 039800400 is a combination of an incubator and shaker capable of maintaining a constant temperature of 36° C± 1°C is used for coliphage testing. Temperatures are recorded in the morning and afternoon for days in use. Temperatures must be 36° C± 1°C to be in control. Incubator is cleaned periodically.

Incubator

Fisher Scientific Model 307C, serial # 101N0016, Frigidaire Home Products Model FFU20F9GW4, serial # WB05116887 is an incubator capable of maintaining 37° C ±1°C and used for the *Helicobacter pylori* or 42 ±1°C for *Salmonella* or 41.0 ± 0.5° C for enterococci. Temperatures are recorded in the morning and afternoon for days in use. Temperatures must be 37° C ± 1°C to be in control for *Helicobacter pylori*. Temperatures must be 42 ±1°C to be in control for *Salmonella*. Temperatures must be 41.0 ± 0.5° C to be in control for enterococci.. Incubator is cleaned periodically.

VWR Scientific Products, Shel Lab, Model # 2005, serial # 1107700 is a forced air incubator capable of maintaining 10°C ± 2° to 50° C that is used for special projects. Temperatures are recorded in the morning and afternoon for days in use. Temperatures must be within ± 2°C to be in control. Incubator is cleaned periodically.

11.6.2.4. Water Bath

(2) Blue M Model 1130A-1, serial numbers: MW-5705; MW-5753; circulating water bath capable of maintaining a constant temperature of 44.5± 0.2°C is used for fecal coliform and *E.coli* analyses. Temperatures are recorded in the morning and afternoon for days in use. Temperature must be 44.5 ± 0.2 ° C to be in control. Water baths are cleaned weekly.

11.6.2.5. Thermometers

All thermometers are calibrated against a NIST thermometer annually and the results recorded or a NIST thermometer is used for temperatures. All mercury thermometers are checked periodically for breaks in mercury column.

11.6.2.6. Autoclaves

2 - Eagle® 3000 Medium Sterilizer, AMSCO Model 3031-S, Serial Number : 010519801 located in 206 (media room); Serial Number : 010659807 located in 206 (glass washing room) equipped with a steam line, pressure and temperature gauges, a recording device that records

date, time, type of cycle, which cycle, internal temperature, time of cycle, psi, problems with the cycle and an automatic timer, is used for sterilization of reagents, media, glassware and contaminated items.

The recording tapes are dated and kept for 1 year only because the ink fades. The date, contents, sterilization time and temperature, total time for each cycle, and analyst's initials are recorded each time the autoclave is used in a logbook.

Chamber shelves are wiped clean frequently and the entire chamber is cleaned periodically. After each cycle the front basket is checked for debris.

A preventive maintenance contract with Steris includes multiple inspections on an annual basis.

The autoclave maintains sterilization temperatures during operation and completes its entire cycle within 45 minutes when items are sterilized 12-15 minutes. The automatic timing mechanism is checked quarterly with a stopwatch. It must be accurate within one minute to be in control. The pressure and time is NIST certified annually by a Steris technician.

11.6.2.7. Hot Air Oven

VWR Scientific Products, Model 1690, Serial Number: 0100298, Watlow control, Series 981, equipped with an accurate long-stemmed thermometer that maintains at a minimum of 170°C for sterilizing glassware, sticks and swabs.

The oven is programmed to sterilize for 3 hours at 185°C. The temperature is recorded in the oven logbook including date, sterilization time and item description. With every sterilization load a strip of *Bacillus subtilis,* population 10^6 organisms, are tested for kill. A thermometer in sand is also placed in the oven and temperature is recorded. The oven is also used at least weekly for sterilization of clinical glassware at night. A spore strip is tested for kill with each load.

The oven is also programmed to dry glassware for 1 hour at 90°C.

11.6.2.8. Refrigerator

(2) Fisher Scientific Isotemp, model 15105A14, serial numbers: Y24H-411208-ZH located in 203 (lab) and Y24H-411207-ZH located in 206 (media room) are maintained at 1-5°C and is used for the storage of prepared media, reagents, cultures and samples.

Temperature is checked twice daily and results are recorded in temperature book. The refrigerator is cleaned and unused materials are periodically discarded. Thermometers are calibrated in 1°C increments and immersed in liquid. Temperature must be between 1 and 5°C to be in control.

VWR Brand by Revco, GS Laboratory Equipment, model R421GA14, serial # X28J-460645-YJ, is maintained at 1-5° and is used for the storage of prepared media and reagents.

Temperature is checked twice daily and results are recorded in temperature book. The refrigerator is cleaned and unused materials are periodically discarded. Thermometers are calibrated in 1°C increments and immersed in liquid. Temperature must be between 1 and 5°C to be in control

Sub-Zero, model # 601R/F, serial # M1597945, is maintained at 1-5° and is used for the storage of prepared media and reagents.

Temperature is checked twice daily and results are recorded in temperature book. The refrigerator is cleaned and unused materials are periodically discarded. Thermometers are calibrated in 1°C increments and immersed in liquid. Temperature must be between 1 and 5°C to be in control.

General Electric Refrigerator with Freezer: no serial number found, is maintained at 1-5°C and is used for the storage of media and reagents. The freezer is maintained at -15°C and is used for the storage of media and reagents.

Temperature is checked twice daily and results are recorded in the temperature book. The refrigerator and freezer are cleaned and unused materials are periodically discarded. The thermometers are calibrated in 1°C increments and immersed in liquid. Temperature must be between 1 and 5°C to be in control for the refrigerator and between -15 to -20°C for the freezer to be in control.

11.6.2.9. Freezer

Fisher Scientific, Model #97-926-1 (no serial number) is maintained at -18°C or less for the storage of reagents and serum. The temperature must be between -18°C and -22°C to be in control. Temperature is checked twice daily and results recorded in logbook.

Sanyo Medical Freezer , model MDF -U536D, serial number: 70705941, (located in room 200) is maintained at -26°C to-34°C for storage of samples and reagents. The temperature must be between -26°C to-34°C to be in control. Temperature is check once daily and results recorded in logbook.

Forma Scientific Freezer, model #8523, serial number: 21272-1991, (located in room 100) is maintained at - 76 to -84°C for storage of samples and reagents. The temperature must be between -76 to -84°C to be in control.

11.6.2.10. Stomacher

Lab Blender Seward Model #3500, serial #28825 for stomaching polypropylene filters. Moved to storage.

11.6.2.11. Beckman J-6M/E centrifuge, serial #J6D801, with swinging bucket.

The rotors have capacity for 15ml, 50ml, and 250ml conical tubes.

The centrifuge calibrated annually and a preventive maintenance contract is maintained. Maintenance records are kept in the maintenance folder in a drawer by the centrifuge.

11.6.2.12. Water Bath

Fisher Scientific ISOTEMP 210, Serial Number: 802N0152 (located in room 203), Fisher Scientific ISOTEMP 215, Serial Number: 810N0227 (located in Room 206), Fisher Scientific ISOTEMP 220, Serial Number: 926N0288 (located in Room 202), Precision Model 288-115, Serial Number: 69081054, (located in Room 202), Precision Scientific Co, No serial number, (located in Room 206) — used for tempering media. The units are adjusted to the correct temperature & the temperature is checked with a thermometer. The baths are cleaned periodically.

11.6.2.13. Incubator — M Blue M

Given to Bioaerosol unit.

11.6.2.14. Thelco-GCA/Precision

No Serial Number, The incubator is adjusted to 41 °C. The incubator must be 41.0 ±

11.6.2.15. Hoefer Manifold (2) - moved to storage

The manifold includes a filter holder (25mm Model FH225), and a Ten Place Filter assembly with collection box for 25 mm filters. It comes complete with ten stainless steel weights, 1" PVC filter holding plate with vacuum gauge and collection box.

The vacuum is adjusted for flow rate with each sample set. The manifold is cleaned after every use with 0.01% Tween 80 solution and then 10-20 ml of type II water. The box is washed with Alconox and rinsed with tap water and DI water. The sealing ring on the bottom of the weights is reground if scratched or dented.

All weights or wells cleaned with each use. They are soaked and autoclaved in DI water and Alconox solution at 121°C for 15 minutes. After autoclaving, they are rinsed three times with DI water. The wells are scrubbed with a brush, rinsed with tap water and DI water.

11.6.2.16. Vortex

Vortex Genie, Model G-560, serial #2-227759 and serial # 211696 for mixing samples. No Q.A. required.

11.6.2.17. Slide Warmer

Fisher, Model 77, serial #30600187, temperature maintained at 37-40°C for clearing filter slides for crypto and Giardia. Slide warmer is calibrated yearly.

11.6.2.18. Microscope

Olympus BMAX, BX50F, serial #3C.823, Burner generator model BH2RF6T3, serial #101079, U-M566(BP-460-490) FU=ITC Wide band IB filter cube, U-M526(BP 330-385) DAPI green, wide band filter cube, U-MWG(BP510-550) Syto 59 Pi, filter cube, U-P100 DIC slider with U-CD119 100X prism and U-CD125 40X oil prism, Super Wide field trinocular Observation Tube with 24 degrees eyepiece inclination.

Objectives - 20X, 40X, 100X for epifluorescence, DAPI, and PI

Objectives - 40X, 100X for DIC (differential interference contrast)

Mercury lamp replaced every 200 hours. Koller illumination established with each use. DIC checked with every control slide.

The scope is cleaned once a year under a microscope maintenance agreement.

11.6.2.19. Laboratory Reagent Grade Water

See General Section 11.1

11.6.2.20. Pure Lab Plus UV/UF ASTM Type I

Pure Lab Polishers provide Type I water which is used in the preparation of media and reagents. There is a service contract with US Filter to maintain the departments. Every 6 months US Filter will exchange the carbon filter, 2 Ultra Pure Mixed bed Cartridges and organic scavenging Type II ultra pure anion resin. The UV filter will be changed based on the number of hours of use. This water is used to prepare media, reagents and dilution/rinse water.

Weekly laboratory staff will sanitize the Polisher departments with chlorine tablets. The date is recorded in the log.

Conductivity and total residual chlorine determination are performed at least monthly, conductivity meter is calibrated monthly with a 0.01 M KCl solution.

The test for bacteriological quality for laboratory pure water is performed once a year on the reagent grade water and the results recorded. This test is no longer required by the EPA for Type II water or better.

Metals analysis for Cd, Cr, Cu, Ni, Pb and Zn is performed on an annual basis and the results recorded.

Heterotrophic Plate Counts are performed by the pour plate method monthly and the results recorded.

Total Chlorine Residual Test is done monthly using DPD and results are recorded. This done by the Biomonitoring Unit and records are kept in their logbooks.

11.6.2.21. Gelman Membrane Filtration

The components of these units are made of autoclavable, UV sterilization polysulfone funnels and bases which lock together by means of twin magnets. With each use the funnels and bases are inspected for scratches or worn spots and checked for worn edges.

Units are washed after each day and autoclaved before the next use by putting in an autoclave bag. They are autoclaved on the dry cycle at 121°C for 15 minutes. Between each sample the units are UV sterilized for a minimum of 2 minutes.

11.6.2.22. UV Sterilization — A Millipore UV sterilizer.

Quarterly, the performance of the sterilizer is checked by streaking petri dishes with a heavy inoculum of *Proteus vulgaris*, and exposing them to the lower and upper bulbs for 2 minutes. A 95% kill is required. Weekly, the insides of the units are cleaned with ethanol. Monthly the UV radiation is check with a radiometer.

11.6.2.23. Forceps

Stainless steel forceps with non-corrugated, blunt tips are used for handling membrane filters. With each use, tips are immersed in small beaker of 95% ethanol and sterilized by igniting in a flame produced by a Bunsen burner (Fireboy).

Splinter tip stainless steel forceps for handling sartorius filters. With each use, washed three times with eluting solution and rinsed three times with Type II (RO) water.

Blunt end tip stainless steel forceps with non-corrugated tips are used for handling membrane filters for crypto and *Giardia* samples.

11.6.2.24. Inoculating Equipment

Inoculating needles and loops (at least 3mm in diameter) are made of B and S gauge 24 platinum-iridium wire (15%) fitted in suitable needle holders possessing nickel-plated, brass clamps. Sterile plastic inoculating loops of at least 3 mm are also used. Wood sticks are hardwood and are dry-heat sterilized.

Metal needles and loops are sterilized before and after use with a Bunsen burner. Wood sticks are packaged in aluminum pipette boxes and dry heat sterilized for a minimum of 2 hours at 170 - 180°C.

11.6.2.25. Glassware Washers — 2 Steris, model Reliance 400 glassware washers with a printer to record the time, date, cycle name, minutes of the cycle and problems.

The cycle used for water microbiology is the standard cycle: 8 minutes of acid wash with Steris Organic Acid Cleaner CIP 220, 8 minutes of detergent wash with Steris Liquid Alkaline Detergent(nonphosphate), 3 minutes of hot water tap rinse, 3 minutes of hot water tap rinse, 10 sec of non-recirculating Type II water, 10 sec of non-recirculating Type II water, and 15 minutes of dry time in washer and, if needed, at least 1 hour in the drying oven.

The glassware is spot checked for pH with 0.04% bromothymol blue.

Detergent suitability testing is performed once a year and results are recorded in the quality control book.

The glasswashers are under a service maintenance agreement.

11.6.2.26. Cimarec 2 Thermodyne Stirrer/heater

Used for mixing and heating media and reagents. No QA required.

11.6.2.27. Lab-Line Incubator

Incubator is in Room 206C , Serial Number: 0798-0243, and is set to 55°C for the incubation of spore ampoules of *Bacillus stearothermophilus*. Temperatures are taken twice daily and recorded in the logbook. The temperature must be within 50°C to 55° to be in control. The incubator is cleaned on a weekly basis.

11.6.2.28. Biological Safety Cabinet

Baker SterilGARD, Model SGII-600, NSF classification: Class II, Type A. The supply and exhaust ULPA filters are zero-probed ULPA. The high-velocity return air slots prevent the escape of particulates and ensure no unfiltered air enters the work area, prevent gases, vapors or particulate from coming up behind the window and escaping into the laboratory, and prevent room air from migrating down behind the window and

contaminating the work area. The SterilGARD has ultraviolet light for decontamination of work surface

The unit is certified annually by UW Environmental Health Services. Before and after every use the surface is disinfected with 70% ethanol. The unit may also be disinfected with the internal UV light.

11.6.2.29. Edgegard Laminar Flow Hood

Edgegard, Baker Company, Model EGB-6252, Serial Number 63982 in Room 206. The circulated air is filtered by a 99.9% HEPA filter so the internal work area is clean and particle free. This hood is used only for media preparation.

The unit is certified annually by UW Environmental Health Services. Before and after every us the surface is disinfected with 70% ethanol.

11.6.2.30. Fume Hood

Fisher Hamilton SafeAire Fume Hood with hookup for nitrogen, vacuum, and water. Fume Hood ID#'s: 8064-039, 8064-040, 8064-037. They are located in rooms 206 and 202. UW Environmental Health Services annually certifies the units.

11.6.2.31. (2) Quantitray Sealer Model 2X

Model # 89-10894-00, serial #01255 and serial #02230 are used for the estimation of bacterial counts. Exterior surface is wiped with water on a weekly basis. Monthly 100 ml of water with dye is sealed in a quantitray. If the dye leaks, the sealer will be replaced.

11.6.2.32. Nalgene 10 L carboy with bottom delivery

11.6.2.33. Corning Stirrer Plate and Stirrer bars, Model #PC351. No QC required.

11.6.2.34. Wrist Action Shaker — Lab-Line multi line wrist action shaker, model #3589 Serial # 0696-0143, located in room 202. The unit has side arms, 8 clamps and variable speeds. Machine is wiped down with alcohol after use.

11.6.2.35. Fairbanks Industrial Scales

Model #: 1124, Serial #: C 23406, located in Room 203. Scales are preset to 50 pounds and used to monitor the weight of MERI barrels for discard.

11.6.2.36. High Pressure Vacuum Filters (2)

High pressure vacuum filter plate set on tripod stand for 142 mm filters. Cleaned by placing in hot sink for 30 minutes between periods of use. Supplier for extra parts is: Aventech, 6723 Sierra CT, Suite A, Dublin Ca 94568, Phone: 800-334-7132

11.6.2.37. Dynal Rotator Mixer, Model RKDYNAL, Serial#: 1197-801, located in Room 202.

11.6.2.38. Dynal MPC-1 Particle concentrator for 10 mL tubes, located in Room 202.

11.6.2.39. Dynal MPC-S Particle concentrator for microcentrifuge tubes.

11.6.3. Laboratory Supplies

11.6.3.1. Membrane Filters

Membranes used are pre-sterilized Millipore Type HA, 47 mm white, grid-marked and have a pore size of 0.45μm for drinking water samples, Kstrep and enterococci. Membranes for fecal coliform and *E.coli* are pre-sterilized Millipore Type HC, 47 mm white, grid-marked and have a pore size of 0.7 μm.

Membrane filters are dated when received and the lot number recorded. The sterility of each lot is checked by the manufacturer and certification sent with each lot. A sterility check is done for each lot received in the lab. During routine use membranes are inspected for misshapen qualities, distorted gridlines, diffusibility and brittleness. After incubation, membranes are examined for colony development and distribution, and for gridline ink interferences.

11.6.3.2. Petri dishes

Pre-sterilized, plastic disposable petri dishes (60 X 15 mm) with tight-fitting lids are used in membrane filter procedures and pure culture isolation.

Pre-sterilized, plastic disposable petri dishes (15 X 100mm) with loose-fitting lids are used for pour plates.

Pre-sterilized, plastic disposable petri dishes (150 X15 mm) with loose-fitting lids are used for pour plates, cultures and filter holders.

11.6.3.3. Sample Bottles

- Bacterial Sample Bottles

  Sterile, wide mouth, high density polyethylene plastic bottles capped with a polyethylene cap . Various sizes are used for testing the most common sizes are 500 mL and 1 L. If the sample is chlorinated sodium thiosulfate is added to each bottle before sterilization. EDTA may also be added to these bottles for high metal content in water. Bottles are sterilized with loose caps and tighten after sterilization. A sterility check is performed on one of each batch of sample bottles and recorded in the quality control logbook.

Sterile, wide mouth, high density polyethylene plastic bottle capped with a black phenol cap with a rubber liner that has a 250 ml capacity is used for sample collection. Prior to sterilization 0.25 ml of a 10% sodium thiosulfate solution is added to each bottle. Bottles are sterilized with loose caps, which are then tightened after sterilization. A sterility check is performed on one of each batch of sample bottles sterilized and recorded in the quality control logbook.

- Sterile Polystyrene with Thiosulfate

  A clear, sterile polystyrene reaction vessel with a tablet of sodium thiosulfate enough to neutralize at least 10 ppm of chlorine in a 165 ml water sample. The manufacturer uses radiation to sterilize bottles. A certificate of irradiation is provided. A sterility check is done on one bottle from each box received. Results are recorded in the quality control logbook. A volume check and fluorescence check is done on one bottle from each box received. Results are recorded in the quality control logbook.

- Sterile Polystyrene

  A clear, sterile polystyrene reaction vessel capped with a polypropylene plastic cap that has 165 mL capacity used for sample collection. A sterility check is done on one bottle from each batch sterilized and recorded in the quality control logbook.

- Wide mouth bottle

  1 gallon part no F10638-1010 (4/cs) Obtained from Bel Arts Products, 800-4BELART or 13082 VWR. Sample bottles are non-sterile, but clean. Recycled after use by rinsing with tap water immediately after emptying. Standard glassware wash cycle#8. Caps are not put through glass washer but washed in hot sink with hot soapy water, rinsed with type II water and air dried.

11.6.3.4. Graduated Cylinders

Class A, Fisherbrand Certified, 250 ml (Cat# 08-557E), serial #'s:F6808, F6867, F6868, F6807; 500 ml (Cat# 08-557F) serial #'s: F7832, F7830, F7834, F7847; 1000 ml (Cat# 08-730-30) serial #'s: B7949, B7901, subdivided in increments of 2 ml, 5 ml, and 10 ml and calibrated to deliver at 20°C are fitted with metal foil covers and sterilized before use.

11.6.3.5. Crypto and Giardia membrane filters

Millipore Isopore membrane filters, 3.0 um, 142 mm, TSTB

Cellulose Acetate 0.2 um, 25 mm

Polycarbonate membranes, 1.0 um, 25 mm

11.6.3.6. Slides, glass microscope

2" x 3", Lot number is recorded in the crypto and giardia Q.C. book.

11.6.3.7. Fingernail polish

Any brand of clear nail polish. After each use the brush is rinsed three times with eluting solution and DI water.

11.6.3.8. Gloves

10% nitrile powder free medical exam gloves. A new set is used any time there is an interruption that requires removal during the processing of a sample, or when any contamination of the gloves occurs.

Powder free latex exam gloves. A new set is used any time there is an interruption that requires removal during the processing of a sample, or when any contamination of the gloves occurs. The gloves are also used by glassware staff for the pickup of contaminated glassware and biohazardous waste.

11.6.3.9. Scalpel — Disposable sterile scalpels, used only once per sample then discarded.

11.6.3.10. 4L Beaker — Plastic with handles.

11.6.3.11. Centrifuge bottles and tubes

Polypropylene 250 ml conical centrifuge tubes, polystyrene 50 ml conical centrifuge tubes, and 15-ml polystyrene conical centrifuge tube.

11.6.3.12. Micropipets: — Gibson p1000 K31978L, p 1000K31967, p200 L14868A. With each use a new tip is used.

11.6.3.13. Cannula — 14 gauge stainless, 4 in.

11.6.3.14. Syringe — Corning 25-mm square, no. 1.5.

11.6.3.15. Flasks

Various sizes of Erlenmeyer borosilicate glass flasks with either a lined screwed cap or metal foil cover are used in preparation of culture media. Sterilized in the autoclave drying cycle for 15 min at 121°C.

11.6.3.16. Culture tubes

Borosilicate glass test tubes (20 X 150 mm) are used with screw caps for multiple tube procedures, agar slants, EC broths.

Borosilicate glass test tubes (10 X 75 mm) are used as fermentation tubes in multiple tube procedures.

Borosilcate glass test tubes (16 X 125 mm) with screw caps are used for agar slants, saline solutions and brilliant green bile broth.

Leighton tube, Dynal part number 740.03

11.6.3.17. Pipettes

Fisherbrand 10-ml sterile disposable polystyrene serological pipette, increments in 0.1 ml with a cotton plug. Calibrated to deliver/blow out at 20°C. Catalog # 13-678-14A

Falcon 2.2-ml sterile disposable polystyrene serological pipette, increments in 1.0, 2.0, and 2.1, 2.2 ml with cotton plug. Calibrated to delivered/blow out at 20°C. Falcon Catalog # 7555.

Fisherbrand 1.0 sterile disposable borosilicate glass, increments in 0.01 ml with cotton plug. Calibrated to deliver/blow out at 20°C. Catalog # 13-678-27C.

Pipettes are used once and thrown in the Meri Barrel for waste disposal.

11.6.3.18. Disposable sterile polypropylene containers — 8oz., 250 ml with sterile lids purchased for *E.coli* 0157 and *Salmonella* procedures.

11.6.3.19. Lens paper — Without silicone, Fisher Cat #11-995

11.6.3.20. Gelman Filter — Sampling capsule with 1300 cm^2 polyethersulfone filter media and one half inch inlet and outlet fittings, product #12110

11.6.3.21. Well Slides — Cel-line Associates (Erie Sceinces), PO Box 648 Newfield, NJ 08344, 800-662-0973, 3 well 11 mm HTC Super Cured Autoclavable white slides part number 10-31 S-white. Order 3 gross.

11.6.3.22. Coverslips — 22 X 50 mm

11.6.3.23. Nalgene 550 Platinum- Cured Silicone tubing, ID 9.5 mm, OD 15.87 mm, Fisher Catalog no 14-176-332L.

11.6.3.24. Plasto-O-Matic 1/2 gallon flow control valves. Obtained from sole supplier, Eagle Supply and Plastics, PO Box 1196, 500 E. Winnebago St., Appleton WI 54912, 920-739-8841.

11.6.3.25. Sample Shipping Containers, Foam Packers part number:489UPS (CP), Corrugated Cartons for packers is part no: 1012-5KD (CP), CP is a case of 6 singles, Polyfoam Packers Corp, 888-765-9362.

Figure 11.4 — Temperature Tolerances

Temperatures of this equipment are checked and recorded on a daily basis unless otherwise specified. The temperatures are checked with NIST traceable thermometers or thermometers that are checked against NIST thermometers annually. If the temperature of any of the equipment listed below exceeds its allowable range, the supervisor is to be notified so that corrective action can be taken. Arrangements will be made for a service visit from the appropriate source (e.g., manufacturer, UW service dept., Dept. of Admin.) and the action will be documented in the appropriate logbook. Any affected samples will be reanalyzed if possible, or results will be qualified noting the equipment problem.

Equipment	Location (Room #)	Required Temperature	Allowable Range
Pesticide Standards Freezer	216D	-15°C	≤-15°C
Volatile Standards Freezer	216D	-15°C	≤-15°C
† Sediment Drying Oven	217	105°C	102 - 108°C
Silica Gel Drying Oven	217	130°C	≥130°C
Pesticide Standards Freezer	217	-15°C	≤-15°C
Pesticide Standards Refrigerator	217	4°C	1 - 4°C
Na_2SO_4 Drying Oven	217	130°C	≥130°C
Tissue Freezer	217E	-15°C	≤-15°C
Waste Enforcement Locker	217B	4°C	1-4°C
Regular Sample Locker	217C	4°C	1 - 4°C
Vial Oven	219	105°C	102 - 108°C
524.2 VOC Sample Locker	219D	4°C	1 - 4°C
8260 VOC Sample Locker	219C	4°C	1- 4°C
GC/MS Standards Freezer	220	-15°C	≤-15°C
Radiochemistry Cooler	201	4°C	1 - 8 °C
Radiochemistry Oven	201	N/A	100 - 120 °C
Ovens (for Solids analysis)	119	104°C	103-105°C
Oven (used for TDS)	119	180°C	178-182°C
Ovens (used for Metals) Checked as used.	119	103°C	101-105°C
Coldroom Walk-in	119C	4°C	1-4°C
BOD incubators	119	20°C	19-21°C
Muffle Furnace (used for Solids) Checked monthly.	119	550°C	500-600°C
Hot blocks (used for digestions) Checked as used.	118	95°C	90-100°C
Water bath (used for mercury) Checked as used.	118	95°C	90-100°C

Figure 11.5 — Biomonitoring & Water Microbiology Floor Plan

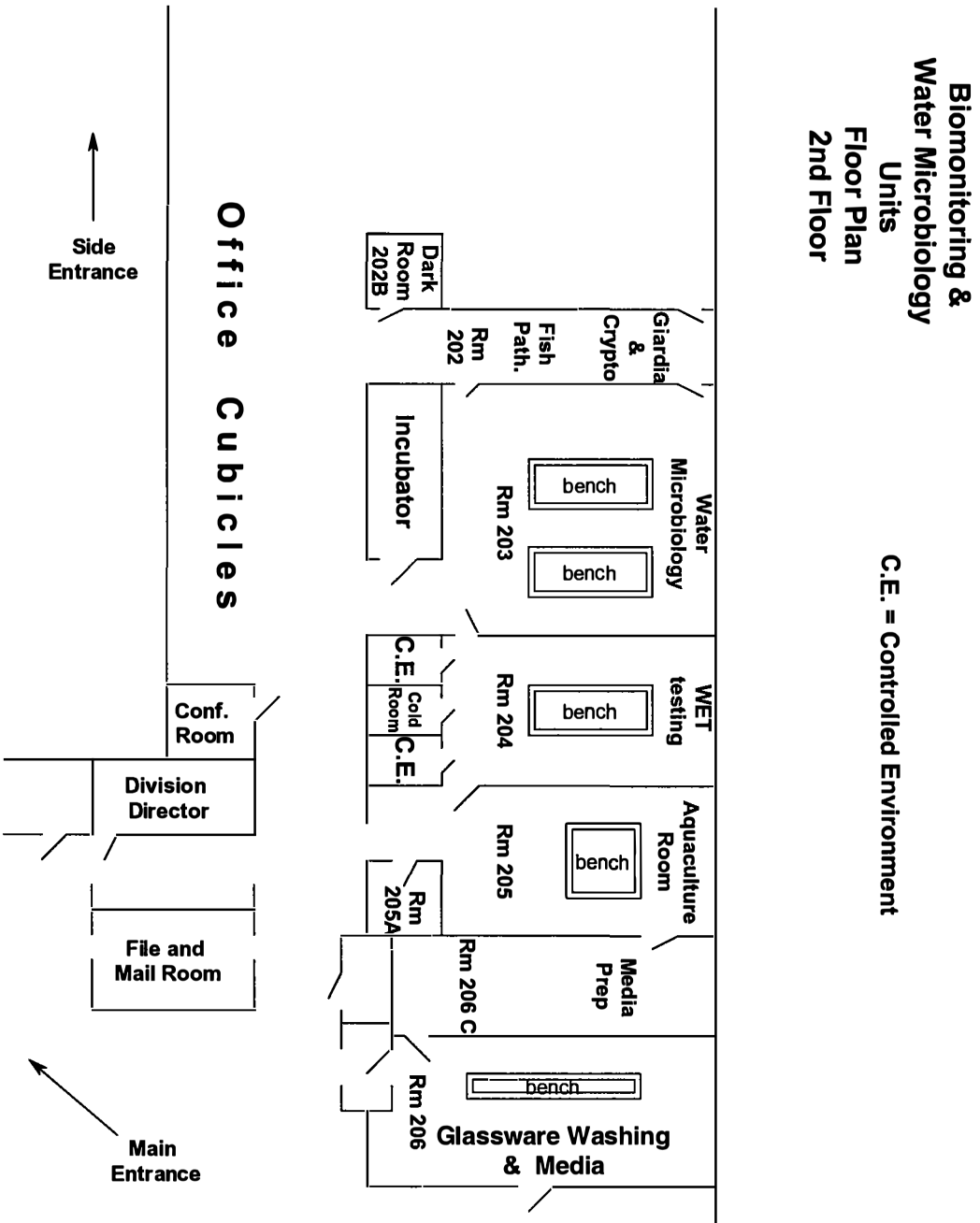


Figure 11.6 — Inorganic Chemistry Floor Plan

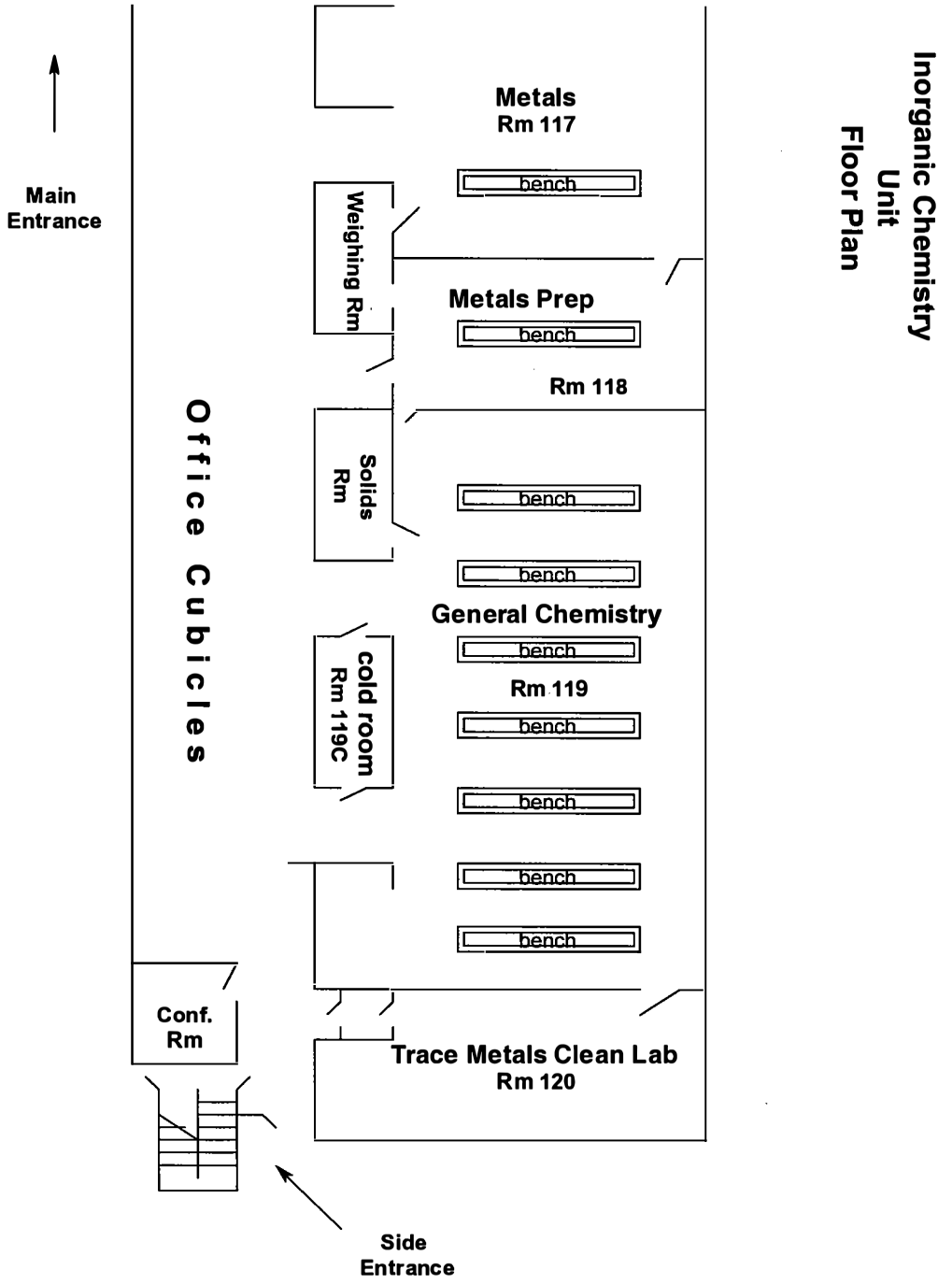


Figure 11.7 — Organic Chemistry Floor Plan

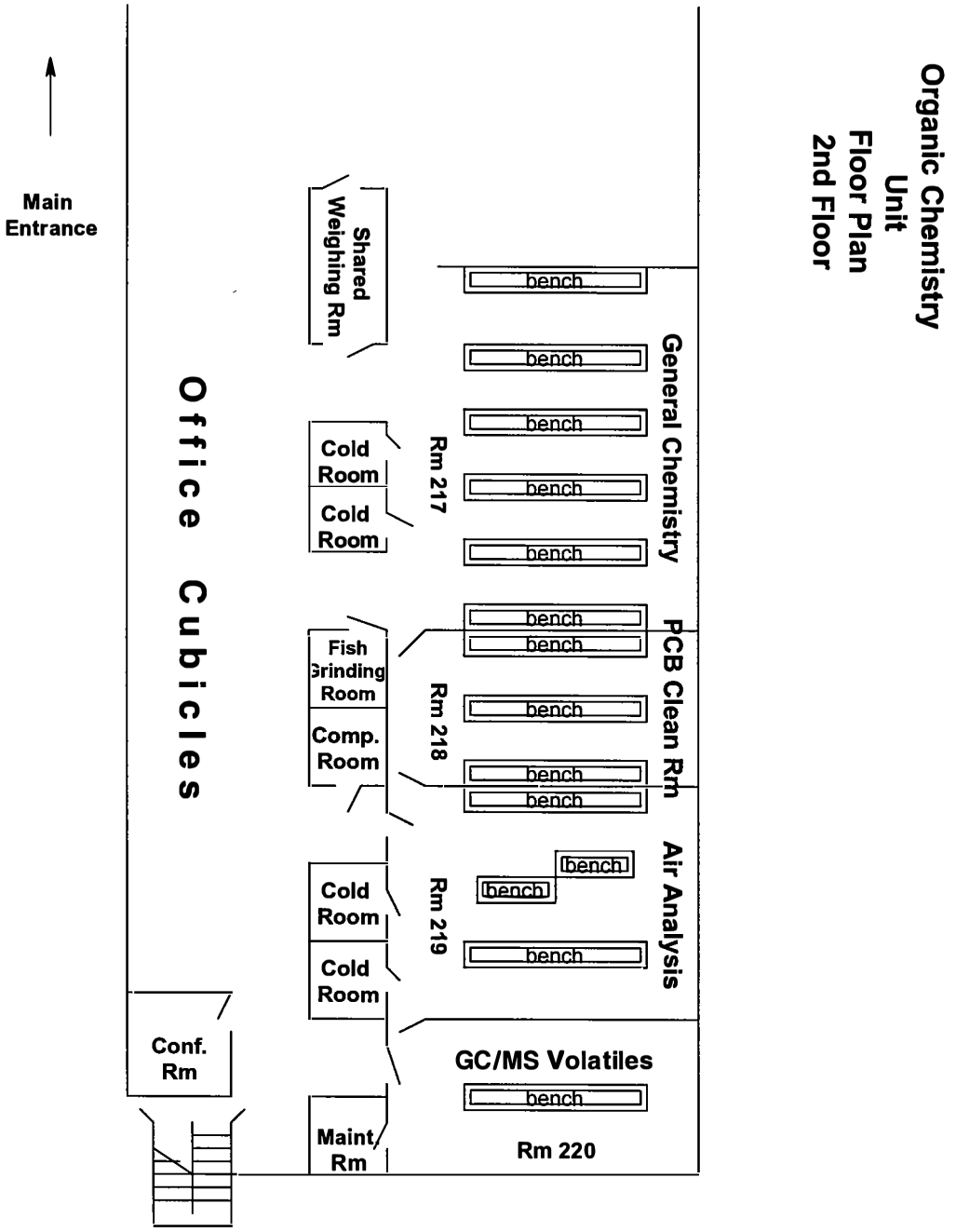
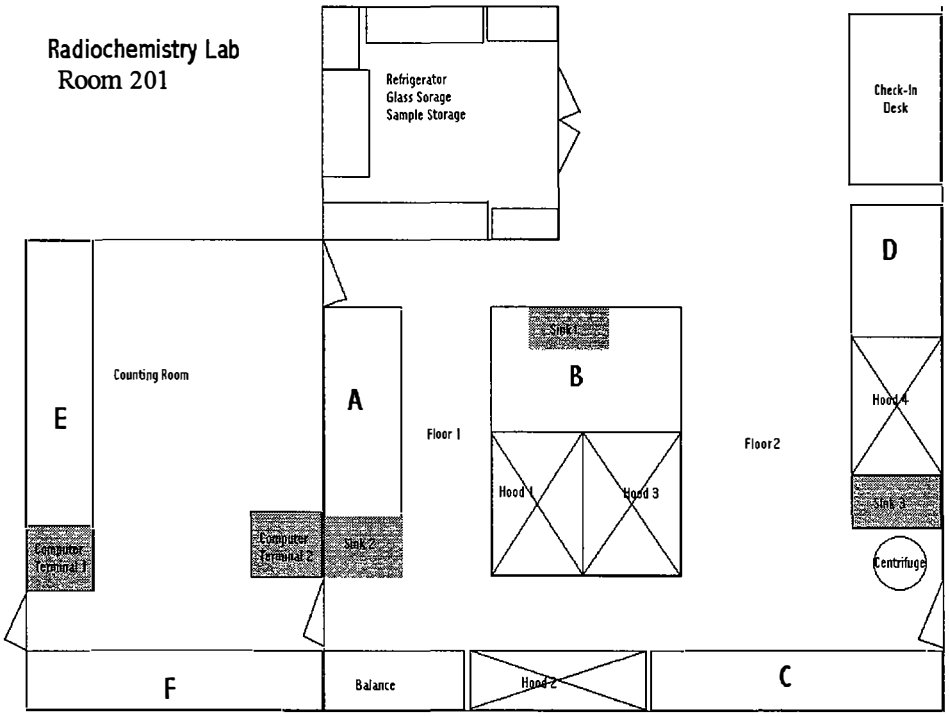


Figure 11.8 — Radiochemistry Floor Plan



12. General Quality Control Procedures

All analytical data are produced to effect a decision. That decision could have legal, regulatory, monitoring, or production consequences. It is the intention of our quality assurance program to guarantee that those decisions are based on the highest quality data possible. To ensure this outcome, routine daily quality control practices are necessary to continually monitor all processes in the laboratory.

12.1. General

Management System Review

The quality system spelled out in this manual will be reviewed by the Division Director (i.e., the NELAC Technical Director) on an annual basis. The review will consist of a report prepared by the Quality Assurance Officers reflecting the effectiveness of the system. It will include proficiency testing results, problem areas and general impressions regarding the laboratory's ability to meet its quality objectives.

12.2. Biomonitoring

12.2.1. Methodology

The methods used in this department are fully documented in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual". Each method includes a section on references which includes documents such as the Federal Register, EPA Methods Manuals, the AOAC Methods Manual or Standard Methods 20th edition (1998). Detailed, specific quality assurance procedures are also included with each method since quality control is such an integral part of all procedures.

12.2.2. Test organisms

12.2.2.1. Selenastrum capricornutum (Algae)

Weekly, algae cultures are examined microscopically for density and morphology. Every third month, a new slant of algae is used to ensure health and vigor of the culture. New slants will be examined to ensure correct cellular morphology. Cultures containing contamination or of the wrong cellular morphology are discarded.

12.2.2.2. *Pimephales promelas* (fathead minnows)

The following conditions are monitored and changes are made when limits are exceeded.

Parameter	Acceptable range	Frequency
Temperature	25-27°C	2x daily
Aeration		visual daily
Unionized ammonia	<0.01 mg/L	weekly
Light- 16 hours	323-1076 lux	visually daily
Egg production	3000-12000/week	daily
Circulation	77 exchanges/day	Visually daily
Heterotrophic plate counts	<50/ml	quarterly

Egg production is graphed weekly and displayed in the lab. Any change in food, equipment or parameters is noted on the egg production graph. Results and responses to problems are recorded in files and log books.

12.2.2.3. *Ceriodaphnia dubia*

Survival data and young production data from all cultures are recorded. Mean young production and adult survival per week is determined and graphed. If young production falls below 20 neonates per female or adult mortality is greater than 20%, then the neonates should not be used for testing.

12.2.3. Laboratory water used for culturing and test dilution water

Laboratory control waters (Synthetic Hard Water, dechlorinated tap water, and Lake Kegonsa water) are tested for hardness, alkalinity, conductivity, pH and DO with every new batch. Results and corrective actions are recorded in culture water logbooks.

Water used for culturing, food preparation and test dilution should be analyzed for toxic metals and organics whenever difficulty is encountered in meeting minimum acceptability criteria for control survival, reproduction or growth.

12.2.4. Food quality

The quality and suitability of the food used for culturing and test feeding will be reflected in the survival, growth and reproduction of the test organisms. Silvercup trout chow is ordered on a quarterly basis to ensure freshness. Yeast, fish food and cerophyll (YFC) have an expiration date of two weeks. If a batch of food is suspected to be defective, the performance of organisms fed with the new food can be compared with the performance of organisms fed with a food of known quality in side-by-side tests. If the food is used for culturing, its suitability can be determined by comparing survival and reproduction of the organisms to that of the organisms fed an earlier batch which will determine the

effect of food quality on growth or reproduction of each of the relevant test species in the culture.

12.2.5. Reference toxicity testing

To ensure the health and sensitivity of the test organisms, reference toxicity testing is performed monthly. Sodium chloride at various concentrations appropriate to the organism is used as a reference toxicant for the following test systems: *P. promelas*- acute and chronic effluent, *C. dubia*- acute and chronic effluent, and *S. capricornutum* - chronic effluent. Reference tests are performed monthly. Results are plotted on graph paper. The system is out of control if any test result is outside the confidence limit of $\pm$ 2 standard deviations. Tests that are not within control limits are redone. Responses to out of control situations are recorded in the permanent logbook.

12.2.6. Proficiency testing

Proficiency testing (Discharge Monitoring Report) samples are done annually. The DMR-QA study is supplied by and results reported to the USEPA. DMR testing is performed for *P. promelas* and *C. dubia* acute and chronic tests. A failed DMR will be rerun after 30 days. DMR samples are currently unavailable.

PT samples known as DMR samples are done once yearly. Environmental Resource Associates (ERA) supplies the PT samples and results are reported to them.

12.2.7. Internal audits

An annual internal audit is conducted that reviews the accuracy of test methods conducted, the completion of labslips and paperwork, the accuracy reporting of test results and to ensure that QC items have been conducted. Deficiencies will be noted in the audit report along with corrective action and a timeline for the corrective action. Annually, SOPs and logbooks are reviewed for completeness and accuracy. Any revision or changes needed are done and noted in the permanent lab logbook. Performance reviews will be given to each analyst to confirm their ability to perform culturing duties and testing duties.

12.2.8. Quality control documentation and data gathering

Quality control documentation is recorded in bound laboratory logbooks or electronically. Some QA data is gathered and recorded in a centralized electronic location on a weekly basis.

12.2.9. Calibration procedures

All instruments are calibrated prior to use. Calibration measurements are recorded in bound logbooks or electronically. Pipettes are checked for accuracy on a quarterly basis. Thermometers and probes are NIST traceable. Specific calibration procedures for specific instruments can be found in the SOP manual.

12.2.10. Procedures for taking corrective action

Procedures for taking corrective action are outlined in the SOP manual for each specific method. All corrective actions are noted either on run charts or in the permanent laboratory logbook. Corrective action is generally performed by the analyst at the time that the problem occurs. Long term corrective actions are decided upon and monitored by the Biomonitoring team.

12.2.11. Departures from documented policies and procedures

Departures from documented policies and procedures will be discussed by the Biomonitoring team. The departure will be noted in the permanent lab log.

12.2.12. Client complaints and concerns

Complaints and concerns are dealt with immediately by the person receiving the complaint. When necessary the analyst directly involved with the test or procedure of concern will handle the customer complaint. Every effort will be made to satisfy the customer.

12.3. Inorganic and Organic Chemistry

12.3.1. Methodology

Analytical methods used in the Inorganic and Organic Chemistry Department are documented in each department's Analytical Methods Manual. These method SOPs are written in enough detail so that an analyst can clearly understand and follow the procedures. In addition each method SOP includes the specific quality control criteria and references the applicable authoritative source. In the case of a non-regulated "in-house" method, references to scientific papers used, or to authoritative methods that have been modified, will be included. A list of documents from which methods have been derived is included in Chapter 15.

All methods used for regulatory purposes, including those for the National Pollutant Discharge Elimination System (NPDES) program, the Resource Conservation and Recovery Act (RCRA), Safe Drinking Water Act (SDWA), and other programs regulated by the Wisconsin Department of Natural Resources Laboratory Certification Program, specifically require the use of approved methodology. Analytical methods for these programs must be recognized as approved in the appropriate Federal publication (Federal Register) and the Wisconsin Administrative Code. However, approved methods may be modified or replaced with improved techniques upon receiving approval from the appropriate regulatory body (e.g., WDNR, the USEPA regional administrator, or the current NELAP accrediting authority). This may be accomplished by submitting a detailed description of the method, accompanied by comparability data showing that the modified method is equivalent to, or better than, the approved method.

12.3.2. Quality Control Documentation and Data Gathering

All QC data that is tracked will be recorded with a LIMS program titled qawrksht. The program is interactive and designed to collect QC results and

their associated biographical data in a database format. qawrksht allows for adding, deleting and modifying QC results, and identifies each result with a unique code (QR key).

For each set of QC results the analyst will prepare a QA worksheet which will contain all biographical information and all results. In addition to the electronic input of the QC data, a hard copy of the worksheet will be retained with the appropriate sample results.

While qawrksht is the most used QA related program it is not the only one. Several other software tools including (but not limited to) those listed below are used to track, calculate and report QC data and statistics. The mechanics of these programs are beyond the scope of this document, however each program has detailed instructions provided in the LIMS online manual. They can be accessed either by typing explain <program name> at any LIMS prompt or by choosing General Information and Read Manual pages from the Main Menu and then entering the manual subject or program name.

12.3.3. LIMS QA Programs

- qawrksht
- qalimits
- ql
- chromres
- chromqa
- mt
- biweight
- printqa
- qr
- uwdsm
- find
- qa
- modql

12.3.4. Calibration Procedures

12.3.4.1. Inorganic Chemistry Department

The construction of calibration curves may depend on specific method criteria. In general, however, curves are constructed with a reagent blank and at least three standards graduated over the concentration range of interest. Curves are established each day before analysis begins. The correlation coefficient (r) of the curve must be within the control limits specified in the method. If the "r" value is exceeded, the source of the

problem will be identified, corrected and the instrument will be re-calibrated before proceeding with analysis.

When stable control samples are available, calibration curves will be immediately verified by analyzing an initial calibration verification (ICV) standard. The ICV is prepared from a source different from the calibration standards and must be within the tolerance specified in the method. If it is not then corrective action is taken and the system is re-calibrated. In addition to the ICV, usually a continuing calibration verification standard (CCV) and a calibration blank are analyzed at least every 10 samples. If the CCV is out of control a new curve will be generated and all samples run after the last acceptable CCV will be reanalyzed.

12.3.4.2. Organic Chemistry Department

Method requirements for calibration curves vary widely in the Organic Chemistry Department, and individual requirements are always spelled out in the method SOPs. In general, however, the regulated methods call for similar constrictions. Calibration curves should include at least three and generally five or more points spread evenly across the expected range. A system blank must not be included in the curve, nor should the curve be forced through the origin.

Correlation coefficients should be greater than 0.995 for most analysis, though values of 0.990 are acceptable where greater variability is expected (e.g., VOC methods). Most methods require calibration check standards to be run before, during and after analysis of samples. If the acceptance criteria for the correlation coefficient or the calibration check is exceeded the problem will be identified and corrective action will be taken. The samples will be rerun or the results may be qualified if rerunning is not possible.

For other calibration verification criteria please see the specific method SOP.

12.3.5. Procedures for Taking Corrective Action

Procedures for taking corrective action in the laboratory are for the most part, instrument specific. When such action is indicated (e.g., when calibration criteria are not met), it may be performed by the analyst involved or may require an outside service call. When any such action is taken it will be noted and described in the appropriate instrument logbook. If outside service is required the Department Supervisor will be consulted.

As noted in 12.3.6 below, the laboratory participates in the NELAP Performance Evaluation Studies (the old WP and WS programs of the USEPA). Semiannually, unknown reference samples are analyzed in conjunction with the NPDES, and SDWA laboratory certification programs. Successful completion of these reference samples is required to maintain laboratory certification under SDWA, the WDNR Laboratory Certification Program (Wisconsin

Administrative Code NR 149), and the National Environmental Laboratory Accreditation Program.

If analysis results of any these samples fall outside the acceptable range, the analyst will complete an evaluation form (Figure 12.1). The form is used to identify the source of the error and describes the corrective action taken. The form is reviewed by the senior analyst in the section, the QA officer, and the Department Supervisor. A follow-up sample will be analyzed as soon as possible, and must be passed or a loss of certification could result.

12.3.6. Inter- and Intra-Laboratory Quality Control Samples

The laboratory participates in a number of inter- and intra-laboratory quality assurance programs including:

12.3.6.1. NELAP Performance Evaluation Studies (WP, WS, SOIL, and other matrices as they become available).

12.3.6.2. Project specific round robin or performance studies (e.g., National Water Research Institute interlab study, Ontario Canada).

12.3.6.3. Field split sample programs, which include the analysis of blind field duplicates and samples split with other public and some private laboratories.

12.3.6.4. Control samples provided by the client. A real world sample of known concentration.

12.3.6.5. Programs of the U.S. Geological Survey Standard Water Reference Sample Program.

12.3.6.6. USEPA Long Range Transport of Atmospheric Pollutants (LRTAP) program.

12.3.6.7. Internal reference samples (blinds) purchased from an outside vendor. These samples are analyzed every quarter and will include all accredited methods for which a reference sample can be found. The acceptable ranges will be known to the QA officer, but not to the analyst. If any result does not fall within the acceptance limits, corrective action will need to be taken.

12.3.7. Internal Quality Assurance Audits

Quality assurance audits will be performed on each analytical method or chemistry section on an annual basis. These audits will be performed by the QA Officers and while they will be more informal than an outside review, will nonetheless be complete and rigorous.

The intention is to audit the analytical method for conformity to the pertinent regulations, point out any inconsistencies, and recommend corrective action. In addition, these audits will serve as an opportunity to audit the analysis process and to enlist the analyst's help in making any possible improvements.

Each audit may consist of a review of the method, a short "on-site" with the analyst and the generation of a report (see ESS ORG QA 0003, and ESS INO QA 108). The report will be given to the analyst and the Department Supervisor, along with recommended corrective action (if there is any) and a time frame for completing the necessary changes. Once the changes are made the report is finalized and kept on file with the QA Officer.

12.3.8. Departures from Documented Policies and Procedures

Invariably there will be exceptions to the policies and procedures documented here. Such deviations will primarily consist of two different types: 1) deviations from standard operating procedures during routine analysis, and 2) unforeseen occurrences for which the lab has no policies or methods and no time to formally develop them (e.g., an environmental emergency).

In case #1 deviations may be made because of instrument problems, sampling errors, or program requests. For instrument and sampling problems the departure from the method SOP will be noted on the analysis form (worklist, lab sheet, etc.). For program requests the deviation would be noted in the analysis contract. In the case of any deviation that specifically involves generated results (i.e., a QC exceedance) the result will be appropriately qualified (See Chapter 20).

In case #2 (a spill for example) every effort will be made to follow approved and documented procedures; however sometimes such procedures do not exist or the requesting agency is unsure what parameters are likely to be found. In such cases the laboratory's senior analysts and supervisors will consult with the requesting agency to arrive at a course of action that provides the most timely and accurate results. In many cases the testing performed will be qualitative in nature to give the proper authorities a better assessment of the problem.

12.4. Radiochemistry

12.4.1. Methodology

Analytical methods used in the Radiochemistry Department are documented in the Department's Analytical Methods Manual. These method SOPs are written in enough detail so that an analyst can clearly understand and follow the procedures. In addition each method SOP includes the specific quality control criteria and references the applicable authoritative source. In the case of a non-regulated "in-house" method, references to scientific papers used, or to authoritative methods that have been modified, will be included. A list of documents from which methods have been derived is included in Chapter 15.

All methods used for regulatory purposes, including Safe Drinking Water Act (SDWA), and other programs regulated by the Wisconsin Department of Natural Resources Laboratory Certification Program, specifically require the use of approved methodology. Analytical methods for these programs must be recognized as approved in the appropriate Federal publication (Federal Register) and the Wisconsin Administrative Code. However, approved methods may be

modified or replaced with improved techniques upon receiving approval from the WDNR, the USEPA regional administrator or the current NELAC accrediting authority. This may be accomplished by submitting a detailed description of the method, accompanied by comparability data showing that the modified method is equivalent to, or better than, the approved method.

12.4.2. Quality Control Documentation and Data Gathering

For each sample analyzed, a unique QC number is associated with any measures of the precision, accuracy, and blank recoveries for that analysis. These QC numbers are entered into the database record with the sample. In a related database the actual values for those accuracy, precision, and blank samples are stored along with the current limits associated with specific analyses. A QC record can be produced for any sample that lists the value determined for the accuracy, precision, and blank sample and the acceptance limits.

12.4.3. Analytical Procedures

A batch is defined as any set of samples (up to twenty) which are started on the same (24hr) day, by the same analyst.

12.4.3.1. Accuracy (Laboratory Control Sample and Matrix Spike)

See 13.3.3 for the equations and procedures for setting control limits.

One matrix spiked (MTX) sample and one laboratory control sample (STD) are analyzed in each batch of samples. A modified Shewart accuracy chart is used to interpret these results and set the acceptance limits. This chart displays the mean, a warning control limit, and an upper control limit. If any of the control samples are outside of the upper control limit, the samples are re-analyzed. Samples may be reported to the client with a qualification if there is insufficient sample to repeat the analysis.

12.4.3.2. Precision

As a check on the precision of analytical procedures one sample per batch is analyzed in duplicate. A modified Shewart precision chart is used to check duplicate results and set the acceptance limits. For procedures that routinely analyze samples with no detectable activity, and therefore, do not produce useful data to prepare precision charts, the results of a second laboratory control sample (LCS) is analyzed for precision monitoring.

12.4.3.3. Blank

One blank sample is run with every batch to check the radioactive purity of all the reagents used throughout the procedure. The standard deviation is calculated and the acceptance limit is set at three sigma.

12.4.3.4. Reporting

Use the following criteria to determine whether or not the sample results may be reported:

If all of the QC data is within the upper control limits, report all results.

If the precision, blank, matrix, or the accuracy results is out of the upper control limits, recount the suspect QC sample. If after recounting the QC sample, it is now within acceptable limits, report all results. If the QC sample remains out of control, repeat all of the samples unless there is insufficient sample, which are then reported with a qualification.

If the blank sample is beyond the control limits, investigate reagents and labware for the source of contamination.

The above criteria are based on the assumption that the instrumentation involved was properly calibrated and has met the QC requirements set in the instrument's SOP. If an instrument problem is suspected, see the trouble shooting section for that instrument.

All problems associated with instruments are documented in an Access database. This file contains the date of the problem encountered, a description of the problem, and steps taken to remedy the situation. All staff have access to this file and can make entries. Additionally, instrument logbooks may contain some of this same information.

12.4.4. External Quality Control

12.4.4.1. Interlaboratory Comparisons

External quality control samples are received from ERA, DOE, and Bowser-Morner, Inc. periodically. Those from Bowser-Morner are done monthly. DOE samples are shipped in March and September.

DOE Standard	Study Shipping Months	Analytes
Air Filter Gamma	Mar & Sep	Mn54, Co60, Cs137
Air Filter Alpha Beta	Mar & Sep	Gross alpha & beta
Water	Mar & Sep	H3, Co60, Cs134, Sr90, U total
Vegetation Gamma	Mar & Sep	K40, Co60, Cs137
Soil Gamma	Mar & Sep	K40, Cs137,Pb212, Bi212, Bi214, Pb214 Ac228
Water Alpha Beta	Mar & Sep	Gross alpha & beta

Those from ERA follow the schedule below

ERA Standard	Study Shipping Months	Analytes
Sr89/90	Jan & July	$Sr_{89/90}$
alpha & beta GroSS	Jan, July & Oct	Th_{230} & Cs_{137}
Iodine-131	Feb & Oct	I_{131}
NaturalS	Feb, June & Sept	Ra_{226}, Ra_{228}, $U_{(nat)}$

ERA Standard	Study Shipping Months	Analytes
TritiuM	March & Aug	H_3
alpha, beta, & gamma MiXeD	April & Oct	Ra_{226}, Ra_{228}, $U_{(nat)}$, $Sr_{89/90}$, Co_{60} & $Cs_{134/137}$
gamma EmitterS	June & Nov	Ba_{133}, Co_{60}, $Cs_{134/137}$ & Zn_{65}

Cross check samples are analyzed at least in triplicate. If the determined values are not precise and accurate based on the final report, then the procedure and the instrument should be evaluated.

Generally, it will not be possible to re-analyze the cross check due to the short halflives of some of the nuclides and because the sample is often used up completely. It is likely that several runs of routine samples have been analyzed between the time the cross check results were reported and the time the final report was issued. If a major problem with the procedure existed it should have been detected by the criteria set forth in the Internal Quality Control section of this procedure. If sufficient cross material is available and the nuclides haven't decayed away reanalyze the sample. If the results still do not fall within the acceptance limits, evaluation of the calculations, instrument efficiency, and reagents will be made. It may also be helpful to request another sample from the supplier and analyze that too.

12.4.5. Internal Quality Assurance

The analytical methods are reviewed and updated yearly by the department supervisor and senior analysts. Each analyst responsible for a given method, is required to review the SOPs with instructions to make sure that the method is being performed as specified in the document.

12.5. Water Microbiology

12.5.1. Methodology

Analytical methods used in the Water Microbiology Department are documented in the Department's Analytical Methods Manual. These method SOPs are written in enough detail so that an analyst can clearly understand and follow the procedures. In addition each method SOP includes the specific quality control criteria and references the applicable authoritative source. In the case of a non-regulated "in-house" method, references to scientific papers used, or to authoritative methods that have been modified, will be included. A list of documents from which methods have been derived is included in Chapter 15.

All methods used for regulatory purposes require the use of approved methodology. Analytical methods for these programs must be recognized as approved in the appropriate Federal publication (Federal Register) and the

Wisconsin Administrative Code. However, approved methods may be modified or replaced with improved techniques upon receiving approval from the WDNR, the USEPA regional administrator or the current NELAC accrediting authority. This may be accomplished by submitting a detailed description of the method, accompanied by comparability data showing that the modified method is equivalent to, or better than, the approved method.

12.5.2. Quality Documentation for Water Microbiology

All QC data that affects the testing is logged into the QC logbooks. Positive and negative performance checks of media and procedures used are recorded. If the correct reactions are not obtained, the media and reagents will not be used. If any matrix spikes or on going precision testing fails established QC, new samples will be requested and new matrix spikes and on going precision testing will be performed after making any corrections. Any deviation from the SOP is logged into the logbook along with what corrective action has been taken.

12.5.3. External Quality Control

The Water Microbiology Department purchases performance samples for every certified method where PE samples are available.

Every analyst in the department does 5 blind PE samples from the Wisconsin Proficiency Testing Program every year. If the analysis results of these samples fall outside the acceptable range, the department supervisor will review the procedure with the analyst. A follow-up sample will be analyzed as soon as possible.

12.5.4. Quality Assurance Audits

Quality assurance audits will be performed on each analytical method on an annual basis. These audits will be performed by the department supervisor or a microbiologist (auditor) from the department.

The intention is to audit the analytical method for conformity to the pertinent regulations, point out any inconsistencies and recommend corrective action. In addition these audits will serve as an opportunity to audit the analysis process and to enlist the analysts help in making any possible improvements.

The auditor will use the checklist from EPA 815-B-97-001 and the internal checklist from the water microbiology department. The auditor will make a report with corrective actions. The Department will make all the corrections.

12.5.5. Departures from Documented Procedures

Invariably there will be exceptions to the policies and procedures documented here. Such deviations may be made because of instrument problems, sampling errors, or program requests. For instrument and sampling problems the departure from the method SOP will be noted on the analysis form. For program requests the deviation would be noted in the analysis contract. In the case of any deviation that specifically involves generated results (i.e., a QC exceedance) the result will be appropriately qualified (See Chapter 18).

Figure 12.1 — Corrective Action Form

Wisconsin State Laboratory of Hygiene

Performance Evaluation Samples - Unacceptable Data

Should "Not Acceptable" data result from analysis of Performance Evaluation Samples, the analyst is to complete the following form. Upon completion, a copy is to be sent to the Department Supervisor, Laboratory Manager, and Laboratory Q.A. Officer.

Parameter:	Sample:
Analyst:	Method:
Report Value:	Date of Analysis:
Acceptable Limit:	True Value:
	Warning Limits

		YES	NO
1.	Transcription Error	____	____
2.	Transposition Error	____	____
3.	Decimal Error	____	____
4.	Department Error	____	____
5.	Calculation Error	____	____
6.	System Not in QC	____	____
7.	Dilution Error	____	____
8.	Contamination	____	____
9.	Standards Error	____	____
10.	Instruction Error	____	____
11.	External Error	____	____
12.	Other	____	____

Comments:

Figure 12.2—Occurrence Management Report Form

o Henry Mall o Ag Drive

Person Initiating Report:_____ **Date:**_____ **Department:**_____

Code: __ __ __ __

Brief Description of Occurrence: (o attachment)

Occurrence Classification: (more than one may apply)

Analytical	o HIPAA	o Customer Feedback
o Pre-testing	o Medicare	o Communication
o Testing	o WSLH Resource	o Other_____
o Post-testing		_____
o QC/PT Documentation		

Immediate Action Taken: (include notification of any affected parties)

Report Submitted to:_____

Report Submitted by:_____ _____
Signature Pr nt name

_____ _____
Unit and Position Date

Form will be only for internal department use: o yes **Department Reference number:**_____

Administrative use only

OM reference #_____ SM resolution required: o yes o no Date reviewed:_____

(If yes, see resolution report) Reviewer:_____

HIPAA Reviewed:_____ _____
Date Initials

13. Quality Control Limit Procedures

13.1. Biomonitoring

Procedure for calculating organism control limits:

Monthly reference toxicant tests with sodium chloride are used to determine organism performance and acceptability for use in toxicity testing. Acute test survival data are input into one of two software programs for calculating an LC50 (concentration lethal to 50% of a population). When there is partial mortality in just one treatment, the software program using the Spearman-Karber analysis is used to calculate the LC50. If more than one treatment has partial mortality, software using Probit analysis is used to calculate the LC50. Chronic reference test data are input into USEPA's Inhibition Concentration Program (IC_p) to calculate an inhibition concentration for 25% of a population (IC25).

Acute and chronic endpoints are entered on control charts for each species in an Excel spreadsheet. New data replace the oldest entry in each graph, thereby maintaining the last 20 monthly data points for each type of test and each species. Each graph plots mean, and plus and minus two standard deviation lines. Data points outside of the two standard deviation lines are considered "out of control" and a quality assurance violation. Corrective measures for culture conditions are discussed and implemented, if necessary. Those reference tests outside of control limits are repeated within 30 days. Effluent testing is suspended until reference test data are within control limits

13.2. Inorganic and Organic Chemistry

NOTE: Most of the QC limits for the Inorganic Chemistry Department are explicit limits. They are based either on historical limits calculated using the biweight statistical analysis function (see LIMS biweight), or on method requirements. The biweight program is used to evaluate the limits annually.

Quality control (QC) limits define the precision and accuracy of all reported data. QC audits insure that the data being reported are valid within the defined limits. Our laboratory uses standard deviation (N-1 weighting) to establish precision and accuracy control limits for each parameter. Alternatively, required limits may be set in accordance with the determinative method. When insufficient data are available to define control limits using statistical techniques, reasonable fixed limits (based upon authoritative sources and analyst experience) will be assigned.

Traditional statistical control limits are established at three sigma (three standard deviations from the mean) and constitute 99% of all valid data. Data not within three sigma of the mean indicate the need for further analysis and/or method examination. Note that these data do not necessarily indicate analyses are out of control, as the remaining 1% of valid data will be expected in this area. However, any sample data associated with QC result outside the control limit is not reported, or it is qualified with an appropriate QC flag.

In cases where a single parameter or a group of related parameters (for example 2,4-D and 2,4,5-TP) are out of the three sigma range, results for those parameters in the

matrix in question will not be considered acceptable until accuracy and precision limits are met or re-established for that batch or group. If several non-related parameters are analyzed in the same matrix, data acceptance may be withheld only for those parameters displaying results out of the three-sigma range. Samples may be reanalyzed for all parameters or only for the parameters in question. Any additional actions are determined by the QC auditor in conjunction with the analyst and after consulting the determinative method.

13.2.1. Precision Control Limits

Precision is defined as the degree of mutual agreement among individual measurements made under defined conditions, (US EPA, 1979). A normalized method for specifying precision is the relative percent difference (P) expressed as follows:

$$P = 100 \frac{|A-B|}{((A+B)/2)}$$

where:

A,B	= The two analysis results (duplicates)
|A-B|	= The absolute difference in the results
((A+B)/2)	= The average of the results

Precision limits are based upon two separate calculation types. For many parameters precision may be concentration dependent. In these instances, control limits are established for a series of sequential concentration ranges. It may be calculated as an absolute difference or, as a relative percent difference. These limits are either updated yearly, when the determinative method requires, or when the test conditions change.

13.2.1.1. Absolute Difference

Absolute difference is generally used for very low concentration ranges where, because of significant figure issues, slight numerical differences would calculate out to be increasingly large percentages. For the most part absolute difference is used exclusively in the Inorganic Department.

13.2.1.2. Relative Percent Difference

The Upper Control Limit (UCL) is the maximum upper acceptable limit for the percent difference between duplicate analyses (R) analyzed within the specified concentration range. The UCL is calculated according to the equation:

UCL = 3s+R

where:

R = The average difference between duplicate analyses for a given range.

3s = Three times the standard deviation using an n-1 weighting.

If a precision result exceeds the UCL two possible paths are followed. The affected samples along with another set of duplicates will be reanalyzed. If subsequent precision results exceed the UCL, the system is considered out-of-control and the data invalid. The system will then be stopped until the problem is identified and resolved. Data collected during the out-of-control situation will be repeated or discarded. If it is not possible or analytically feasible to reanalyze the affected data (due to lack of sample, holding times, etc.), the collected data will be qualified with an appropriate QC flag. This flag is associated with each specific sample result in the affected batch, and is provided to the client. (See Appendix D for possible QC flags.)

13.2.2. Accuracy Control Limits

Accuracy is the difference between an average value and the true value when the latter is known or assumed (USEPA, 1979). Accuracy control limits are based upon the mean and standard deviation of the percent recoveries of natural or synthetic samples spiked with standard solutions and analyzed using the same methodology applied to real samples.

A spike involves adding a known concentration of a particular standard to a real sample matrix to evaluate the accuracy of the test procedure. Such accuracy measurements can also include standard matrices (i.e., solvent), or a laboratory matrix (i.e., sea sand) which may be used for calibration check verifications, laboratory control samples, outside source check standards, etc. The percent recovery (P) is calculated using the formula:

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

where:

A = The observed concentration of the spiked sample

B = The background concentration of the sample

C = The known concentration of the spike

The Upper Control Limit (UCL) is the maximum upper acceptable percent recovery (P) for spiked samples. The Lower Control Limit (LCL) is the maximum lower acceptable percent recovery (P) for a spiked sample.

The UCL and LCL are calculated using the following equations:

$$UCL = P + 3Sp$$

$$LCL = P - (3Sp)$$

where:

P = The mean percent recovery for a series of spiked samples analyzed over a given period of time.

Sp = The standard deviation of the percent recovery for a series of spiked samples analyzed over a given period.

Percent recovery values are checked against upper and lower control limits, and actions taken similar to those described above for percent difference.

13.2.3. Matrices

There are two types of matrix designations used in the laboratory: field matrix, and QC matrix. Field matrix refers to the designation given to the sample by the client (usually the WDNR), while QC matrix refers to the matrix designation assigned for quality control purposes. A number of similar field matrices may be bundled together under one QC matrix for statistical purposes. For example, the field matrices private well, sample tap, and municipal well are all considered drinking water for QC purposes.

In addition to the real world matrices there are several "non-standard" designations used for QC purposes. Those include laboratory matrix, standard matrix, and reagent matrix. These designations are used when a "real" matrix code would be misleading (e.g., using Ottawa sand as a simulated matrix for soil samples).

13.2.4. Limit Identification

Each QC limit is uniquely identified with a "QL key". The key is a code number consisting of the LIMS test code, prompt number (which corresponds to a specific parameter), the QC type (spike, duplicate, check, performance, or blank), the two-letter QC matrix code, and a range designation.

13.2.5. Limit Creation and Modification

Limits are modified, created and deleted using either the LIMS program UWDSM or the program modql. Currently statistical limits are recalculated and republished on an annual basis (See Chapter 21). However, for newly created tests or for determinative methods with explicit updating criteria, the limits may be recalculated more frequently. All modifications will be made by the QA Officers or their designee. For the Organic Chemistry Department the changes will be recorded in the QL record logbook (M:\EHD\ESS(4900)\ESS

Org(4940)\MSOffice\QL record log.doc). For the Inorganic Department, see ESS INO QA 113.

13.2.6. Significant Figures and Rounding

Limits are established for both minimum (lower) and maximum (upper) reportable values. Additionally, a maximum number of significant digits and decimal places (for rounding) are specified for each combination of method, matrix, and analyte (QL key). Raw results are checked against limits and rounded before reporting or export to other databases. However the raw results are retained on the LIMS system.

13.3. Radiochemistry

13.3.1. Setting of Control Limits for Instruments

13.3.1.1. Frequency.

Control Limits can be set at any time by selecting the "QC" option of the "WORK" program on the Data General (DG) computer.

Multiple copies of the same record and other erroneous data in the file should be removed before setting control limits.

Limit calculations should be based on 20 data points (if there are that many available).

Packard LS Analyzer Counting Control and Background Control limits are based on minimum performance recommended by the manufacturer.

Aptec Gamma System Control limits are dynamically set based on previous history.

Protean Control limits are set based the mean and the standard deviation for the daily checks. The background values are set based on instrument performance.

Gamma Products Control limits are dynamically set based on previous history. The background values are set based on instrument performance.

Ludlum background limits is set at 0.9 cpm. This allows for the required sensitivity to be achieved for a 1L sample with a 100 minute count

13.3.2. Displaying Control Charts

13.3.2.1. Frequency.

Control charts can be displayed at any time by selecting the program r:/ehd/ess(4900)/essradiochem(4970)/programs/qc/analysis/analyisqc.exe on the NT server. Multiple copies of the same record and other erroneous data in the file should be removed before creating plots.

13.3.3. Setting Method Control Limits

13.3.3.1. Frequency.

Control limits for any procedure can be set at any time by selecting the "QC" option of the "WORK" program on the Data General (DG) computer, however one time per year is sufficient. Multiple copies of the same record and other erroneous data in the file should be removed before creating new limits.

Limit calculations should be based on 20 data points. When the temporary data file is brought up by the program for editing, search the file for the last plotted data point. Remove all data in the file beyond this point. Then go back up through the file removing any points that were outliers on the previous plot, or should otherwise not be included in the control limit calculation. When 20 acceptable values have been selected, delete all data prior to this point.

Occasionally the limits calculated may be unrealistically wide or narrow due to the inclusion of too many points just within the limits, or consistently excellent performance. A comparison with previous limits should be used to judge if this is the case. If so, go back through the previous steps, this time accepting or deleting data to modify the limit accordingly.

For all methods a modified Shewart **accuracy** chart is used with these limits:

$$\sigma = \left[\frac{\Sigma\left(X-\overline{X}\right)}{N-1}\right]^{1/2}$$

$$UCL = \%\overline{R} \pm 3\sigma$$
$$WCL = \%\overline{R} \pm 2\sigma$$

For all analytical methods a modified Shewart **precision** chart is used with these limits:

$$UCL = 3.27\left(\overline{\Delta}\right) \qquad WCL = 2.51\left(\overline{\Delta}\right)$$

For all analytical methods a blank chart is used with these limits:

$$\sigma = \left[\frac{\Sigma\left(X-\overline{X}\right)}{N-1}\right]^{1/2}$$

$$UCL = \overline{X} \pm 3\sigma$$

$$WCL = \overline{X} \pm 2\sigma$$

13.4. Water Microbiology

The characteristics of data that measure accomplishment of a specified purpose can be expressed in terms of representativeness, comparability, precision, accuracy and completeness.

13.4.1. Representativeness

Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environment condition.

Appropriate selection of sample site and sampling procedure is critical to obtaining a sample that is representative of the environment in which it is collected.

13.4.2. Comparability

Comparability expresses the confidence with which one data set can be compared to another. Comparability of data is assured by following standard analytical procedures and calculating and reporting all data in generally accepted departments.

13.4.3. Precision

Precision is a measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions."

13.4.4. Accuracy

Accuracy is defined as the degree of a measurement (or an average of measurements of the same thing), X, with an accepted reference or true value, T, usually expressed as the difference of the reference or true value, 100(X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in a system.

13.4.5. Completeness

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 correct normal conditions.

Completeness of data is dependent upon both field and laboratory personnel. Improper sample collection, sample contamination, and out-of-control analytical procedures can cause the loss of data.

Table 13.1 — LIMS Calculation Types for QC Limits

The way in which QC results are calculated from the raw result is determined by the CALC field in the QL database. (The QL database is where limits are kept and described). The calculation codes are as follows:

Code	Description	Formula
1	absolute difference	| I - Q |
2	% difference on average	|((I - Q) / ((I + Q)/2)) * 100 |
3	% recovery	|(I - Q) / S * 100 |
4	% dif on ideal value	|((I - Q)/ I) * 100 |
5	result alone	Q
6	signed % ideal difference	((I - Q) / I * 100)

** I = reference result S = spike amt Q = raw QC result

It should be noted that calculation types 4,5, and 6 are rarely used.

14. Traceability

14.1. Biomonitoring

See Chapter 12

14.2. Inorganic and Organic Chemistry

14.2.1. Standards

Standards of the highest purity are obtained from various suppliers including Aldrich, Chem Service, Fisher Scientific, LabChem, Scott Specialty Gases, UltraScientific, VWR, and others. When available these standards are certified and traceable to the National Institute of Standards and Technology (NIST). The manufacturer's certificate of analysis is labeled with the same unique standard code number (see below) that will be marked on the bottle. This certificate is then kept on file. Commercially obtained standards may be pure materials or solutions that are ready to use. The lot number, expiration date, and concentration are indicated on the bottle or cylinder. Also all standards are dated when received to monitor the shelf life. All chemicals will be replaced before exceeding their expected shelf life (generally one to three years, unless otherwise indicated).

All standard solutions, including any necessary serial dilutions, are recorded in the proper standard logbook (see Figures 14.1, 14.2, 14.3 and 14.4 for sample pages), and are assigned a unique standard code number. When a working standard is prepared, the compound(s), standard code number, date prepared, analyst, expiration date, and solvent are noted in the logbook. All working standards are kept in containers and at temperatures that will not alter their integrity. All containers are clearly labeled with compound names, concentrations, unique standard code number, preparer, date prepared, expiration date, and solvent. The stability of all solutions is carefully monitored. They are re-standardized and/or prepared at a frequency determined by the appropriate analytical method.

14.2.2. Solvents and Gases

Any background contamination in secondary reagents can seriously affect the quality of an analysis. As a result only the highest purity solvents, dry chemicals and gases are used in the laboratory. All solvents used in the laboratory are ACS, pesticide grade, or better. Their purity may be checked by evaporating a quantity equal to that used in the analysis and analyzing it on the appropriate system. Alternatively, many analytical methods call for the analysis of a reagent and/or method blank, which would reveal any problems.

Gas cylinders are currently supplied by GasTech and delivered on a regular basis. The laboratory also uses a number of gas generators, both for UHP air and for UHP hydrogen. In addition, the laboratory draws gaseous nitrogen and argon from bulk liquid tanks located near the building. The gases are plumbed throughout the lab and the tank levels are checked on a regular basis. Gases that are used specifically for analysis, (not cryogenic cooling or powering

autosamplers) are UHP (99.999%) or better and may be equipped with additional traps (e.g., activated carbon, moisture) to increase their performance.

14.2.3. Dry Chemicals

The primary dry chemicals used in the laboratory and their proper preparations are listed below

14.2.3.1. Sodium Sulfate — ACS grade used as a drying agent in various analyses. It is prepared by heating at 130 °C for at least 24 hours.

14.2.3.2. Sodium Chloride — ACS grade used as a drying agent or as an ion source in liquid/liquid extraction procedures. It is prepared by heating in a muffle furnace at 400 for a minimum of 4 hours.

14.2.3.3. Florisil — 60/100 mesh used for column chromatography and for sample clean up. It is prepared by heating at 130 °C for 24 hours. Each new lot is checked for its lauric acid value and to determine the proper volume of elution solvent to achieve the desired separation. A spiking solution of the compounds of interest is prepared, spiked onto a column of 8 grams of florisil and eluted with the current volumes (50 mL of 94/6 hexane/ether for first fraction and 100mL 50/50 hexane/ether for the second). If there is significant carry-over of pesticides or splitting of dieldrin then adjustments will be made to the volumes. If the final volume changes it will be posted on the oven used for heating.

14.2.3.4. Silica Gel — 100/200 mesh used for column chromatography and for sample clean up. It is prepared by heating at 130 °C for 24 hours. Each new lot is checked to determine the proper activation necessary to achieve the desired separation. A spiking solution of the compounds of interest is prepared, spiked onto a column of 5 grams of silica gel which has been deactivated with 3.5% water and eluted with 45 mL of hexane and then 60mL 75/25 hexane/ether. If there is significant carry-over of PCBs or splitting of pesticides then adjustments will be made to the deactivation percentage. If the deactivation changes it will be posted on the oven used for heating.

All records for the above determinations are kept in the Organic Chemistry Department.

14.2.4. The Inorganic Chemistry Department has a stock reagent logbook (Book # 1) located in the dry chemical storage area / weigh room. Each chemical received by the lab is assigned a unique reagent code number, which is entered into the log along with data such as date received, date opened, date expires, lot #, and source. The code and above data are also written on the chemical container.

14.2.5. The Inorganic Chemistry Department also has working reagent logbooks located near their area of use. Whenever a working reagent is made, it is assigned a unique

code number. This code number is marked on the reagent container and recorded in the logbook along with the preparer's initials, date prepared, date expires, and traceability to stock reagents.

14.3. Radiochemistry

All radioactive materials are ordered though the University of Wisconsin Radiation Safety Department's Central Office of Receiving and Distribution (CORD). Each standard comes with a certificate of calibration and where appropriate is NIST traceable. Additionally, CORD must be notified of radioactive calibration sets that are delivered with new instruments.

Radioactive standards are given a unique number on arrival at the lab. Subsequent working solutions made from the stock are given derivations of this original number. This number is used to track the standard from receipt through disposal. Currently, the standards logbook is in the hard copy format (see Figure 14.5). An ACCESS database as also been created to track standards. Routine reagents (amount used, lot #, etc.) are recorded and tracked in a hard copy logbook. The preparation and procurement of all non-radioactive reagents is described in ESS RAD GENOP 018. The procurement of essential laboratory supplies is described in ESS RAD GENOP 001

A detailed procedure is in place for the procurement and disposal of radioactive materials (see ESS RAD GENOP 008).

14.4. Water Microbiology

14.4.1. Standards and Commercial Media

All standards and commercial media are dated when received to monitor the shelf life. They are disposed of when shelf life of the manufacturer is exceeded. An expiration date will be given as 6 months after the bottle is open for commercial media as requested by the NELAC auditor, but will be used until the expiration date of manufacturer. The shelf life begins when produced by manufacturer. (See Figure 14.5)

14.4.2. Media and Reagents

All media and reagents are prepared according to the recipes in Standard Methods or other test references.

14.4.2.1. Chemicals — All chemicals used in the microbiology laboratory are ACS or AR grade.

14.4.2.2. Dyes — All dyes used in the microbiology laboratory are certified by the Biological Stain Commission for bacteriological use.

14.4.2.3. Fluorescent Antibody — Meriflour Cryptosporidium/Giardia, Meridian Diagnostic, Cat # 250050, Cincinnati OH.

14.4.2.4. Elution Solution — lot number is recorded on bench records. Prepared in media room per recipe. Used within one week.

14.4.2.5. Phosphate Buffer Solution — Prepared in media room per recipe.

14.4.2.6. 8:2 Buffered Fixative — Prepared per recipe.

14.4.2.7. Percoll-Surcrose Flotation Solution — Prepared per recipe. Used within one week and refrigerated. Specific gravity checks with hydrometer. Specific gravity between and 1.10 to 1.13. Adjusted if out of control limits. No longer used for samples.

14.4.2.8. Sodium Thiosulfate Solution (2%) — Prepared in media room per recipe.

14.4.2.9. Sodium Dodecyl Sulfate Stock Solution (1%) — Prepared in media room per EPA recipe. Research only.

14.4.2.10. Tween 80 Stock Solution (1%) — Prepared in media room per EPA recipe.

14.4.2.11. Goat serum — Lot number is recorded in crypto and giardia Q.C. book. Purchased and stored at -15°C.

14.4.2.12. Difco Salmonella O Antiserum, poly A-I and Vi.

14.4.2.13. Oxiod Diagnostic Reagent for *E.coli* O157 test is used

14.4.2.14. Laureth-12 — PPG Industries, Gurnee, IL, cat # 06194

14.4.2.15. 1 M Tris — Made per recipe in media room.

14.4.2.16. 0.5 M EDTA — Made per recipe in media room.

14.4.2.17. Sodium Hydroxide - ASC grade

14.4.2.18. Hydrochloric Acid - ASC grade

14.4.2.19. Acetone

14.4.2.20. Glycerol

14.4.2.21. Ethanol

14.4.2.22. Methanol

14.4.2.23. Antifoam A, Sigma Chemical Co, cat # A5758

14.4.2.24. Dynabeads GC - Combo, Dynal Cat# 730.02, 730.12

14.4.2.25. Phosphate buffered saline (PBS)

14.4.2.26. 4'-6-diamidino-2-phenylindole (DAPI) stain, Sigma Cat #A5758

14.4.2.27. Concentrated stock phophate buffer solution

14.4.2.28. Magnesium Chloride solution

14.4.2.29. Dextrin Agar, Tech Pac, cat # 401019

14.4.2.30. Ampicillin sodium salt, Sigma Cat # A0166 or Biolife Cat # 4240012

14.4.2.31. Vancomycin hydrochloride, Sigma Cat # V2002

14.4.2.32. Nutrient Agar

14.4.2.33. Dry Slide BBL, Cat # 231746

14.4.2.34. 0.5% Trehalose, Sigma Cat # T0167

14.4.2.35. Purple Broth Base, Difco, cat # 0222-17

14.4.2.36. Tryptone Broth, Oxoid Cat # CM0087B

14.4.2.37. Kovac's reagent - Remel, Indole Reagent, cat # 21227

14.4.2.38. Readycult, EM Science

14.4.2.39. Chromocult, EM Science

14.4.2.40. Simplate - IDEXX

14.4.2.41. Colilert, Colisure, Colilert-18 and Enterolert

Purchased directly from manufacture for immediate use. Discarded after expiration date.

Colilert, Colilert-18 and Enterolert stored at room temperature. Discarded after manufacturer's expiration date.

Colisure stored in a refrigerator at 1 - 5°C. Discarded after manufacturer's expiration date.

14.4.2.42. Culture Media — When possible, media is ordered in small quantities. Each item is dated when received and when opened.

14.4.2.43. Opened media

Opened media supplies are stored in a cupboard unless indicated. After media jar is opened, it is disposed before manufacturer's expiration date. Per NELAC auditor a 6 month expiration date will be put on the bottle after being opened even though the powder will be used until the manufacturer's expiration date.

A magnetic stirrer or stirrer plus boiling water bath are used where needed for complete dissolution of media.

Sterilization of media is performed immediately after dissolving, or dissolving and dispensing, and method of sterilization is recorded in media preparation sheet or logbook.

Melted agar is held in the water bath incubator until ready to use (44-46°C) as measured by a thermometer inserted in to a similarly treated water bottle.

A permanent record for media prepared includes:

date of preparation

type of medium

sterilization time and temperature

final pH

technician's initial

14.4.2.44. Storage of Prepared media

Flasks, bottles, baskets of plates or racks of test tubes containing media are label with description of contents, date of preparation and initial of preparer.

MF agars are stored in inverted petri dishes with tight-fitting lids at 4°C for a maximum of 2 weeks.

Tubes with loose caps are prepared fresh whenever possible or are stored at room temperature for a period of less than 1 week.

Tubes and bottles in screw caps are stored at room temperature for less than three months.

Salmonella and *E.coli* agars are stored in sealed bags at 4°C for 1 month.

Media quality control tests are performed as follows:

A portion of each batch of medium is incubated at the temperature and time as specified in the procedure, inspected for growth and sterility check results are recorded in lab quality control book.

Positive and negative performance checks of media and procedures used are recorded in the quality control book.

If growth occurs, media is not used.

Figure 14.1 — Stock Standard Logbook for the Atomic Spectroscopy Area

Stan. Log Code #	Manf.	Elements & Conc.	Catalog #	Lot #	Date Recd	Date Exp	Anal.
2-1	Mallinckrodt	Tl (1000ppm)	H584	H584KEHT-P	9-20-90	5-92	JAT
2-2	Baker	Zn (1000ppm)	6946-01	C40612	3-21-90	10-91	JAT
2-3	Conostan	As (100 ppm wt/wt)	------	134	8-89	8-91	JAT
2-4	Conostan	Hg (100ppm wt/wt)	------	120	8-89	8-91	JAT
2-5	Conostan	Se (100ppm wt/wt)	-------	117	8-89	8-91	JAT
2-6	Conostan	Ag, Al, B, Ba, Ca, Cd, Cr, Cu, Fe, Mg, Mn, Mo, Na, Ni, P, Pb, Si, Sn, Ti , V, Zn (All 500 ppm wt/wt)	--------	21182	8-89	8-91	JAT

[1]Standard log code # consists of: Stock Standard logbook page # followed by the logbook line #. \usr\jk\asa.lb

Figure 14.2 — Working Standard Logbook Atomic Absorption Area

Working Standard Logbook
for the Atomic Absorption Spectroscopy Area
(Matrix - 0.5% HNO_3) **Page 1**

Date	Time	Anal	Work. Stan. Code #[1]	Stock Stan. Log Code #	Elements and Concentration (µg/L)	Use or Calibration	Purpose 2nd Source or Other	Comments
11-26-90	8:30	JAT	Ag-1	2-14	Ag (2,5,10)	√		
12-10-90	9:45	JAT	Ag-2	2-14	Ag (2,5,10)	√		
12-10-90	11:00	JAT	PC-1	3-6	As,Sb(50);SE(40);Pb,Cr,Cu,Tl(20);Ag(4);Cd(2)		√	Control Sample
1-14-91	11:00	JAT	SP-1	3-4	High Std:As(100);Se(80);Cu,Cr, Pb(40);Cd(4);mid=½;Low =¼;	√		
1-22-91	8:30	JAT	SP-2	3-4	" (Same as above)	√		
1-23-91	2:30	AFC	K-1	2-7	K(0.5,1,5,10 ppm)	√		
1-29-91	8:45	AFC	Zn-1	2-2	Zn (10,50,100)	√		

[1] Code # consists of: Element(s); analysis type (I-ICP, A-AA); # of times \usr\jk\icbc\abs.lb

Figure 14.3 — VOC Standards Log

Date	Analyst	Parent standard & source	Lot #	Dilution	Comment
11/3/97	MR	ULTRAScientific BFB @ 2000µg/mL & 1,2-Diclbenz-d4 @ 2000µg/mL	BFB: L-0733 d4: L-0771	100µL of each made up to 1mL in MeOH. Final conc. 200µg/mL	This dilution used for a working standard to spike calibration standard solutions. Final conc. matches that of the 60 comp. standard. (DWM-580)
11/3/97	MR	ULTRAScientific 60 component volatiles standard @ 200µg/mL DWM-580	L-1030	none	used for calibration and as the working solution for CCV spikes
11/17/97	MR	ULTRAScientific 60 component volatiles standard @ 200µg/mL DWM-580	L-1030	none	used for calibration and as the working solution for CCV spikes
12/08/97	MR	ULTRAScientific 60 component volatiles standard @ 200µg/mL DWM-580	L-1030	none	used for calibration and as the working solution for CCV spikes
12/09/97	MR	ULTRAScientific BFB @ 2000µg/mL & 1,2-Diclbenz-d4 @ 2000µg/mL	BFB: L-0733 d4: L-0771	937.5 µL of each made upto 25mL in MeOH.	used for surrogate addition on the Precept. Standard Vessel #2. Yields a final concentration of 3 µg/L, before loop volume is factored in.
12/09/97	MR	Supelco Fluorobenzene @ 2000µg/mL	LA66012	937.5 µL in 25mL of MeOH	used for Internal standard. Added to standard vessel #1, yielding a final concentration of 3 µg/L.
12/15/97	MR	ULTRAScientific 60 component volatiles standard @ 200µg/mL DWM-580	L-1030	none	used for calibration and as the working solution for CCV spikes
12/23/97	MR	ULTRAScientific 60 component volatiles standard @ 200µg/mL DWM-580	L-1030	none	used for calibration and as the working solution for CCV spikes. Also used for calibration on 12/29/97.
01/06/98	MR	ULTRAScientific 60 component volatiles standard @ 200µg/mL DWM-580	L-1030	none	used for calibration and as the working solution for CCV spikes

Figure 14.4 — ESS Organics Standard Preparation Log

PAH STOCK 20 ID: _ _ _ _ - _ _ _ _ _ _ - _ _ - _ _

Aliquot (mL)		Conc. (ug/mL)
_____mL of _____ug/mL Naphthalene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Acenapthylene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL 1-Methylnaphthalene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL 2- Methylnaphthalene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Acenaphthene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Fluorene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Phenanthrene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL 3,6 Dimethylnaphthalene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Anthracene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Fluoranthene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Pyrene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Benzo(a)anthracene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Chrysene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Benzo(e)pyrene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Benzo(b)fluoranthene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Benzo(k)fluoranthene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Benzo(a)pyrene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Dibenzo(a,h)anthracene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Benzo(ghi)perylene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	
_____mL of _____ug/mL Indeno(1,2,3-c.d.)pyrene	_ _ _ _ - _ _ _ _ _ _ - _ _ _ _	

SOLVENT: _____ **FINAL VOLUME:** _____**(mL)** **EXPIRATION DATE:** _____

*Note: The ID # includes the preparer's initials and the date.

Figure 14.5 — Radiochemical Standard Preparation

Standard Number (yr.pg.0):_____

Isotope: _____ Halflife:_____

Tech's Initials:_____ Supplier Soln. Number: _____

Supplier Calibration Date: _____ SLH Calibration Date: _____

A_0 in nCi/g:_____ Today's Date: _____

Gross Weight: _____ Tare Weight: _____ Net Weight: _____

Total Activity (A_0 in pCi):_____ Diluted To:_____ **Activity (pCi/mL):** _____

Matrix Prepared with Stock Standard

Description:_____

Tech's Initials: _____ Standard Number: _____

Previous Calibration Date: _____ New Calibration Date:_____

A_0 in pCi/mL: _____ Volume Used: _____

Total Activity (A_0 in pCi): _____ Diluted To:_____ **Activity (pCi/mL):** _____

Matrix Prepared with Stock Standard

Description:_____

Tech's Initials: _____ Standard Number: _____

Previous Calibration Date: _____ New Calibration Date:_____

A_0 in pCi/mL: _____ Volume Used: _____

Total Activity (A_0 in pCi): _____ Diluted To:_____ **Activity (pCi/mL):** _____

Figure 14.6 — Total Coliform Reagent Quality Log

Date	Prod. Exp. Date	Trtmnt	Color Change	Fluorescence	Initials

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- WDNR, "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual, Edition 1", PUBL-WW-033-96 (1996)
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16.3. Standard Operating Procedures and Policies

16.3.1. WSLH Organizational Policies

The full text of these policies can be found on-line at:
http://www.slh.wisc.edu/local/admin/policy/policybook.html

Absence and Tardiness
Accident Reporting
Authorship Guidelines
Awards of Recognition
Bench Training
Blood Donations and Selling Blood
Breakroom Policy
Bulletin Board Use
Compensatory Pay and Time Off
Computers - Use of WSLH Computers at Home
Computers - Usage Policy at WSLH
Confidentiality Policy of WSLH
Dress Code Policy
Eating and Drinking at WSLH
Employee Assistance
Entertainment at Special Activities
Use of WSLH Equipment, Supplies, Personnel and Facilities
Eye Protection
Footwear in the Laboratory
Hepatitis B Vaccination
Key Distribution
Laboratory Coat Required Use
Laboratory Tests to Assist Colleagues or Outside Investigators
Legal Holidays
Leave of Absence
Letterhead Use
Meal Breaks
Operational Hours of WSLH

Orientation of New Employees

Overtime and Professional Time

Parking Permits

Parties During Holidays

Paycheck and Earnings Statement

Personal Holidays

Personnel Files

Microsoft Word Document

Political Activities

Press Releases, Conferences, Interviews

Professional Development Funding

Professional Organizations - WSLH Work For

Radio Usage

Recognition Activities

References and Recommendations

Rest Periods and Work Breaks

Sick Leave

Signature Authority

Smoking and Use of Tobacco Products

Telephone Use

Timesheet Policies

Tours of WSLH

Tuition Reimbursement

Vehicles - Use of WSLH

Visitors Policy

WSLH Internal Web Page

Work Hours

Web Pages

Workshops, Seminars, Training Courses

WSLH Professional Memberships

16.3.2. Environmental Health Division General Operating Procedures

EHD GENOP 000 SOP	Table of Contents.doc
EHD GENOP 001 SOP	Numbering System.doc
EHD GENOP 002 SOP	LIMS PP Accounts.doc
EHD GENOP 003 SOP	Ordering Office Supplies.doc
EHD GENOP 004 SOP	Ordering Lab Supplies.doc
EHD GENOP 005 SOP	LIMS AC Accounts.doc
EHD GENOP 006 SOP	Processing Contracts.doc
EHD GENOP 007 SOP	Administrative Reports.doc
EHD GENOP 008 SOP	Personhr Application.doc
EHD GENOP 009 SOP	Billing.doc
EHD GENOP 010 SOP	Records.doc
EHD GENOP 011 SOP	Security.doc
EHD GENOP 012 SOP	Classified Hiring Process.doc
EHD GENOP 013 SOP	Cooler, Freezer, Incubator Repair.doc
EHD GENOP 014 SOP	Academic Staff Hiring.doc
EHD GENOP 015 SOP	Yearly Balance Service.doc
EHD GENOP 016 SOP	Cost Account.doc
EHD GENOP 017 SOP	Complaints
EHD GENOP 018 SOP	Budgeting
EHD GENOP 019 SOP	Processing Grants
EHD GENOP 020 SOP	Billing Grants
EHD GENOP 021 SOP	Tank Room
EHD GENOP 022	Chain of Custody/Enforcement Sample Procedure for Sample Control & Receiving Dept.
EHD GENOP 023	Management System Review
EHD GENOP 024	ESS Sample Tracking Program
EHD GENOP 025	Sample Tracking Queries & Reports
EHD GENOP 026	Chemical Hygiene Plan

16.3.3. Biomonitoring Department

16.3.3.1. General

ESS BIO GENOP 010	Temperature Monitoring
ESS BIO GENOP 015	Sample Collection for Bioassay Toxicity Testing
ESS BIO GENOP 020	Sample Receiving
ESS BIO GENOP 030	Monitoring and Maintenance of Water Treatment and Purification Systems
ESS BIO GENOP 040	Sediment Preparation
ESS BIO GENOP 050	Balance Calibration
ESS BIO GENOP 060	Carbon Dioxide Entrapment for pH Control
ESS BIO GENOP 080	Cytofluor
ESS BIO GENOP 201	Estimation of Algal Concentration Using a Hemacytometer
ESS BIO GENOP 210	Preparation of Yeast/Fish Food/Cereal Leave Food
ESS BIO GENOP 200	Culture of *Selenastrum capricornutum*
ESS BIO GENOP 220	Brine Shrimp Hatching
ARCHIVED	Preparation of Food for *Daphnia magna*
ESS BIO GENOP 240	Preparation of Synthetic Hard Water
ESS BIO GENOP 242	Collection of Kegonsa Water
ARCHIVED	Collection of Black Earth Creek Water
ESS BIO GENOP 250	Preparation of Selenium and Vitamin B12 Supplement
ESS BIO GENOP 300	Culturing of *Pimephales promelas*
ARCHIVED	Mass Culture of *Ceriodaphnia dubia*
ESS BIO GENOP 325	Individual Culture of *Ceridaphnia dubia*
ARCHIVED	Mass Culture of *Daphnia magna*
ARCHIVED	Mass Culture of *Chironomus tentans*
ARCHIVED	Mass Culture of *Hyalella azteca*

16.3.3.2. Analytical Methods

ESS BIO METHOD 100	Alkalinity
ESS BIO METHOD 101	Total Hardness
ESS BIO METHOD 102	pH
ESS BIO METHOD 103	Conductivity
ESS BIO METHOD 104	Dissolved Oxygen
ESS BIO METHOD 105	Total Ammonia

ESS BIO METHOD 106	Total Residual Chlorine
ESS BIO METHOD 400	96 Hour Static Renewal Acute Toxicity Test with Juv. FHM
ESS BIO METHOD 10	48 Hour Static Renewal Acute Toxicity Test with *Ceriodaphniadubia*
ESS BIO METHOD 415	96 Hour Static Renewal Accute Toxicity Test with *Hyalella azteca*
ESS BIO METHOD 416	Microtox Screen Test
ESS BIO METHOD 420	Larval FHM Survival and Growth Test
ESS BIO METHOD 430	*Ceriodaphnia.dubia* Survival and Reproduction Test
ESS BIO METHOD 431	TIE Phase I
ESS BIO METHOD 432	TIE Phase II
ESS BIO METHOD 435	48 Hour Acute Sediment Test with *Ceriodaphnia.dubia*
ESS BIO METHOD 440	48 Hour Sediment Test with *Daphnia magna*
ESS BIO METHOD 460	10 Sediment Test with *Chironomus tentans*
ESS BIO METHOD 480	10 Day Sediment Test with *Hyalella azteca*
ESS BIO METHOD 500	*Selenastrum capricornutum* 96-h Growth Test
ESS BIO METHOD 505	96 Hour Static Acute Reference Toxicity Test with *Pimephales promelas*
ESS BIO METHOD 510	48 Hour Static Acute Reference Toxicity Test with *Ceriodaphnia dubia*
ESS BIO METHOD 515	Larval *Pimephales promelas* Survival and Growth Reference Toxicity Test
ESS BIO METHOD 520	*Ceriodaphnia dubia* Survival and Growth Reference Toxicity Test
ESS BIO METHOD 600	Report Writing
ESS BIO METHOD 1000	Glassware Cleaning
ESS BIO METHOD 1020	Randomization
Archived	Microsomal Preparation for EROD
ESS BIO METHOD 2030	Screen Test for the Cyanobacteria *Cylindrospemopsis*

16.3.4. Inorganic Chemistry Department

16.3.4.1. General

ESS INO GENOP 001	How to Write an SOP
ESS INO GENOP 100	Annual Thermometer Check
ESS INO GENOP 102	Data Transfer from TJA 61E ICP
ESS INO GENOP 103	Sample Acceptance Policy, Inorganic Chem. Dept.
ESS INO GENOP 104	Checking in Samples for Metals Analysis
ESS INO GENOP 105	Raytek Thermometer--Annual Calib. & Weekly Check
ESS INO GENOP 106	Inorganic Sample Receipt
ESS INO GENOP 108	Procedure for Creating a WL Worklist
ESS INO GENOP 110	Sample Disposal Protocol
ESS INO GENOP 112	Maintenance of Personnel Training Files
ESS INO GENOP 200	Pipette Performance Checks
ESS INO GENOP 201	Volumetric Dispenser Performance Checks
ESS INO GENOP 202	Calibration, Maintenance, and Accuracy Verification Procedure for Balances
ESS INO GENOP 203	Handling, Maintenance, & Calibration of Weights
ESS INO GENOP 210	Saturday Work Procedures
ESS INO GENOP 250	Operation & Maintenance of the Trace Metal Clean Lab
ESS INO GENOP 300	Daily Temperature Check
ESS INO GENOP 500	Guidelines for Method Development Work
ESS INO GENOP 1000 series	LIMS Procedures

16.3.4.2. Quality Assurance

ESS INO QA 101	Bottle Check Procedure
ESS INO QA 103	Q.C. Audit of Analytical Runs — Metals
ESS INO QA 104	Q.C. Audit of Analytical Runs —ESS TMCL
ESS INO QA 107	Q.C. Audit of Analytical Runs--Wet Chemistry Area
ESS INO QA 108	Internal Audits
ESS INO QA 109	PT Sample Procedures
ESS INO QA 113	Quality Control Limit Updates
ESS INO QA 114	qawrksht Instructions
ESS INO QA 115	Initial DOC & Annual Continued Proficiency Check Procedures
ESS INO QA 116	LOD Procedures

16.3.4.3. Instrument Operating Procedures

ESS INO IOP 100	Computer Protocol Manual
ESS INO IOP 150	Chlorophyll, Spectrophotometric, IOP
ESS INO IOP 200	IOP for QuikChem 8000 Auto Ion Analyzer
ESS INO IOP 260	BOD Computer Procedures
ESS INO IOP 300	Solids Transfer
ESS INO IOP 400	IOP for Beckman DU-650 Spectrophotometer
ESS INO IOP 500	IOP for the TJA 61-E ICP
ESS INO IOP 540	IOP for the FIMS 100

16.3.4.4. Analytical Methods

ESS INO METHOD 100.1	Sample Preparation, Sediments and Sludges
ESS INO METHOD 100.2	Filtering Procedure
ESS INO METHOD 105.1	Ultra Trace Level Cleaning and Tracking Procedures
ESS INO METHOD 115.1b	Automated Alkalinity, pH, and Conductivity
ESS INO METHOD 141.0	Chloride
ESS INO METHOD 141.3	Chloride in Solids
ESS INO METHOD 150.1	Chlorophyll, Spectrophotometric
ESS INO METHOD 150.2	Periphyton Chl A
ESS INO METHOD 151.1	Chlorophyll *a*, Fluorescence
ESS INO METHOD 170.1	Color, Visual Comparison Method
ESS INO METHOD 180.1	Cyanide, Total and Amenable to Chlorination
ESS INO METHOD 180.2	Cyanide, Solids, Total
ESS INO METHOD 180.3	Cyanide, Weak Acid Dissociable
ESS INO METHOD 200.2	Hardness (Calculation Method)
ESS INO METHOD 200.3	Hardness Screening Test
ESS INO METHOD 210.1	Methylene Blue Active Substances (MBAS), Colorimetric
ESS INO METHOD 210.2	Methylene Blue Active Substances, Screening Technique
ESS INO METHOD 220.2	Ammonia N & Nitrate + Nitrite N Digests in Solids
ESS INO METHOD 220.3	Ammonia Nitrogen & Nitrate + Nitrite Nitrogen
ESS INO METHOD 220.8	Nitrite Nitrogen, Manual Colorimetric
ESS INO METHOD 220.9	Nitrate & Nitrite Nitrogen, Fluoride
ESS INO METHOD 221.0	Ammonia, Ion Selective Electrode
ESS INO METHOD 230.3	Total Kjeldahl Nitrogen with Copper Sulfate
ESS INO METHOD 230.4	Total Nitrogen in Kjeldahl Digests of Soils
ESS INO METHOD 250.4	HEM by Extraction and Gravimetry (Oil & Grease; Total Petroleum Hydrocarbons)
ESS INO METHOD 250.5	Oil and Grease
ESS INO METHOD 260.1	Biochemical Oxygen Demand

ESS INO METHOD 260.2	Biochemical Oxygen Demand, Long Term
ESS INO METHOD 260.3	Calculation for Ultimate Carbonaceous BOD
ESS INO METHOD 280.2	Chemical Oxygen Demand
ESS INO METHOD 290.1	Oxygen, Dissolved (Modified Winkler Technique)
ESS INO METHOD 295.0	Soil and Sediment pH
ESS INO METHOD 300.0	pH, Electrometric
ESS INO METHOD 301.0	pH, Electrometric, Hazardous Waste
ESS INO METHOD 310.2	Phosphorus, Total, Persulfate Digestion
ESS INO METHOD 310.3	Phosphorus, Dissolved
ESS INO METHOD 310.4	Total Phosphorus Digests of Soil
ESS INO METHOD 320.1	Total Dissolved Solids (Dried at 180°C)
ESS INO METHOD 330.1	Total Solids, Gravimetric (Dried at 103 - 105°C)
ESS INO METHOD 330.2	Percent Solid--Solid and Semi-solid Samples
ESS INO METHOD 331.0	Bulk Density Analysis of Sediments
ESS INO METHOD 340.1	Total Suspended Solids, Volatile Suspended Solids
ESS INO METHOD 340.2	Total and Volatile Suspended Solids (0.7 um Particle Retention Size)
ESS INO METHOD 360.2	Silica, Dissolved, Low Level
ESS INO METHOD 360.2a	Silica, Dissolved, Micro Level
ESS INO METHOD 370.3a	Sulfates
ESS INO METHOD 370.4	Sulfates in Solids
ESS INO METHOD 375.1	Sulfide, Titrimetric Iodine
ESS INO METHOD 375.2	Sulfide, Colorimetric--Methylene Blue
ESS INO METHOD 376.1	Sulfite, Iodometric
ESS INO METHOD 378.1	Total Sulfur/Sulfate
ESS INO METHOD 380.3	Turbidity, Nephelometric
ESS INO METHOD 400.2	Inductively Coupled Plasma--Emission Spectrometry
ESS INO METHOD 400.3	Graphite Furnace Atomic Absorption Spectrometry
ESS INO METHOD 400.4	Trace Elements in Water by ICP-MS
ESS INO METHOD 400.5	Graphite Furnace Atomic Absorption Spectrometry (Tl)
ESS INO METHOD 470.3	Chromium, Hexavalent
ESS INO METHOD 470.4	Chromium, Hexavalent, Solids
ESS INO METHOD 470.5	Hexavalent Chromium by Ion Chromatography
ESS INO METHOD 540.2	Digestion of Non-Aqueous Samples for CVAA-Hg
ESS INO METHOD 540.3	Digestion of Aqueous Samples for CVAA-Hg
ESS INO METHOD 540.4	Digestion of Tissue Samples for CVAA-Hg
ESS INO METHOD 541.1	Tot. Mercury--Cold Vapor Atomic Fluorescence Spectroscopy
ESS INO METHOD 541.2	Tot. Mercury--Automated Cold Vapor Atomic Fluorescence Spectroscopy

ESS INO METHOD 560.1	Potassium, Direct Aspiration Atomic Absorption
ESS INO METHOD 620.2	Digestion for Metals in Tissue by ICP
ESS INO METHOD 715.1	TCLP
ESS INO METHOD 730.1	Digestion of Total Liquids for ICP
ESS INO METHOD 740.1	Digestion of Total Liquids for GFAA
ESS INO METHOD 750.1	Digestion of Solid Samples for ICP
ESS INO METHOD 750.2	Digestion of Solid Samples for GFAA
ESS INO METHOD 760.1	Digestion of Total Liquids for As and Se by GFAA
ESS INO METHOD 780.3	Digestion of Total Recoverable Liquids or Ag for ICP
ESS INO METHOD 780.5	Digestion of Total Recoverable Liquids for GFAA
ESS INO METHOD 780.6	Digestion of Total Recoverable Liquids for GFAA (Tl)
ESS INO METHOD 805.1	Neutralization Procedure for Acidic Wastes

16.3.5. Organic Chemistry Department

16.3.5.1. General

ESS ORG GENOP 0000	Standard Operating Procedures - Table of Contents
ESS ORG GENOP 0025	Solvent Disposal
ESS ORG GENOP 0026	PCB Waste Disposal
ESS ORG GENOP 0027	Summa Canister Cleaning
ESS ORG GENOP 0028	Sample Receipt
ESS ORG GENOP 0029	Sample Disposal
ESS ORG GENOP 0030	Sample Release
ESS ORG GENOP 0031	Disposal of Surplus Chemicals and Dry Solids
ESS ORG GENOP 0032	Silica Gel Calibration
ESS ORG GENOP 0033	Lauric Acid Value (for florisil)
ESS ORG GENOP 0034	Hg Waste Disposal
ESS ORG GENOP 0035	Data Transfer
ESS ORG GENOP 0036	General Chemistry Standard Log book
ESS ORG GENOP 0037	VOC Standard Logbook
ESS ORG GENOP 0038	Air Standard Logbook
ESS ORG GENOP 0039	Fish Grinding
ESS ORG GENOP 0040	Mailer/Kit Preparation
ESS ORG GENOP0039	Tissue Grinding
ESS ORG GENOP 0040	Mailer/Kit Preparation
ESS ORG GENOP 0041	Ordering Procedures
ESS ORG GENOP 0042	Disposal of Glass Containers, Broken Glass and Other Sharps
ESS ORG GENOP 0043	Results Updating Protocol
ESS ORG GENOP 0044	Sample Tracking Protocol
ESS ORG GENOP 0045	Sample Entry Protocol
ESS ORG GENOP 0046	Data Backups
ESS ORG GENOP 0047	Glassware Washing
ESS ORG GENOP 0048	Preparation of Resin Columns and Filters
ESS ORG GENOP 0049	Thermometer Calibration
ESS ORG GENOP 0050	Preparation and Cleaning of XAD2 Resin
ESS ORG GENOP 0051	Protocol for Generating an Enforcement Disposition Letter
ESS ORG GENOP 0052	Entering SDWA Requests

ESS ORG GENOP 0053	Shipping SDWA Mailers
ESS ORG GENOP 0054	Shipping WDNR Mailers
ESS ORG GENOP 0055	SDWA Rejection and Resample
ESS ORG GENOP 0100	Methods Manual Construction and Maintenance
ESS ORG GENOP 0100	Methods Manual Construction and Maintenance

16.3.5.2. Quality Assurance

ESS ORG QA 0001	qawrksht
ESS ORG QA 0002	Blind Samples
ESS ORG QA 0003	Internal Audit
ESS ORG QA 0004	QC Updates
ESS ORG QA 0005	Pipette Checks
ESS ORG QA 0006	PT samples
ESS ORG QA 0007	Bottle Checks
ESS ORG QA 0008	Data Auditing
ESS ORG QA 0009	Choosing and Validating Samples Used for Laboratory Matrices

16.3.5.3. Instrument Operating Procedures

ESS ORG IOP 0110	Operation of the Gel Permeation Chromatograph
ESS ORG IOP 0120	Operation of the Total Organic Carbon Analyzer
ESS ORG IOP 0130	Operation of the HP 5890 Packed Column GC
ESS ORG IOP 0140	Operation of the HP 5890 Capillary Column GCs
ESS ORG IOP 0150	Operation of the Seimens Heart Cutting GC
ESS ORG IOP 0160	Operation of the HP 6890 Capillary Column GCs
ESS ORG IOP 0170	Operation of the HP 5890 NPD
ESS ORG IOP 0180	Operation of the Waters HPLC
ESS ORG IOP 0190	Operation of the HP 1090 HPLC
ESS ORG IOP 0200	Operation of the Nutech/HP5890 GCs for PAMS Analysis
ESS ORG IOP 0210	Operation of the Nutech/HP5890 GCs for Air Toxics Analysis
ESS ORG IOP 0220	Operation of the HP5890/5973 for SDWA Volatiles
ESS ORG IOP 0230	Operation of the HP5890/5973 for Volatiles in Water and Waste
ESS ORG IOP 0240	Operation of the HP5890/5973 for Volatiles in Sediment and Soil

ESS ORG IOP 0250	Operation of the Finnigan Magnum GC / Ion Trap MS
ESS ORG IOP 0260	Operation of the INCOS 50 GC/MS
ESS ORG IOP 0270	Operation of the Tekmar Purge & Trap / Tracor 540 GC for GRO
ESS ORG IOP 0280	Operation of the HP5890 FID for DRO
ESS ORG IOP 0290	Placeholder
ESS ORG IOP 0300	Blending Gas Standards for Air Analysis
ESS ORG IOP 0310	Operation of the Sonic Dismembrator
ESS ORG IOP 0320	Flashpoint Apparatus
ESS ORG IOP 0330	Operation of the Karl Fischer Apparatus

16.3.5.4. Analytical Method Procedures

ESS ORG Method 1206	Neutral Extractable Pesticides and Metabolites
ESS ORG Method 1207	Determination of Nitrogen and Phosphorus Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector - EPA Method 507
ESS ORG Method 1208	Determination of Chlorinated Pesticides and PCBs in Water by Gas Chromatography with an Electron Capture Detector - EPA Method 508
ESS ORG Method 1210	Neutral Extractable Pesticides and PCBs
ESS ORG Method 1215	Determination of Chlorinated Acids in Water by Gas Chromatography with an Electron Capture Detector - EPA Method 515.1
ESS ORG Method 1220	Acid Extractables
ESS ORG Method 1241	Determination of Nitrogen and Phosphorous Containing Pesticides in water by Gas Chromatography with a Nitrogen Phosphorous Detector - SW846 Method 8141A - Revision 1.0 - (September 1994)
ESS ORG Method 1247	Determination of Glyphosate in Drinking Water by Direct Aqueous Injection HPLC, Post Column Derivatization and Flourescence Detection - EPA Method 547
ESS ORG Method 1248	Determination of Endothall in Water by Ion Exchange Extraction and Flame Ionization Detection - EPA Method 548.1
ESS ORG Method 1249	Determination of Diquat and Paraquat in Drinking Water by Liquid - Solid Extraction and HPLC with an Ultraviolet Detector - EPA Method 549
ESS ORG Method 1252	Measurement of N-methylcarbamoyloximes and

	N-methylcarbamates in Water by Direct Aqueous Injection HPLC with Post Column Derivitization - EPA Method 531.1
ESS ORG Method 1253	Determination of Haloacetic Acids and Dalapon In Drinking Water By Liquid-Liquid Extraction, Derivatization and Gas Chromatography With Electron Capture Detection - EPA Method 552.2
ESS ORG Method 1289	Determination of Formaldehyde in Water by High Performance Liquid Chromatography (HPLC) - SW846 - EPA Method 8315
ESS ORG Method 1293	PCBs and Pesticides in Surface Water by XAD-2 Resin Extraction
ESS ORG Method 1322	Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series - EPA Method 8021
ESS ORG Method 1323	Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series - EPA Method 502.2
ESS ORG Method 1324	EDB and DBCP in Water by Microextraction and Gas Chromatography - EPA Method 504
ESS ORG Method 1325	Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Mass Spectroscopy - EPA Method 524.2
ESS ORG Method 1330	Petroleum Products in Water
ESS ORG Method 1335	Gasoline Range Organics (GRO) and Petroleum Volatile Organic Compounds (PVOC) in Water by the Wisconsin Modified GRO Method
ESS ORG Method 1336	Diesel Range Organics (DRO) in Water by the Wisconsin Modified DRO Method
ESS ORG Method 1360	Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Mass Spectroscopy - EPA Method 8260
ESS ORG Method 1410	Pesticide and PCB Residues in Tissue
ESS ORG Method 1420	Chlorophenols in Tissue
ESS ORG Method 1440	PCB Analysis in Tissue
ESS ORG Method 1460	Polynuclear Aromatic Hydrocarbons in Fish Tissue by HPLC - SW846 Method 8310 (Rev. 0, September 1986)
ESS ORG Method 1510	Sediment and Soil for Pesticide and PCB Residues

ESS ORG Method 1532	Sediment and Soil for 2,4-D Residues
ESS ORG Method 1540	Chlorophenols in Soils
ESS ORG Method 1550	Volatile Organic Compounds in Soil by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series - EPA Method 8021
ESS ORG Method 1551	Volatile Organic Compounds in Soil by Purge-and-Trap using Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS) - EPA Method 8260B - Revision 2.0 - (December, 1996)
ESS ORG Method 1555	Gasoline Range Organics (GRO) and Petroleum Volatile Organic Compounds (PVOC) in Soil by the Wisconsin Modified GRO Method
ESS ORG Method 1556	Diesel Range Organics (DRO) in Soil by the Wisconsin Modified DRO Method
ESS ORG Method 1560	Total Organic Carbon in Sediment by the Slurry Method
ESS ORG Method 1570	Base/Neutral and Acid Extractable Priority Pollutants in Soils and Sediments by GC/MS analysis
ESS ORG Method 1571	Priority Pollutant Pesticides and PCBs in Sediment and Soil
ESS ORG Method 1580	PAHs in Sediment and Soil
ESS ORG Method 1581	Polynuclear Aromatic Hydrocarbons in Soil and Sediment by HPLC - SW846 Method 8310 (Rev. 0, September 1986)
ESS ORG Method 1590	Petroleum Products in Soil
ESS ORG Method 1608	Determination of Chlorinated Pesticides and PCBs in Wastewater and Storm Run-off Water by Gas Chromatography with an Electron Capture Detector - EPA Method 608
ESS ORG Method 1611	Polynuclear Aromatic Hydrocarbons in Wastewater by HPLC - EPA Method 610
ESS ORG Method 1612	Glycols in Water by Gas Chromatography - Modified EPA Method 8015A - July 1992
ESS ORG Method 1620	Chlorophenols in Effluents
ESS ORG Method 1660	Total Organic Carbon in Water
ESS ORG Method 1662	Dissolved Organic Carbon in Water
ESS ORG Method 1663	Dissolved Inorganic Carbon
ESS ORG Method 1670	Base/Neutral and Acid Extractables in Priority Pollutants in Effluents by GC/MS analysis (EPA Method 625)
ESS ORG Method 1671	Priority Pollutants - Pesticides/PCBs in Effluents

	by Gas Chromatography with Electron Capture Detection (EPA method 608)
ESS ORG Method 1682	PCBS in Municipal Sewage Sludge (EPA Method 8082A)
ESS ORG Method 1710	Volatile Organic Compounds in Waste and Waste Oils by Purge and Trap Capillary Column Gas Chromatography with Photoionization \ and Electrolytic Conductivity Detectors in Series - EPA Method 8021
ESS ORG Method 1720	Flash Point - ASTM D93-85
ESS ORG Method 1721	Water Content in Waste - ASTM 3792-91
ESS ORG Method 1722	Alcohol Content of Waste - NEIC Method 09/17/93
ESS ORG Method 1730	PCBs in Waste Oils and Transformer Fluid
ESS ORG Method 1740	TCLP Analysis - EPA Method 1311
ESS ORG Method 1760	Volatile Organic Compounds in Waste and Waste Oils by Purge-and-Trap using Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS) - EPA Method 8260B - Revision 2.0 - (December, 1996)
ESS ORG Method 1810	Blood Serum for PCB Congeners and Pesticides
ESS ORG Method 1820	Urine for Base/Neutral Pesticides and Metabolites
ESS ORG Method 1910	C2 - CESS ORG Method 12 Hydrocarbons in Ambient Air
ESS ORG Method 1911	Reformulated Gasoline Compounds (RFG) in Ambient Air
ESS ORG Method 1912	Toxic Organic Compounds in Ambient Air - EPA Method T014
ESS ORG Method 1920	Ambient Air for Pesticide and PCB Residues - EPA Method T04

16.3.6. Radiochemistry Department

16.3.6.1. General

ESS RAD GENOP 000	Table of Contents SOPs.doc
ESS RAD GENOP 001 SOP	Kit Preparation.doc
ESS RAD GENOP 002 SOP	Customer Orders.doc
ESS RAD GENOP 005 SOP	Bubbler Washing.doc
ESS RAD GENOP 006 SOP	Computer Programs.doc
ESS RAD GENOP 007 SOP	Oven Check.doc
ESS RAD GENOP 008 SOP	Radioactive Standards.doc
ESS RAD GENOP 009 SOP	Refrigerator Check.doc
ESS RAD GENOP 010 SOP	Neutralization Operation.doc
ESS RAD GENOP 011 SOP	Sample Disposal.doc
ESS RAD GENOP 012 SOP	Emergency Response.doc
ESS RAD GENOP 013 SOP	Emergency Action Plan.doc
ESS RAD GENOP 014 SOP	Rad Records.doc
ESS RAD GENOP 015 SOP	Recretpo.doc
ESS RAD GENOP 016 SOP	Pipette Calibration.doc
ESS RAD GENOP 017 SOP	File Transfers.doc
ESS RAD GENOP 018 SOP	Reagent Guide.doc
ESS RAD GENOP 019 SOP	Rad Billing.doc
ESS RAD GENOP 020 SOP	Sample Checkin.doc
ESS RAD GENOP 021 SOP	Radon Air VB App.doc
ESS RAD GENOP 022 SOP	RVU Report.doc

16.3.6.2. Instrument Operating Procedures

ESS RAD IOP 000	Table of Contents .doc
ESS RAD IOP 001	Berthold Alpha Beta.doc
ESS RAD IOP 002	Aptec Gamma Spectroscopy.doc
ESS RAD IOP 003	Packard LSC.doc
ESS RAD IOP 004	Ohaus Balances.doc
ESS RAD IOP 005	Octete-Plus Alpha Spectroscopy.doc
ESS RAD IOP 006	Gamma Products Alpha Beta.doc

16.3.6.3. Analytical Method Procedures

ESS RAD METHOD 000	Table of Contents.doc
ESS RAD METHOD 001	Alpha Beta SDWA.doc
ESS RAD METHOD 002	Alpha Beta CWA.doc
ESS RAD METHOD 003	Alpha Beta Gamma Air Filters.doc

ESS RAD METHOD 004	Lead 210 Sediment Dating.doc
ESS RAD METHOD 005	Uranium Thorium Alpha Spectroscopy.doc
ESS RAD METHOD 006	Efficiency Determination Gamma.doc
ESS RAD METHOD 007	Iodine131 Milk and Water.doc
ESS RAD METHOD 008	Polonium 210.doc
ESS RAD METHOD 009	Radium 226 and 228 SDWA.doc
ESS RAD METHOD 010	Radon Air.doc
ESS RAD METHOD 011	Radon SDWA.doc
ESS RAD METHOD 012	Strontium 90 Milk.doc
ESS RAD METHOD 013	Strontium 89 and 90 SDWA.doc
ESS RAD METHOD 014	Radium 226 Sludge.doc
ESS RAD METHOD 015	Tritium SDWA.doc
ESS RAD METHOD 016	Total Uranium Chemical.doc

16.3.7. Water Microbiology Department

16.3.7.1. General

ESS MIC GENOP 100	Opening Samples
ESS MIC GENOP 102	Checking-In Municipal Samples
ESS MIC GENOP 104	Checking-In Private Samples
ESS MIC GENOP 106	Chain of Custody Procedures
ESS MIC GENOP 108	Read-Outs
ESS MIC GENOP 400	Record Keeping Tasks and Sample Tracking for Samples with requests for Fluoride or Nitrate Analysis
ESS MIC GENOP 402	Record Keeping Tasks and Sample Tracking for Samples with requests for Iron Bacteria or SO4 Reducing Bacteria
ESS MIC GENOP 404	Reporting Procedure for Municipal Public Water Reports

16.3.7.2. Quality Assurance

ESS MIC QA 200	Temperature Monitoring and Calibration of Thermometers
ESS MIC QA 202	ONPG-MUG Quality (Colilert, Colilert-18 and Colisure)
ESS MIC QA 204	Media Sterility and Quality (Plate ube/Miscellaneous Media)
ESS MIC QA 206	Maintenance of Stock Cultures for QC
ESS MIC QA 208	Surface Water Temperature Check
ESS MIC QA 210	Membrane Filter Sterility
ESS MIC QA 212	Water Sample Bottle Sterility/Calibration/Fluoresence
ESS MIC QA 214	Quanti-Tray Sterility Check
ESS MIC QA 215	Measurement: Calibration of Pipets
ESS MIC QA 216	Verification Procedures
ESS MIC QA 218	Quanti-tray Sealer Check
ESS MIC QA 220	Conductivity Measurement for Laboratory Water (YSI Model 35 Method)
ESS MIC QA 222	Nitrate (NO3) and Ammonia (NH4) Levels in laboratory Water
ESS MIC QA 224	Heterotrophic Plate Count (HPC) for Laboratory Water
ESS MIC QA 226	Chlorine Check for Laboratory Water
ESS MIC QA 228	Technician Compare

ESS MIC QA 230	UV Sterilizer
ESS MIC QA 231	Verification of ESS MIC GENOP 10-tube MPN
ESS MIC QA 232	Inhibitory Residue Test
ESS MIC QA 234	Heavy Metals in Laboratory Water
ESS MIC QA 236	Distilled Water Suitability Test
ESS MIC QA 238	Thermometer Calibration
ESS MIC QA 240	Funnel Calibration

16.3.7.3. Analytical Methods

ESS MIC METHOD 300	Total Coliform/E. coli (Chromogenic / Fluorogenic Assay), Colilert, Colisure, Colilert-18 and Quanti-Tray Methods
ESS MIC METHOD 302	10-Tube Most Probable Number - Total Coliform
ESS MIC METHOD 304	Heterotrophic Plate Count (HPC)
ESS MIC METHOD 306	Iron Bacteria
ESS MIC METHOD 308	Sulfate Reducing Bacteria
ESS MIC METHOD 310	Basic Membrane Filtration Procedure
ESS MIC METHOD 312	Total Coliform Membrane Filtration (including fecal coliform analysis)
ESS MIC METHOD 314	Fecal Coliform Membrane Filtration
ESS MIC METHOD 316	Fecal Streptococci Membrane Filtration
ESS MIC METHOD 318	Enterococci Membrane Filtration
ESS MIC METHOD 320	E. coli Membrane Filtration
ESS MIC METHOD 322	Aeromonas Membrane Filtration
ESS MIC METHOD 324	Clostridia perfringens Membrane Filtration
ESS MIC METHOD 326	Sludge Testing
ESS MIC METHOD 328	ID of Total Coliform Using API 20E System
ESS MIC METHOD 330	Microscopic Analysis of Water Samples
ESS MIC METHOD 332	Oxidase Test
ESS MIC METHOD 334	Catalase Test
ESS MIC METHOD 336	Pseudomonas
ESS MIC METHOD 338	Glycol-Reducing Bacteria
ESS MIC METHOD 340	Detection of Total Coliform and E. coli from Ambient Air
ESS MIC METHOD 342	Bacteriophage Assay: FRNA Coliphage, Double Agar Layer Method
ESS MIC METHOD 344	Detection of E. coli O157 in Water Samples

ESS MIC METHOD 346	Detection of E. coli O157 in Fecal Samples
ESS MIC METHOD 348	Detection of Salmonella in Water Samples
ESS MIC METHOD 350	Detection of Salmonella in Fecal Samples
ESS MIC METHOD 352	EColite for Total Coliform/E.coli
ESS MIC METHOD 354	Clark's Presence/Absence for Total Coliform/E.coli

17. Test Methods for Accredited Parameters

17.1. Biomonitoring (WET)

17.1.1. Drinking Water — N/A

17.1.2. Non-Potable Water

Regulatory Method	Description	UWSLH Method
EPA/600/4-90/027F	Acute WET test for Ceriodaphnia dubia	ESS BIO METHOD 410
EPA/600/4-90/027F	Acute WET test for Pimephales promelas	ESS BIO METHOD 400
EPA 1002	Chronic WET test for Ceriodaphnia dubia	ESS BIO METHOD 430
EPA 1000	Chronic WET test for Pimephales promelas	ESS BIO METHOD 420
EPA 1003	Chronic test for *Selenastrum capricornutum*	ESS BIO METHOD 500

17.1.3. Solid & Chemical Materials — N/A

17.2. Inorganic Chemistry

17.2.1. Drinking Water

Regulatory Method	Description	UWSLH Method
EPA 150.1	pH, Electrometric	ESS INO METHOD 300.0
EPA 200.7	Inductively Coupled Plasma-Emission Spectrometry	ESS INO METHOD 400.2
EPA 200.9	Atomic Absorption Spectroscopy--Tl	ESS INO METHOD 400.5
SM3113 B	Atomic Absorption Spectroscopy	ESS INO METHOD 400.3
EPA 245.1	Mercury - Atomic Absorption	ESS INO METHOD 540.3
EPA 325.2	Chloride	ESS INO METHOD 141.0
SM 4500CN-E	Cyanide, Total	ESS INO METHOD 180.1
Technicon 380-75WE	Fluoride	ESS INO METHOD 220.9
EPA 353.2	Nitrate & Nitrite Nitrogen	ESS INO METHOD 220.9
EPA 375.2	Sulfate	ESS INO METHOD 370.3a
SM 2130 B	Turbidity, Nephelometric	ESS INO METHOD 380.3
SM 2510B	Automated Alkalinity, pH, and Conductivity	ESS INO METHOD 115.1
SM 2320B	Automated Alkalinity, pH, and Conductivity	ESS INO METHOD 115.1
SM 2540C	Total Dissolved Solids	ESS INO METHOD 320.1
SM 4500NO2B	Nitrite Nitrogen, Manual Colorimetric	ESS INO METHOD 220.8
SM 4500PE	Phosphorus, Reactive Dissolved (Orthophosphate)	ESS INO METHOD 310.3
SM 4500Si-F	Silica, Dissolved, Low Level	ESS INO METHOD 360.2

17.2.2. Non-Potable Water

Regulatory Method	Description	UWSLH Method
EPA 1664	Oil and Grease	ESS INO METHOD 250.4
EPA 200.7	Inductively Coupled Plasma-Emission Spectrometry	ESS INO METHOD 400.2
EPA 6010 B	Inductively Coupled Plasma-Emission Spectrometry (Sludge rule)	ESS INO METHOD 400.2
SM3113 B	Atomic Absorption Spectroscopy	ESS INO METHOD 400.3
EPA 200.9	Atomic Absorption Spectroscopy--Tl	ESS INO METHOD 400.5
SM 3111B	Potassium, Direct Aspiration AA	ESS INO METHOD 560.1
EPA 245.1	Mercury	ESS INO METHOD 540.3
EPA 1631	Mercury - Atomic Fluorescence	ESS INO METHOD 541.1
EPA 7470	Mercury (Sludge rule)	ESS INO METHOD 540.2
EPA 325.2	Chloride	ESS INO METHOD 141.0
SM 4500CN-E	Cyanides, **Total** & Amenable to Chlorination	ESS INO METHOD 180.1
EPA 351.2	Total Kjeldahl Nitrogen with Copper Sulfate	ESS INO METHOD 230.3
EPA350.1	**Ammonia** & Nitrate	ESS INO METHOD 220.3
EPA 353.2	Ammonia & **Nitrate**	ESS INO METHOD 220.3
EPA 375.2	Sulfate	ESS INO METHOD 370.3a
ASTM D1252-88B	Chemical Oxygen Demand	ESS INO METHOD 280.2
EPA 445.0	Chlorophyll	ESS INO METHOD 151.1
EPA 160.2	Total Suspended Solids	ESS INO METHOD 340.1
SM 2540C	Total Dissolved Solids	ESS INO METHOD 320.1
SM 2540E	Volatile Suspended Solids	ESS INO METHOD 340.1
SM 2540G	% Volatile Solids	ESS INO METHOD 330.2
SM 4500NH3F	Ammonia, Ion Selective Electrode	ESS INO METHOD 221.0

SM 4500NO2B	Nitrite Nitrogen, Manual Colorimetric	ESS INO METHOD 220.8
EPA 365.1	Total Phosphorus	ESS INO METHOD 310.2
SM 4500PE	Phosphorus, Reactive Dissolved (Orthophosphate)	ESS INO METHOD 310.3
SM 5210B	Biochemical Oxygen Demand (includes carbonaceous BOD)	ESS INO METHOD 260.1
SM 2340B	Hardness (Calculation Method)	ESS INO METHOD 200.2
SM 2320B	Alkalinity	ESS INO METHOD 115.1
SM 2540B	Total Solids	ESS INO METHOD 330.1
USGS I-1230-85	Chromium, Hexavalent	ESS INO METHOD 470.3

17.2.3. Solid & Chemical Materials

Regulatory Method	**Description**	**UWSLH Method**
EPA 1311	Toxicity Characteristic Leaching Procedure	ESS INO METHOD 715.1
EPA 6010 B	Inductively Coupled Plasma-Emission Spectrometry	ESS INO METHOD 400.2
EPA 7470/7471	Mercury, Solids	ESS INO METHOD 540.2
EPA 9045C	Soil and Sediment pH	ESS INO METHOD 295.0
EPA 9040B	pH, Electrometric, Hazardous Waste	ESS INO METHOD 301.0
EPA 9010B	Total Cyanide, Solids	ESS INO METHOD 180.2
EPA 9071B	Oil and Grease, Total Recoverable from Sediments and Sludges	ESS INO METHOD 250.5

17.3. Organic Chemistry

17.3.1. Drinking Water

Regulatory Method	Description	UWSLH Method
EPA 507	Determination of Nitrogen and Phosphorus Containing Pesticides in Water by Gas Chromatography with a Nitrogen-Phosphorus Detector	WSLH 1207
EPA 524.2	Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Mass Spectroscopy	WSLH 1325
EPA 552.2	Determination of Haloacetic Acids and Dalapon In Drinking Water By Liquid-Liquid Extraction, Derivatization and Gas Chromatography With Electron Capture Detection	WSLH 1253

17.3.2. Non-Potable Water

Regulatory Method	Description	UWSLH Method
EPA 608 & 608.2	Determination of Chlorinated Pesticides and PCBs in Wastewater and Storm Run-off Water by Gas Chromatography with an Electron Capture Detector	WSLH 1608
EPA 610	Polynuclear Aromatic Hydrocarbons in Wastewater by HPLC	WSLH 1611
EPA 624	Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Mass Spectroscopy	WSLH 1360
EPA 625	Base/Neutral and Acid Extractables in Priority Pollutants in Effluents by GC/MS analysis	WSLH 1670
SM5310C	Total Organic Carbon in Water	WSLH 1660

17.3.3. Solid & Chemical Materials

Regulatory Method	Description	UWSLH Method
EPA 1010 & EPA 9095	Flash Point - ASTM D93-85	WSLH 1720
EPA 1311	TCLP Analysis for VOCs	WSLH 1740
EPA 8141	Determination of Nitrogen and Phosphorous Containing Pesticides in water by Gas Chromatography with an NPD	WSLH 1241
EPA 8260	Volatile Organic Compounds in Soil by Purge-and-Trap using Capillary Column Gas Chromatography/Mass Spectrometry	WSLH 1551
EPA 8260	Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Mass Spectroscopy	WSLH 1360
EPA 8260	Volatile Organic Compounds in Waste and Waste Oils by Purge-and-Trap using Capillary Column Gas Chromatography/Mass Spectrometry	WSLH 1760
EPA 8270	Base/Neutral and Acid Extractable Priority Pollutants in Soils and Sediments by GC/MS analysis	WSLH 1570
EPA 8310	Polynuclear Aromatic Hydrocarbons in Soil and Sediment by HPLC	WSLH 1581
EPA 8315	Determination of Formaldehyde in Water by High Performance Liquid Chromatography	WSLH 1289
EPA 9060	Total Organic Carbon in Sediment by the Slurry Method	WSLH 1560

17.4. RadioChemistry

17.4.1. Drinking Water

Regulatory Method	Description	UWSLH Method
EPA 900.0	Gross Alpha	RAD 001
EPA 900.0	Gross Beta	RAD 001
EPA 901.1	Gamma Emitters	RAD 002
EPA 903.1	Radium 226	RAD 009
EPA 904.0	Radium 228	RAD 009
EPA 905.0	Strontium-89(calc)	RAD 013
EPA 905.0	Strontium 90	RAD 013
EPA 906.0	Tritium	RAD 015
EPA 908.0	Uranium	RAD 016

17.4.2. Non-Potable Water

Regulatory Method	Description	UWSLH Method
EPA 900.0	Gross Alpha	RAD 002
EPA 900.0	Gross Beta	RAD 002
EPA 903.1	Radium 226	RAD 014

17.4.3. Solid & Chemical Materials—N/A

17.5. Water Microbiology

17.5.1. Drinking Water

Regulatory Method	Description	UWSLH Method
SM9215B	Heterotrophic Plate Count	224
SM9221B	Multiple Tube Fermentation	302
SM9221D	Clark's Presence Absence	354
SM9221E	Fecal Coliform	312
SM 9222B	Membrane Filtration	312
SM9223/National Field - AEM	Colilert/Colilert -18	300
SM9223/ Federal Register	Colisure	300
SM9221F/Federal Register	*E.coli* (tube MUG) EC-MUG	312
Federal Register 12-1-99	E Colite Test *	352

17.5.2. Non-Potable Water

Regulatory Method	Description	UWSLH Method
SM9213F	*Psuedomonas aeruginosa*	336
SM9222D/EPA 600/8-78-017	Fecal coliform	314
EPA 600/8-78-017	Fecal streptococci	316
SM9230C	Enterococci	318
SM9213	*E.coli*	320

17.5.3. RCRA Solid & Chemical Materials — N/A

18. Qualifiers Used in the Sample Comment Field

18.1. Biomonitoring

- Lab accident – No data available
- SHW-synthetic hard water
- Dechlor- dechlorinated tap water
- Variability too high- test results may not be reliable
- Total residual chlorine ND- no detection
- Sample number RW- receiving water

18.2. Inorganic Chemistry

- A list of qualifiers used by Inorganic Chemistry can be found in Excel spreadsheet format at R:\Ehd\ESS (4900)\ESS Inorg (4910)\Admin\QC issues\LIST Qualifiers 2.xls.

18.3. Organic Chemistry Department

- Analysis not possible due to foaming.
- Aroclor identification is not possible—**.
- Calibration exceeds quality control limit—*C.
- Characteristic ions present below report limit—*CI. ***(urine study)***
- Coextracted interference indicated by *.
- Congener is detected above LOD in the lab blank—*B.
- Cyanazine amide is detected.
- Deethylatrazine is detected at _____ug/L. ***(for SDWA analysis)***
- Dry weight concentration is indeterminate—*E.
- Each vial contained a large air space—no work done ***(for SDWA analysis)***
- ____-day analysis holding time was exceeded.
- ____-day extraction holding time was exceeded.
- Extraction solvent was not added within 10 days.
- Fingerprint is not a perfect match to aroclor ____.
- Flash point cannot be determined on a solid.
- Hydrocarbon interference indicated by *.
- Insufficient sample—no work done.
- Interference indicated by *I.

- LOD not achievable due to dilution—*D.
- LOD unachievable due to instrument sensitivity—*S.
- Lab accident—no work done.
- Lab accident—no results reported—*A.
- Lab matrix spike lower (upper) QC limit exceeded—*LML (*LMU).
- Large air space in vials—rejected—mailer resent. ***(for SDWA Analysis)***
- Lower (Upper) QC limit for calibration check exceeded—*QL (*QU).
- Lower (Upper) QC limit for precision is exceeded—*QL (*QU).
- Matrix spike does not meet lower (upper) QC limit—*MSL (*MSU).
- Not received in WSLH vials-rejected-mailer resent. ***(for SDWA Analysis)***
- Not analyzed per _____'s instructions—*NA.
- Oxygenated hydrocarbons present.
- Peak splitting—estimated value indicated by *>.
- Possible matrix effect indicated by *ME. ***(urine study)***
- Possible vial contamination indicated by *V
- QC limit for precision is exceeded indicated by *QP
- Quantitation done per D. Grande's instructions — *DG
- Recovery of surrogate spike # ____(14, 65, 166) does not meet ____(lower/upper) QC limit.
- Report limit not achievable due to dilution—*D.
- Report limit not achievable due to foaming—**.
- Results are estimates due to sample matrix.
- Results not confirmed by an alternate technique.
- Sample container(s) received broken—no work done.
- Sample is <5% dry solids.
- Sample not collected in proper WSLH container.
- Sample not properly preserved—no work done.
- Sample not received at lab on ice—no work done. ***(for SDWA analysis)***
- Sample not valid—no work done.
- Sample passed the paint filter test.
- Sample vials contain large air space.

- Sample was acidified in the lab with HCl to pH<2.
- Sample was acidified with HCl (H_2SO_4) in the laboratory.
- Sample was not acidified with HCl to pH<2.
- Sample was received at ____ □ C.
- Sample weight exceeds 35.0 g—no work done.
- Sample weight that was analyzed was ____ grams.
- Standard deviation is ____%.
- TCLP not tested — sample could not be filtered. ***(Use in test O1740P4)***
- Teflon discs were improperly inserted in vials.
- Temperature was not taken upon arrival.
- The internal standard QC limit is exceeded — *IS
- This sample contains peaks (before) (after) the DRO window.
- Trip blank contains ____ at ____ µg/L.
- Trip blank data unavailable—analytical problems.
- Vials not iced; also large air space—no work done. ***(for SDWA analysis)***
- Water content is (greater/less) than 40% by weight.

18.4. Radiochemistry

Note: If the entire sample has been used and it has not been possible to meet the quality control criteria for the requested analysis, then an explanation memo is written for the client instead of placing qualifiers on the report.

18.4.1. SDWA

- Composite not acidified
- Sample received more than five days after collection
- Insufficient sample quantity
- Sample past holding time
- Laboratory accident, no test done

18.4.2. Radon Water Samples

- Sample received more than 4 days after collection
- Air bubbles
- Sediment present
- Laboratory accident, no test done

18.4.3. Radon air samples

- No closed house conditions
- Sample received more eight days after start of collection
- Kit past expiration date
- Non-standard collection interval
- Cap was loose
- Laboratory accident, no test done

18.5. Water Microbiology

- No test old sample
- Sample over 30 hours old when received.
- Bacteria test uncertain if over 48 hours old when received
- Results uncertain. Sample received warm
- Surface water exceeds 6 hour holding time, Sample tested within 6to 24 hours.
- Surface water exceeds 6 hour holding time. Sample tested within 24 to 48 hours
- No test chlorine in sample
- No test received frozen.
- No test shipping problem
- No test laboratory accident
- No test overgrown

19. Glossary

The following list of definitions has evolved over the years from a variety of sources. For more definitions please see the glossary section of the NELAC standards.

- **Accuracy** - A measure of the degree of conformity of a value generated by a specific procedure to the assumed or accepted true value, including both precision and bias.
- **Aroclor** - Monsanto trademark for polychlorinated biphenyls, designated by the percent total chlorination of the mixture.
- **Baseline** - The level of minimum response as determined by detector background signal level and noise.
- **Batch** - A group of samples which are carried through the preparatory and analysis procedures together. Generally it consists of nine samples, one duplicate, one method blank and one spiked or standard sample.
- **Bias** - A systematic error due to the experimental method that causes the measured value to deviate from the true value.
- **Blank** – There are a variety of different types of blanks used in the laboratory. the following is a breakdown of the various types:
    - **Reagent Blank** – A sample of a carrier agent (generally a liquid such as Milli-Q water, organic solvent or an acid solution) that is normally used to capture a material or analyte of interest. A reagent blank is subjected to all of the usual analytical processes.
    - **Method Blank** – A blank made up of the same matrix as the sample, and subjected to the entire analytical process (including digestion, filtration, column chromatography, etc.). Such a blank may also be referred to as a digested blank (metals), wash (continuous flow), or a filter blank.
    - **Calibration Blank** – Also referred to as "0" (zero) standard when analyzed with calibration standards. It is made up of Milli-Q water and acidified with the same acid matrix as the calibration standards. The calibration blank is a zero standard and is used to calibrate the instrument. NOTE: This definition applies exclusively to the Inorganic Chemistry Department.
    - **Continuing Calibration Blank** – A calibration blank that is measured at regular intervals throughout an analytical run to verify that the instrument calibration (baseline or zero concentration) has not changed. NOTE: This definition applies exclusively to the Inorganic Chemistry Department.
    - **System Blank** - A sample of a carrier agent (generally a liquid such as Milli-Q water, organic solvent or an acid solution) that is normally used to capture a material or analyte of interest. This sample is simply "injected" onto the instrument (not taken through the entire method) to monitor instrument performance.
    - **Field Reagent Blank** – Reagent water placed in a sample container in the laboratory and treated as a sample in all respects, including exposure to sampling site conditions,

storage, preservation and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

  - **Trip Blank** – Similar to the FRB above but the sample is not exposed to the sampling site conditions.

- **Blind Standard** – A standard or a sample from an external source with a validated concentration of analytes. The concentration of the analytes is known to the laboratory manager or Quality Assurance Officer.
- **Calibration Standard** – A solution prepared from the dilution of high purity standards (generally NIST traceable). The calibration standards are used to calibrate the instrument response to a known analyte concentration.
- **Check Sample** - A solution having a known value and obtained from inside the laboratory. It may be analyzed as a blind sample or as a check on the instrumentation.
- **Check Standard** – A standard with a known concentration of analytes. It is analyzed at a regular interval (every 10-20 samples depending on the method) to verify that the instrument is staying within calibration. Also known as a "Check" or "Gain".
- **Coefficient of Determination** – Denoted by "r-squared" it is the square of the correlation coefficient. Many software programs use this in place of "r".
- **Coefficient of Variation** – Relative standard deviation expressed as a percent, i.e., the standard deviation of a set of data expressed as a percentage of the mean.
- **Co-eluting Compounds** - Compounds exhibiting the same retention times.
- **Common Sense Detection Limit** – A limit of detection that is set using a common sense approach. The statistical LOD is used as a basis, but values may be adjusted higher depending on matrix effects, "real" instrument response and analyst experience. NOTE: No common sense detection limit may be lower than the statistical limit.
- **Confidence limit, 95 percent** - The limits of the range of analytical values within which a single analysis will be included 95 percent of the time (Note that in practice the constant '1.96' will be rounded to '2').

  95% CL = x plus or minus 1.96S

  where:

  CL = confidence level

  S = standard deviation

  x = mean

- **Continuing Calibration Verification (CCV)** – See Check Standard
- **Control Sample (1)** – A second source standard, independently prepared, with which the instrument performance and calibration standards can be verified. Also see Laboratory Control Sample and Second Source Standard.

- **Control Sample (2)** - A previously analyzed sample that is run with consecutive batches to determine relative recovery of parameters.
- **Correlation Coefficient** – Denoted by the letter "r" it is a statistically derived measure of the quality of a curve fit to a set of data points. It is used to decide whether a given calibration curve is valid.
- **Detection Limit** – See Limit of Detection
- **Digested Spike (DS)** – See Laboratory Fortified Blank.
- **Dissolved Constituents** – The constituents in a water sample that will pass through a 0.45 µm membrane filter.
- **Duplicate Sample** - A sample that has been homogenized, split, and sent through the entire preparatory and analysis procedure to determine reproducibility (precision) of results.
- **Error** – The difference between an observed or measured value and the best obtainable estimate of its true value.
- **Ghosting** - A gas chromatographic interference, showing as a peak, which appears at the same elution time (co-eluter) as a known component.
- **Initial Calibration Verification Standard (ICV)** – See Laboratory Control Sample.
- **Initial Demonstration of Capability (IDC)** – Procedure to establish the ability to generate acceptable accuracy and precision. The procedure consists of analyzing four to seven replicate samples prepared in the laboratory and taken through the entire method. The standard deviation, mean, mean recovery and percent relative standard deviation is determined and compared to the required values in the determinative method.
- **Instrument Performance Check Solution (IPC)** – See Check Standard
- **Interference** - An extraneous constituent (or constituents) in a sample which causes a bias in the analytical results.
- **Internal Audit** – An internal process conducted by the laboratory QA Officer and used to determine continuing compliance with established method SOPs and with external regulatory requirements.
- **Internal Standard** - A standard added to a sample extract just before GC analysis to determine relative retention time and quantitation for parameters of interest.
- **Laboratory Control Sample** – A check standard obtained from an independent source (other than the calibration standard) and made up in a laboratory matrix (e.g., Ottawa sand to simulate sediment). It is taken through the method just like a sample and used to determine the amount of matrix effect by comparing a "clean" matrix with a true sample matrix.
- **Laboratory Fortified Blank** – An aliquot of "blank" sample matrix (Milli-Q water, Ottawa Sand), to which known quantities of the method analytes are added. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements at the required MDL.

- **Laboratory Fortified Matrix** – See Matrix Spike.
- **Laboratory Performance Check Solution (LPC)** – See Check Standard
- **Limit of Detection (LOD)** – The minimum concentration of a substance which can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The LOD is typically determined from replicate analyses (minimum of seven) of natural or synthetic samples. The LOD is calculated using the following equation:

  LOD = s (t-value; 99%CL n-1 DF)
- **Limit of Quantitation (LOQ)** – The concentration of a substance above which quantitative results may be obtained with a specific degree of confidence (99% CL). Confidence in the apparent analyte concentration increases as the analyte signal increases above the LOD. The LOQ equals ten times the standard deviation of the LOD replicate analysis.
- **Linearity** - The range of concentration over which the detector maintains a constant sensitivity.
- **Lower Control Limit (LCL)** – The maximum allowable limit for a given QC result. Usually defined as the statistical mean minus three standard deviations (99% confidence limit). It applies to accuracy measurements. See also Upper Control Limit and Lower Warning Limit.
- **Lower Warning Limit (LWL)** – The point at which the analyst needs to be more concerned with the integrity of the analytical process. Usually defined as the statistical mean minus two standard deviations (95% confidence limit). It applies to accuracy measurements. See also Upper Warning Limit and Lower Control Limit.
- **Matrix** – See Sample Matrix and QC Matrix.
- **Matrix Spike** – A sample (preferably a real sample but may be a laboratory matrix) which is spiked with some or all of the method parameters and treated just as a sample would be. It is an accuracy measurement whose purpose is to determine whether the methodology is in control.
- **Matrix Spike Duplicate** – A second Matrix spike. The pair are used in lieu of a real sample duplicate (generally when analytes are not expected to be found in the real world) to gauge the precision of the method.
- **Mean (arithmetic)** - The mean (or average) of a set of n values is the sum of the values divided by n.
- **Method Detection Limit** – See Limit of Detection.
- **Minimum Sensitivity** - The lowest amount (mass) that produces a sample peak of ten percent full-scale deflection.
- **Noise** - An extraneous electronic signal that affects baseline stability.
- **Precision** - Relative to the data from a single test procedure, the degree of mutual agreement among individual measurements made under prescribed conditions.

- **Performance Sample** - A solution obtained from an outside source having a known value and analyzed as a blind sample or as a check on the instrumentation.
- **Quality Assurance** – The sum of all policies and activities which laboratory utilizes to ensure that quality control is carried out.
- **Quality Assurance Plan** – A document stating the quality policy, quality system, and quality practices of the organization. It may also be referred to as the Quality Assurance Manual.
- **Quality Control** – The actual implementation of Quality Assurance policies. The Quality Control program monitors the reliability (precision and accuracy) of results through a specific series of measurements. The QC program is designed to test the analyst, the analytical process, and the instrument performance by the use of quantifiable measurements.
- **Quality Control Audit** - A systematic review of analytical data at the bench level, conducted by an authorized chemist. The chemist will verify that proper Quality Control procedures were followed before any data may be reported.
- **Quality Control Matrix** – A group of samples with similar matrices that are grouped together for quality control purposes. (E.g., Sample Tap, Municipal Well, Private Well are all Drinking Water for QC purposes.)
- **Range (1)** - The difference between the highest and lowest values reported for a sample.
- **Range (2)** – A classification used to differentiate different concentrations from each other. Analyte results may be in a low concentration (range 1), mid-level concentration (range 2), or high level (range 3). Each level may have different quality control criteria associated with it.
- **Recovery (percent)** - A measure of the ratio, expressed as a percentage, of the amount of the determinant found to the true amount known to be present in the sample. This gives an indication of the presence or absence of interfering substances in a sample.
- **Reference Sample** – A sample used to determine accuracy acquired from a source other than the laboratory conducting the analysis, in which the true value and acceptance limits are unknown to laboratory at the time of analysis.
- **Relative Percent Difference** – The difference between measurements divided by the average, expressed as a percent.

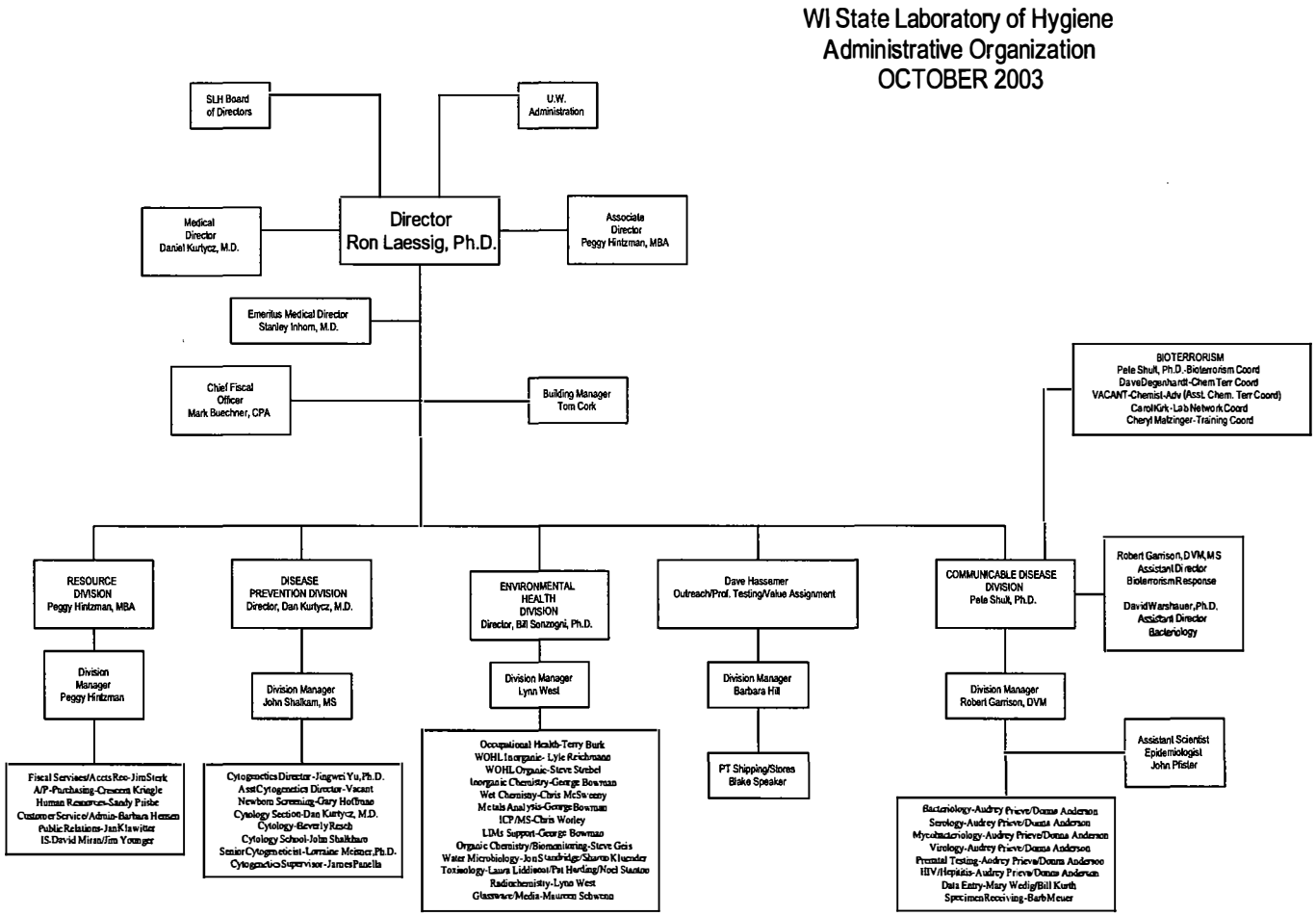
  RPD = (Difference/Average) x 100
- **Relative Response** - The ratio of responses of a given parameter to a reference parameter.
- **Relative Retention Time** - The ratio of retention times of a given parameter to a reference parameter.
- **Relative Standard Deviation** - The standard deviation of a set of data expressed as a percentage of the mean.
- **Report Limit** - The lowest level for a particular parameter determined to be significant. This is a subjective judgement based on analyst experience and instrument performance. The report limit is used as a minimum reportable concentration of an analyte when a statistically determined LOD has not been developed.

- **Resolved Peak** - A peak that begins and ends at the baseline.
- **Response** - The signal amplitude to mass ratio for a given parameter.
- **Retention Time** - The time that elapses from the introduction of the sample until the component's peak maximum is reached.
- **Sample Matrix** - The mechanical, physical and chemical properties of the sample. See also QC Matrix.
- **Sample Weathering** - The effects of the environment on individual components of a complex mixture (i.e., Aroclors, toxaphene, or technical chlordane).
- **Spiked Sample (spike)** – See Matrix Spike or Laboratory Fortified Blank. Also a generic term for any standard fortified matrix.
- **Standard Addition** – The method of standard addition is an analytical technique used to quantify samples whose matrices differ significantly form those of the standards. This technique is generally utilized in Atomic Absorption furnace analysis. It is accomplished by analyzing the sample mixed one to one with standards and blank, plotting the response vs. added concentration and extrapolating the x-intercept for the unknown concentration.
- **Standard Deviation** - The most widely used measure to describe the dispersion of a set of data about the mean. Normally $x + s$ will include 68 percent, and $x + 2s$ will include about 95 percent of the data from a study.
- **Standard Operating Procedure (SOP)** – A written document which details the method of an operation, analysis or action whose techniques and processes are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.
- **Standard Sample** - A previously analyzed sample run with consecutive batches to determine relative recovery of parameters. Also called a control sample.
- **Surrogate Spike** - A standard, not found in samples and chemically similar to the parameters of interest, that is added to each sample at the beginning of the analytical procedure to determine extraction/recovery efficiency for each sample.
- **Upper Control Limit (UCL)** – The maximum allowable limit for a given QC result. Usually defined as the statistical mean + three standard deviations (99% confidence limit). It applies to both precision and accuracy measurements. See also Lower Control Limit and Upper Warning Limit.
- **Upper Warning Limit (UWL)** – The point at which the analyst needs to be more concerned with the integrity of the analytical process. Usually defined as the statistical mean + two standard deviations (95% confidence limit). It applies to both precision and accuracy measurements. See also Lower Warning Limit and Upper Control Limit.
- **Unresolved Peak** - A peak that begins or ends on the shoulder of a neighboring peak.
- **Working Standard** - Standard solutions which may be directly injected for GC calibration.

20. Organizational Charts

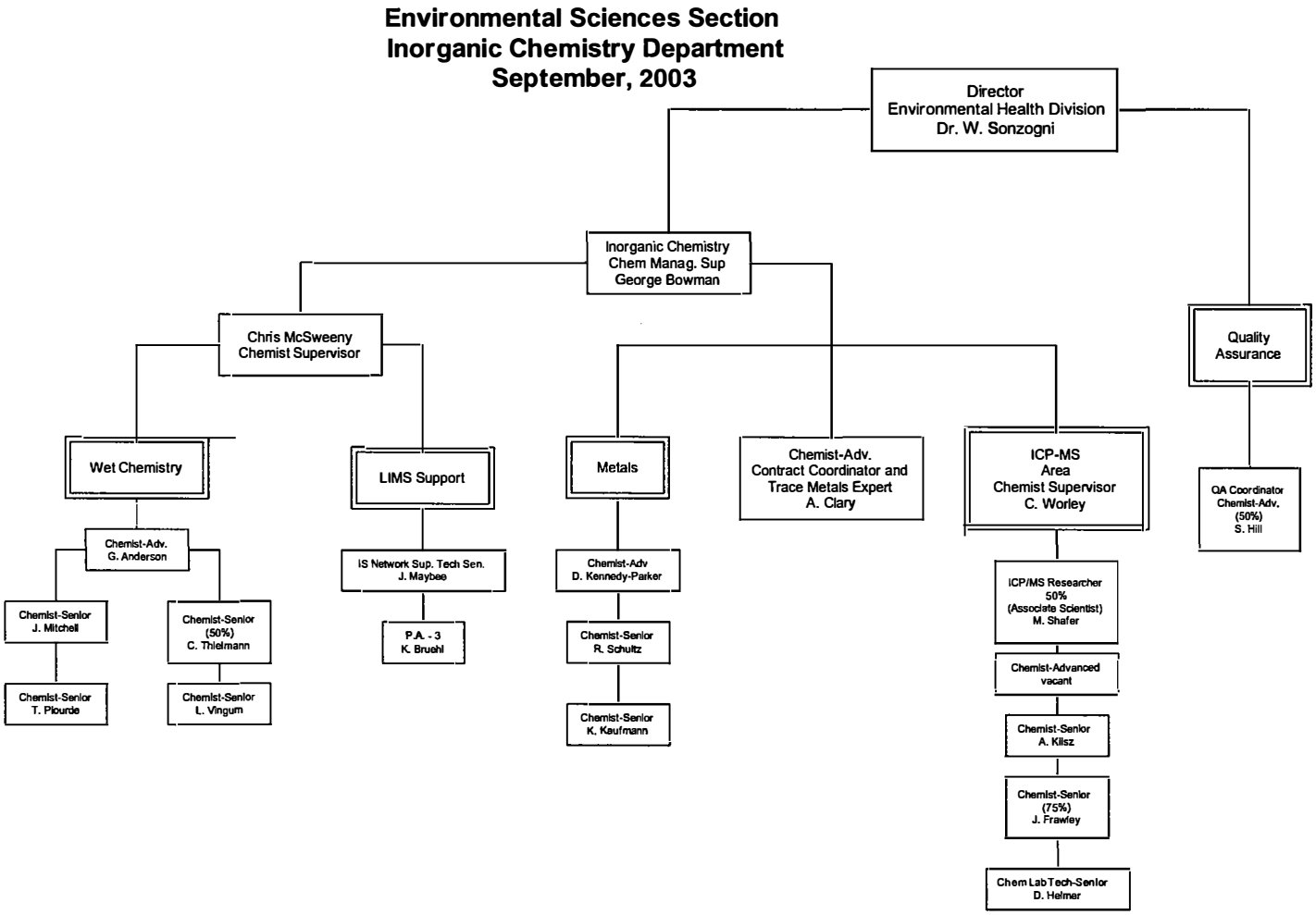
The following charts represent the organizational structure of the State Laboratory of Hygiene in general, and the Environmental Sciences Section in particular.

20.1. WSLH Structure



WSLH.flo

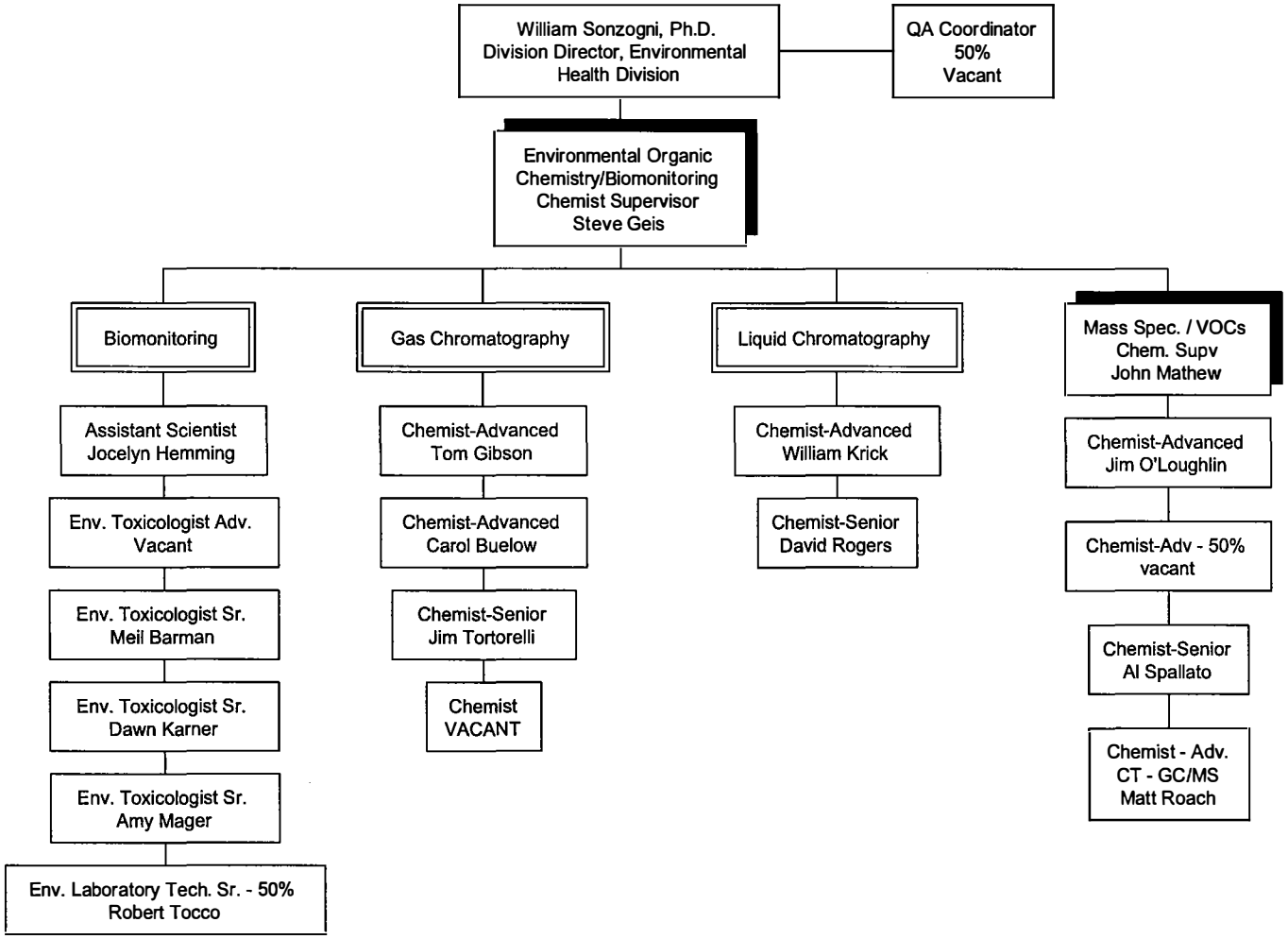
20.2. Inorganic Chemistry



20.3. Organic Chemistry & Biomonitoring Department

WISCONSIN STATE LABORATORY OF HYGIENE

ENVIRONMENTAL SCIENCES SECTION
ENVIRONMENTAL ORGANIC CHEMISTRY/BIOMONITORING
SEPTEMBER 2003

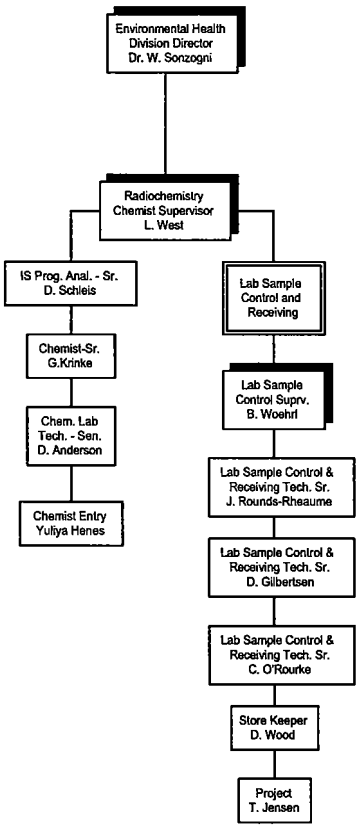


20.4. RadioChemistry

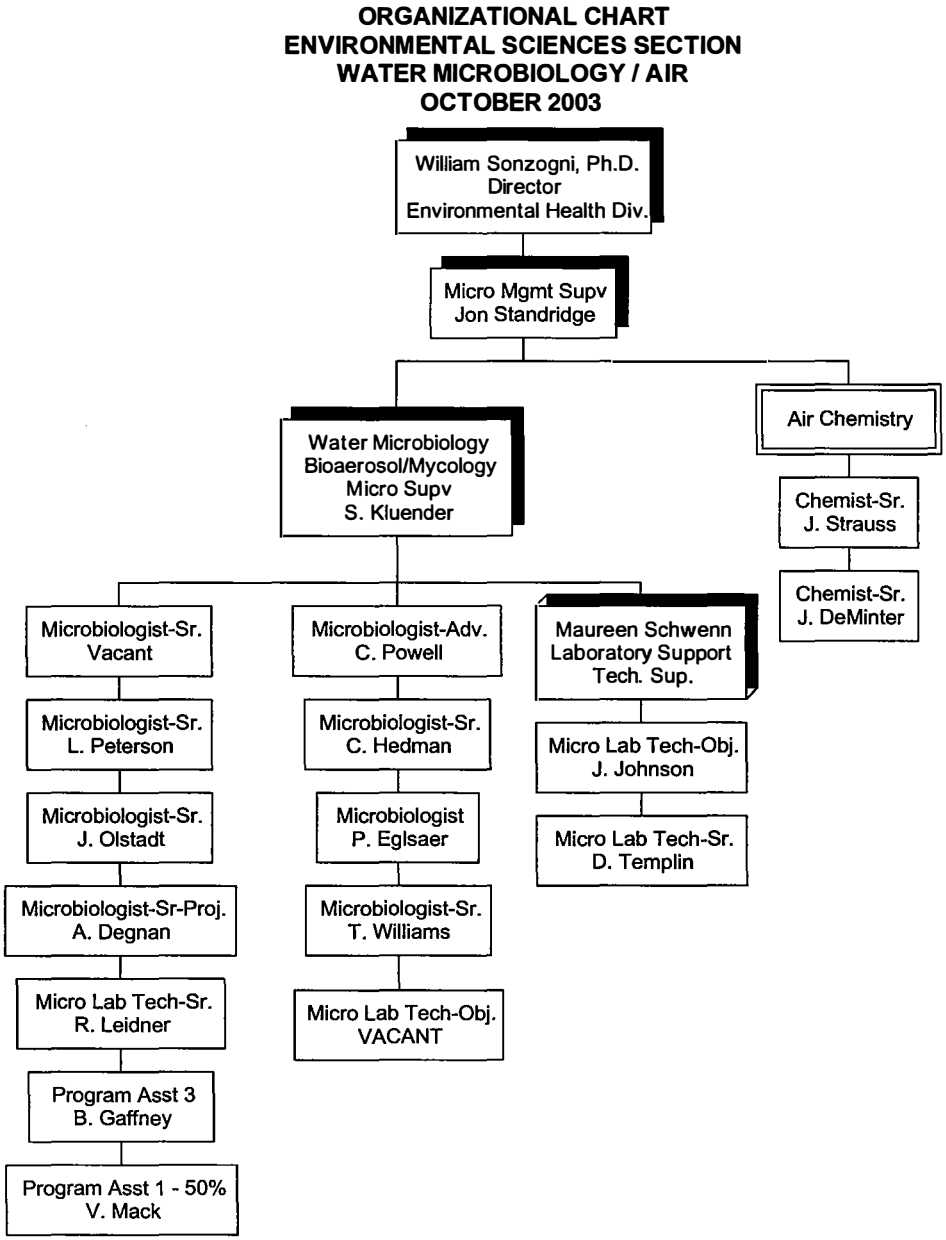
Environmental Sciences Section

RadioChemistry

January 2002



20.5. Water Microbiology



M:\HR\Orgcharts\H2O.Biomonitoring.flo

21. Quality Control Limits

21.1. Biomonitoring

See Chapter 12 for limits

21.2. Inorganic Chemistry

Limits are contained on-line and in MS EXCEL spreadsheets. They are readily available to all staff and will be included in Department copies of this document. However, because of their size (> one hundred pages) they are not included here.

21.2.1. Performance Samples

21.2.2. Check Samples

21.2.3. Blank Samples

21.2.4. Spike Samples

21.2.5. Duplicate Samples

21.3. Organic Chemistry

Limits are contained on-line and in MS EXCEL spreadsheets. They are readily available to all staff and will be included in Department copies of this document. However, because of their size (> one hundred pages) they are not included here.

21.3.1. Blanks, Checks and Performance Samples

21.3.2. Spiked Samples

21.3.3. Duplicate Samples

21.4. Radiochemistry

Limits are readily available to all staff and will be included in Department copies of this document. However, because of their size (> one hundred pages) they are not included here.

21.5. Water Microbiology

The acceptance criteria for total coliform and fecal coliform (*E.coli)* is the ability to find one coliform in 100 milliliters of water for drinking water samples (SDWA).

The quality control acceptance limits for Cryptosporidium

Performance test	Acceptance criteria
Initial precision and recovery Mean recovery (percent) Precision (as maximum relative standard deviation)	 24-100 55
On going precision and recovery (percent)	11-100
Matrix spike/matrix spike duplicate (for method modifications) Mean recovery (as percent) Precision (as maximum relative percent difference)	 13-111 61

Quality Control acceptance criteria for *Giardia*

Performance test	Acceptance criteria
Initial precision and recovery Mean recovery (percent) Precision (as maximum relative standard deviation)	 24-100 49
Ongoing precision and recovery (percent)	14-100
Matrix spike/matrix spike duplicate (for method modifications) Mean recovery (as percent) Precision (as maximum relative percent difference)	 15-118 30

The QC Acceptance Criteria for Method 1605 (*Aeromonas*)

QC Specification	Max. Acceptable Precision
Initial demonstration of capability (IDC): This test will require the analysis of 4 spiked reagent water samples.	RDS = 22%
Ongoing demonstration of capability (ODC): This test will require the analysis of 2 spiked reagent water samples.	RPD = 37%
Matrix Spike/Matrix Spike duplicate (MS/MSD) precision: This test will require the analysis of 2 spiked finished water (matrix) samples.	RPD = 48%

The State of Wisconsin

DEPARTMENT OF NATURAL RESOURCES

Hereby grants

Wisconsin Certification under NR 149

under the provisions of ch. NR 149, Wisconsin Administrative Code to:

US Filter/Enviroscan
301 West Military Road
Rothschild, WI 54474

Lab ID Number: **737053130**

Issued Date: **August 23, 2004**

Expiration Date: **August 31, 2005**

for the following test categories:

* Oxygen Utilization
  - Biochemical Oxygen Demand
  - Carbonaceous BOD
* Nitrogen
  - Ammonia as N
  - Nitrite as N
  - Nitrate as N
  - Total Kjeldahl Nitrogen
* Phosphorus
  - Orthophosphate
  - Total Phosphorus
* Physical
  - Oil and Grease (HEM)
  - Oil and Grease (Freon)
  - Total Dissolved Solids
  - Total Solids
  - Total Suspended Solids
  - Total Vol. Suspend Solids
  - Total Volatile Solids
* General I
  - Alkalinity/Acidity
  - Bromide
  - Chlorophyll a
  - Color
  - Hardness
  - Silica
  - Silicate
  - Surfactants
* General II
  - Chloride
  - Cyanide
* General II
  - Chemical Oxygen Demand
  - Fluoride
  - Total Phenolic Compounds
  - Sulfide
  - Sulfate
* General III
  - Corrosivity
  - EP Toxicity
  - Waste Fingerprinting
  - Ignitability
  - Total Releasable Cyanide
  - Reactivity
  - Total Releasable Sulfide
  - SPLP
  - TCLP
  - Total Organic Carbon
* Metals I
  - Silver
  - Aluminum
  - Arsenic
  - Boron
  - Barium
  - Beryllium
  - Calcium
  - Cadmium
  - Cobalt
  - Chromium (Total)
  - Copper
  - Iron
  - Chromium (Hexavalent)
* Metals I
  - Mercury
  - Potassium
  - Magnesium
  - Manganese
  - Molybdenum
  - Sodium
  - Nickel
  - Lead
  - Antimony
  - Selenium
  - Tin
  - Strontium
  - Thallium
  - Vanadium
  - Zinc
* Metals II
  - Gold
  - Lithium
  - Silicon
  - Titanium
* Organics; Purgeable
  - Acrolein & Acrylonitrile
  - Purgeable Aromatics
  - Glycols
  - Purgeable Halocarbons
  - Volatile Organics (VOCs)
* Semivolatiles by GC
  - Nonpurge Chl Hydrocarbons
* Semivolatiles by GC/MS
  - Base/Neutral/Acid Extract
* Semivolatiles by GC/MS
  - PAHs by GC/MS-SIM
* Liquid Chromatography
  - Aldehydes & Ketones by LC
  - PAHs by LC
* Pesticides
  - Acid Herbicides
  - Nitrogen Pesticides
  - Organophosphorus Pests.
  - Triazines and Metabolites
* Petroleum Hydrocarbons
  - Diesel Range Organics
  - Gasoline Range Organics
  - Petroleum VOCs
* Organics; Organochlorine
  - PCBs
  - Organochlorine Pesticides
* Safe Drinking Water
  - Acid Herbicides by GC
  - Arsenic
  - Barium
  - Beryllium
  - Cadmium
  - Chl. Hydrocarbon by GC
  - Cyanide
  - Chromium
  - Copper
  - EDB and DBCP
  - Fluoride
  - Mercury
  - Nitrate + Nitrite

David Webb

Chief, Environmental Science Services

P. Scott Hassett

Secretary

Certification or registration by the State of Wisconsin is not an endorsement or guarantee of the validity of data generated by this laboratory. This certificate is valid unless revoked or suspended and supersedes all previous certificates.

The State of Wisconsin

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Hereby grants

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US Filter/Enviroscan
301 West Military Road
Rothschild, WI 54474

Lab ID Number: **737053130**

Issued Date:

Expiration Date: **August 31, 2005**

for the following test categories:

- Safe Drinking Water
  - N/P Pesticides by GC
  - Sodium
  - Nickel
  - Nitrite
  - Nitrate
  - PAHs by HPLC
  - Lead
  - Antimony
  - Selenium
  - Sulfate
  - Thallium
  - Total Trihalomethanes
  - Volatile Organics
- Any Single Analyte
  - Malathion
  - Pentachlorophenol
  - Parathions

David Webb

Chief, Environmental Science Services

P. Scott Hassett

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QUALITY ASSURANCE
QUALITY CONTROL
MANUAL

for

USFilter Enviroscan Services

REVISION 5.0
Revised: 02/05/04

UNCONTROLLED COPY

INTRODUCTION

This Quality Assurance/Quality Control Manual is a description of USFilter, Enviroscan Services policy and procedures for maintaining an effective and comprehensive quality assurance program. This document is based on the requirements as set by the Wisconsin Department of Natural Resources (DNR), the United States Environmental Protection Agency (USEPA) protocols for environmental sample analysis, and the Chapter 5 standards as presented in the National Environmental Laboratory Accreditation Conference (NELAC) Quality Systems Manual, May 25, 2001.

USFilter, Enviroscan Services employs a full time Quality Assurance Manager to ensure that the laboratory meets the requirements as presented in the aforementioned guidelines and regulations.

USFilter, Enviroscan Services quality assurance program provides the basis to meet specifications of accuracy, precision, and comparability to assure that only data of the highest quality is reflected on our final reports. This commitment is made by Enviroscan Services, regardless of your sample matrix or regulatory requirements.

This document, its contents, and its commitments have been reviewed and approved by the following:

Director/Manager _____ Date: ____/____/____
Jim Salkowski

Technical Director _____ Date: ____/____/____
Bruce Schertz

Listed above are USFilter, Enviroscan Services' approved signatories. These individuals are permitted to verify and approve all documentation relevant to the analysis and/or reporting of samples analyzed by Enviroscan Services.

USFilter CORPORATE VISION

To provide customers worldwide a single, fully integrated resource to solve all of their water treatment problems.

Our MISSION STATEMENT

USFilter:
Drive a sustainable and dependable recurring revenue stream from an expanding installed base of core water and wastewater technologies

USFilter: Systems Group:
Become the customer-preferred supplier of integrated water & wastewater systems and solutions with annual revenue of $1 billion by 2003

USFilter: Systems Group: Zimpro Products/Enviroscan Services:
To be the top performing Business Center in support of the USFilter and Systems Group missions through leverage of our product expertise and our core competencies in:

Technical & Analytical ability
Piloting & Manufacturing capability
Process systems integration experience
Experience & expertise in HPI market
Market leadership in WAO

USFilter CORE VALUES

Customer Satisfaction

Profitability

Integration and Communication

Entrepreneurial Spirit

Enviroscan Services CODE OF ETHICS

To produce results within the guidelines defined by the Quality Assurance/Quality Control Manual, EPA and state methodologies, the National Environmental Laboratory Accreditation Program, and any other policies established by USFilter management.

To be honest and forthright in all job duties completed as an employee of USFilter, Enviroscan Services.

To aid our fellow employees so that scientific knowledge and development of professionalism has a nurturing environment in which to grow.

To render services to all clients in a manner that allows us to protect the confidentiality and proprietary rights of all parties involved.

To educate employees on the importance of ethics and ethical behavior in an analytical laboratory environment. Management will continue to remind all employees of their ethical and legal responsibilities through continuing education and training.

QUALITY ASSURANCE OBJECTIVES

USFilter, Enviroscan Services shall meet the requirements of the quality assurance program as outlined in this Quality Assurance/Quality Control Manual. The laboratory shall implement these quality procedures and maintain traceable analytical data. The QA/QC program, through corrective/preventative action and routine internal audits, shall be continually evaluated and improved upon. Reasonable precautions shall be taken to protect the client's sample from theft, loss, or damage. Management shall ensure close supervision of all operations and adequate training of all personnel.

TABLE OF CONTENTS

SECTION 1.0: DEFINITIONS OF TERMS

This section contains definitions for terminology often used in the quality assurance program at USFilter, Enviroscan Services. Words are in alphabetical order.

1.1 Definitions

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards.

Accuracy: A measurement close to the known true value which is assessed using reference samples and percent recoveries of check standards and matrix spikes.

Aliquot: A measured portion of sample taken for analysis.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques.

Analyte: Chemical element or compound of interest to the laboratory.

Analytical Batch: Composed of prepared samples (extracts, digestates or concentrates) and/or those not requiring preparation. A analytical batch con include samples originating from various matrices.

Analytical Sample: Solution or media introduced into an instrument for analysis, not including calibration.

Area Counts: Term used in gas chromatography indicating the peak area of a compound, which is proportional to the concentration of analyte in the sample.

Audit: A systematic check to determine the quality of some function or activity.

Batch: Samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

Blank: A sample introduced into the analysis process to check for contamination in sample collection, preparation, or analysis.

BNA: refers to the pH condition of the extractable compounds as base, neutral and acid.

Calibrate: To determine, by measure or comparison, the correct value of each reading or setting.

Calibration: Establishes the relationship between values indicated by a measuring device.

Chain of Custody (COC): Documents designed to trace samples from the time of collection through delivery to the laboratory for analysis.

Chlorinated Hydrocarbons: Organic compounds containing chlorine including a class of persistent, broad-spectrum insecticides that linger in the environment.

Coelution: The lack of separation between peaks eluting from a chromatographic column. The summation of the peaks are used for quantitation of analytes.

Colorimetric: Analyses based on the measurement of the color that develops during the test for a specific analyte. The intensity of the color development is usually measured at a specified wavelength on a spectrophotometer.

Confirmation: A second column or detector may be used to demonstrate that the compound was present in first analysis.

Corrective Action: Action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Demonstration of Capability (DOC): a procedure used to establish the ability of the analyst to generate acceptable data.

Dissolved Metals: Liquids which have passed through a 0.45 µm filter before analysis, usually field filtered.

Dry Weight: The weight of a sample after drying in an oven at a specified temperature, known as percent solids.

Gas Chromatography (GC): Organic compounds are identified by separation and quantification through a gas medium.

Gas Chromatograph/Mass Spectrometry (GC/MS): Organic compounds are fragmented and exit the GC as known ions.

Graphite Furnace (GFAAS): Metals are identified using an electrical current with a photo detection lamp.

Headspace: The area in a container not completely filled by the sample in which gases may collect.

Heavy Metals: Metals with high atomic weights. Examples of heavy metals are mercury, chromium, cadmium, arsenic, and lead. Heavy metals are not limited to only these five.

High Performance Liquid Chromatography (HPLC): Organic compounds are identified by separation using a solvent as the carrier medium.

Holding Time: The maximum time allowed between sample collection with preservation and analysis prior to analysis.

Inductively Coupled Argon Plasma (ICP): Metals are identified using extremely high temperatures with a photo detection lamp.

Inorganic: Chemical substance of mineral origin that does not contain carbon.

Instrument Tuning: A technique used in GC/MS procedures to verify proper instrument calibration.

Instrument Blank: A clean matrix, processed through the instrumental steps of the measurement process, used to determine instrument contamination.

Laboratory Control Sample: A sample matrix, free from the analytes of interest and other interfering analytes , spiked with a known amount of the analytes of interest. Used to demonstrate the laboratories ability to take a sample through all processing steps with acceptable performance.

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. Used to evaluate laboratory precision.

Leachate: Contaminated liquid resulting from water seeping through waste, agricultural pesticides or fertilizers.

Limit of Detection (LOD): Defined as the lowest concentration level that can be determined to be statistically different from a blank with 99% confidence. Limits of detection are matrix, method, and analyte specific.

Limit of Quantitaion (LOQ): Defined as the level above which quantitative results may be obtained with a specified degree of confidence. Mathematically defined as equal to 10 times the standard deviation of the results for a series of replicates used to determine a justifiable limit of detection.

Matrix: The component or substrate containing the analyte of interest. For batch type determination, the following matrix types shall be used:

Drinking water: Any aqueous sample that has been designated as a potable or potential potable water source.

Wastewater: Any aqueous sample collected prior to discharge into a body of surface water.

Groundwater: Any aqueous sample that is not designated as potable and is recovered from an underground well.

Water/Liquid/Other: Any aqueous sample not covered by the previous terms. May be grouped with waste or groundwater based on sample knowledge.

Soil/Solid/Sludge: Any matrices with >15% solids content.

Leachate: Any sample recovered from an approved leaching procedure or specifically designated as a landfill leachate.

Matrix Spike: Prepared by adding a known amount of analyte to a matrix specific sample. Evaluated by calculating percent recovery.

Matrix Spike Duplicate: A second replicate matrix spike used to obtain a measure of the precision of the recovery for each analyte.

Method Blank: A sample of a matrix similar to the batch of associated samples free of target analytes or interferences. Analyzed at a frequency of 1 per analytical batch.

Method Detection Limit (MDL): Defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Section 5.3.1 contains the equation for the MDL calculation.

National Environmental Laboratory Accreditation Conference (NELAC): A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP.

National Environmental Laboratory Accreditation Program (NELAP): The overall program, which establishes the guidelines for National accreditation.

Organic: Chemical compound containing carbon and hydrogen bonds.

PCBs: Polychlorinated biphenyls consisting of toxic chemicals used in transformers, capacitors, and gas pipelines.

Percent Recovery: A measure of accuracy that is calculated as the measured value relative to the true value.

Practical Quantitation Limit (PQL): The PQL is the lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory analysis. The PQL must be a level that can be supported at the lowest level of the instrument calibration range.

Precision: A measure of the ability to reproduce analytical results.

Preparation Batch: Composed of 1 to 20 samples of the same matrix, under identical operating conditions, within a 24-hour period.

Preservative: A chemical or reagent added to a sample to slow decomposition or degradation of a target analyte or physical process.

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Quality Assurance: maintenance and implementation of all quality control procedures in the laboratory to demonstrate to our clients that the reported data is accurate, dependable, and reproducible.

Quality Control: quality checks and procedures to ensure that our clients receive the highest quality product and services.

Quality Program: activities of quality assurance and quality control required to ensure the meeting of goals and objectives

Quantitative Analysis: A measured amount of analyte with a set degree of precision and accuracy.

Raw Data: Any original information from a measurement activity or study recorded that are necessary for the reconstruction and evaluation of the report of the activity or study.

Reagent Blank: A clean matrix consisting of all reagents, used in equal proportion, to those in the sample processing steps.

Resolution: The degree of separation between peaks needed to properly identify analytes.

Retention Time: A specific compound has a characteristic time for elution from a specific GC column. This information is used to determine the identity and amount of the analyte.

Reporting Limit: is an arbitrary number below which data is not reported. The reporting limit may or may not be statistically determined, or may be an estimate that is based upon the experience and judgment of the analyst.

Solid Waste: Waste that is of nonsoluble material which includes sewage sludge, agricultural refuse, demolition wastes, mining residues, etc.

Solvent: A substance capable of dissolving or dispensing one or more other substances.

Standard Operating Procedure: Written description of an established series of steps used in confirming the laboratory's performance.

Suspended Solids: Small pollutant particles that float on the surface of, or are in, a substance that resist removal by conventional means.

Technical Director: The person who has overall responsibility for the technical operations of the environmental testing laboratory.

Total Metals: Metals that are digested prior to analysis.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards through an unbroken chain of comparisons.

Validation: the process of substantiating specified performance criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

Work Cell: a well defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented.

SECTION 2.0: LABORATORY STRUCTURE AND SERVICES

USFilter, Enviroscan Services consists of an experienced staff and high quality instrumentation to ensure accurate analytical results. The lab is divided into primary work areas to prevent cross contamination of samples and reagents, to permit proper climate control, and to ensure security. The numerous laboratories combined encompass over a 7200 square foot area with approximately 36 feet of bench space per analyst.

2.1 Building and Equipment

The USFilter, Enviroscan Services building is climate controlled for the operation and maintenance of the analytical laboratory equipment. See Figure 2.1: USFilter, Enviroscan Services Laboratories and Offices for a layout of the main laboratory structure. Records are maintained and continually updated for each instrument. New instruments and equipment are acquired to add to the present capacity, or to expand into new areas based on our clients needs and government regulations. Specific laboratory equipment and instrumentation are covered in Sections 4.0 and 6.0.

2.2 Organization

The USFilter, Enviroscan Services organization is headed by USFilter's General Manager. See Figure 2.2: USFilter, Enviroscan Services Organizational Chart. Resumes of key personnel are in appendix A. Qualifications of additional personnel may be obtained upon request.

2.3 Personnel

USFilter, Enviroscan Services has a full time staff consisting of laboratory director, quality assurance officer, technical directors, professional chemists, analytical technicians, sales and support personnel. Each staff member is an integral part of the overall function of the laboratory.

Beginning with the Application for Employment, we gather the data to determine the abilities and educational background of a prospective employee. This data is verified through interviews of "qualified" candidates. Questionable data can be verified by our Human Resources Dept. by direct contact with listed References, college transcripts or contacting previous employers. Work assignments will be made based on the employee's overall interests and abilities.

Once employed, each member of the staff must repeatedly demonstrate sufficient education, training, technical knowledge and experience to fulfill the assigned task.

Training and cross training is encouraged at all levels and may include college courses, seminars, and in-house training programs. All personnel are expected to remain current in their field, i.e., professional journals, approved methodologies, and changing regulations.

All employees shall uphold the USFilter, Enviroscan Services Code of Ethics as written. All employees are assigned a *USFilter Employee Handbook*. The employee is then required to sign an affidavit attesting that they have read the handbook, that they understand the policies and procedures, and most importantly that they will comply with these policies and procedures. Under the corporate Compliance Program, the first purpose is to "*Sustain a culture where ethical and legal conduct is recognized, valued, and exemplified by all of our employees*". The program details examples of unethical or illegal activities and also gives a suggested list of questions that each employee should ask himself or herself to test ethics or legal issues.

The Compliance Program also clearly states that disciplinary action will be imposed for 1) actual violations; 2) ignoring and not reporting violations; 3) failing to reasonably detect violations (managers); 4) refusal to cooperate with an investigation; and 5) retaliation. Employees may report any violations or suspicions of violations to their supervisors or managers. They can also use the Compliance Hotline 888-773-2514 if they feel they cannot safely report a violation to the local management.

On-going ethics education and training will be done in a number of different ways. These include the USFilter intranet site, training sessions, computer-based training, periodic ethics updates and distribution of all new or revised policies to all employees.

Job Descriptions and Requirements by Position

2.3.1 Laboratory Director

The laboratory director is in charge of the internal organization of the laboratory and is responsible for supervising all laboratory personnel employed by USFilter, Enviroscan Services. The lab director designates the lines of responsibility in the laboratory. This designation is documented and demonstrates the responsibility, authority, and interrelationship of all personnel who supervise, perform or approve work affecting the quality of data.

Management helps develop, implement, and enforce quality control and quality assurance practices through the use of an annual review of the quality system. It is through this annual review that the laboratory managerial and supervisory personnel review new work and introduce any necessary changes or improvements in the quality system or laboratory operations.

The laboratory director is responsible for the development of a proactive program for prevention and detection of improper, unethical or illegal actions. The QA/QC officer will assist in the development of this program by offering internal proficiency testing samples and conducting internal audits.

* In the event of a prolonged absence by the laboratory director, the Inorganic Chemistry Technical Director has been nominated as deputy laboratory director.

2.3.2 Quality Assurance/Quality Control (QA/QC) Officer

It is the responsibility of the QA/QC officer to develop, implement, and enforce quality assurance and quality control practices and procedures. The QA/QC officer directs the development, implementation and enforcement of quality assurance and quality control practices. QA/QC personnel have unrestricted access to the general manager of USFilter, Enviroscan Services. At this level the appropriate quality oriented decisions are made without productivity or financial pressures.

The appointed QA/QC officer must be experienced and knowledgeable in quality systems standards, and possesses a working knowledge of the analytical methods. With this knowledge, he or she will manage lab-testing protocol, ensuring accuracy through internal systems tests and performance audit data review.

The QA/QC officer functions independently from the laboratory and is able to evaluate data and perform assessments without influence from the laboratory or technical directors. The QA/QC officer performs no daily analytical testing in the laboratory except out of necessity. The QA/QC

officer is the focal point for all QA/QC procedures and questions. QA is in daily contact with the laboratory and technical directors, analysts, and technicians.

The QA/QC officer is responsible for documenting and verifying that the analyst(s) have completed a demonstration of capability study for the testing for which they are responsible or the parameters for which they provide back-up support. This procedure is accomplished by closely tracking all performance sample results, internal blind sample results and demonstration of capability studies. A table is used to track each analyst and the testing they perform. When the analyst completes a DOC, they are granted permission to begin, continue, or resume performing that analytical procedure.

The QA/QC officer keeps employee job descriptions and training records accurate and up-to-date on file in the QA Office.

In the event of a prolonged absence by the QA/QC Officer, the laboratory director is nominated as deputy QA/QC officer until a replacement can be found.

2.3.3 Technical Directors

The laboratory is divided into two technical units based on the appropriate fields of testing. These units are designated as Organic and Inorganic Chemistry units. The technical director for each unit is a full-time member of the analytical staff and performs day-to-day supervision of laboratory operations in that unit as well as daily analytical testing. The technical director is the immediate contact for issues concerning that unit of the laboratory.

If the technical director is absent for 15 consecutive calendar days, the deputy technical director as named below, will be responsible for his duties during the absence period. In the event that the absence exceeds 65 days, a permanent replacement will be made and all accrediting authorities will be notified in writing of his replacement. The technical director must possess a Bachelors of Science degree in a chemical or biological field and have at least two years of experience in the analysis of analytes for which he/she is named.

The technical director is in charge of the technical operation of the unit of the laboratory they oversee. The technical director shall certify that personnel have the appropriate education, technical background or adequate training to perform the tests they are assigned. This shall be documented through acceptable performance on the initial and continuing demonstration of capability studies.

At this time, USFilter designates Bruce M. Schertz the Inorganic/Organic Chemistry Technical Director.

At this time, USFilter designates James R. Salkowski the Director/Manager.

In the event of prolonged absence by the Inorganic Chemistry Technical Director, the Laboratory Director will fulfill the duties of deputy technical director until the position is officially filled.

In the event of prolonged absence by the Organic/Inorganic Chemistry Technical Director, the Director/Manager will fulfill the duties of deputy technical director until the position is officially filled.

2.3.4 Analysts and Technicians

Analysts and Technicians are directly responsible for physically performing the test in accordance with the standard operating procedures. Analysts and Technicians work together as a team to generate a result for the testing required.

Any employee hired by USFilter, Enviroscan Services to fulfill the role of analytical chemist, organic chemist or inorganic chemist must possess at a minimum, a bachelors of science degree in chemistry or science related field. This requirement can be replaced if the potential employee possess a minimum of 2 years experience in a related profession.

Any employee hired by USFilter, Enviroscan Services to fulfill the role of analytical technician or inorganic chemistry technician need not possess any prior laboratory knowledge or experience. The conditions of employment rely solely on the person's ability to follow the standard operating procedures and complete the demonstration of capability requirements.

All personnel are responsible for adhering to approved methods and procedures, as well as, maintain quality control requirements that pertain to their technical function. These requirements are found in the Wisconsin Administrative Code - NR149, NELAP Chapter 5 Quality Systems and this QA/QC manual. Each staff member must demonstrate knowledge of their particular function and a general knowledge of laboratory operations, test methods, QA/QC procedures and data records management by successfully completing an internal audit and analyzing a performance evaluation (PE) sample for each area of testing they are assigned to. Once a year, the analyst is required to review the latest revisions of the analytical method, standard operating procedure, QA/QC manual, NR149, and NELAP Chapter 5. This will be officially documented by the QA/QC officer.

2.4 Services

USFilter, Enviroscan Services is a full service, certified environmental analytical laboratory which performs most environmental chemical analyses associated with water, wastewater, soils and sludge. Analyses shall adhere to USFilter, Enviroscan Service's Quality Assurance Program.

2.5 Capabilities

USFilter, Enviroscan Services has capabilities to analyze a number of organic, inorganic and synthetic compounds. See the USFilter, Enviroscan Services - Environmental Guidance Document for a list of analytes and the associated reporting limits.

2.5.1 Regulatory Programs

USFilter, Enviroscan Services participates in various regulatory programs including:

- CWA (Clean Water Act)
  - Wastewater Monitoring
  - Stormwater
  - Compliance Monitoring
  - Discharge Monitoring
  - Municipal sludge
  - NPDES permits

- CERCLA (Comprehensive Environmental Response, Compensation and Liability Act)
  Superfund, SARA (Superfund Amendments & Reauthorization Act)
  CLP-like testing and reporting
  Target compound list analysis
- RCRA (Resource Conservation and Recovery Act)
  TCLP and SPLP leaching procedures
  Groundwater monitoring (Landfill and Industrial)
  Landfill acceptance protocols
  Hazardous Waste Management
  Waste Characterization Analysis
  Ignitability
  Reactivity
  Corrosivity

- UST (Underground Storage Tanks)
  Tank Remediation
  Diesel Range Organics
  Gasoline Range Organics
  Petroleum Volatile Organic Compounds
  Polynuclear Aromatic Hydocarbons

- SDWA (Safe Drinking Water Act)
  Municipal Water
  Potable Water
  Well water for new and existing wells
  Primary and secondary pollutants

- TSCA (Toxic Substance Control Act)
  PCBs

- NPDES (National Pollutant Discharge Elimination System)
  Discharge Permit
  Wastewater
  Industrial Effluents
  Total Toxic Organics

2.6 Certifications

USFilter, Enviroscan is accredited by the National Environmental Laboratory Accreditation Program (NELAP) with certification in Wisconsin, Indiana, Michigan, Minnesota, Massachusetts, Maryland, Montana, Oklahoma and Washington.

National Environmental Laboratory Accreditation Program (NELAP)
Oregon Environmental Laboratory Accreditation Program
Public Health Laboratory
Organic, Inorganic, Metals Testing

CWA, SDWA, RCRA
ID# ORELAP-WI-100001

State of Wisconsin Certification
Wisconsin Department of Natural Resources
Category 1-16, 19 and Safe Drinking Water
Laboratory ID #: 737053130

State of Louisiana
Department of Environmental Quality
Organic, Inorganic, Metals Testing
CWA, SDWA, RCRA
AI #88968 LELAP Cert # 04026

Minnesota Certification
Safe Drinking Water Organics and Inorganics
Clean Water Organics and Inorganics
Resource Conservation & Recovery Program Organics and Synthetics
Laboratory ID #: 055-999-302Petro Fund # 1467

Maryland Certification
State Certified Water Quality Laboratory
Metals 1,2; Inorganics 1,2,3,5; Pesticides 1,3,4(Screen Only);
Herbicides; THM; VOC 1,2
Certification #: 276

The Commonwealth of Massachusetts
Department of Environmental Protection
Certified for the chemical analyses of potable and non potable water
Laboratory ID# M-WI006

Oklahoma Department of Environmental Quality
Certified for General Water Quality/Sludge Testing
Laboratory ID No.: 9925

State of Washington Department of Ecology
ACCREDITED LABORATORY
Non-potable water for the following parameters
Inorganics, Metals, and Organics

State of Indiana
Indiana State Department of Health
Certified for the chemical analyses of potable water
Laboratory number C-WI-01

Montana Approved List
For the following parameters:

DRO; DRO as Diesel; DRO as TEH; BTEX; MTBE; GRO as Gasoline; GRO as TPH

2.7 Memberships and Professional Affiliations

USFilter, Enviroscan Services is a member of the following organizations:

-
- Wisconsin Environmental Laboratories Association (WELA)
- American Water Works Association (AWWA)
- Water Environment Federation (WEF)
- Federation of Environmental Technologies (FET)
- Central Wisconsin Quality Improvement Network (CWQIN)
- Central Wisconsin Groundwater Association
- Wisconsin Wastewater Operator's Association
- Marathon County Solid Waste Committee

FIGURE 2.1: USFILTER, ENVIROSCAN SERVICES - LABORATORIES AND OFFICES

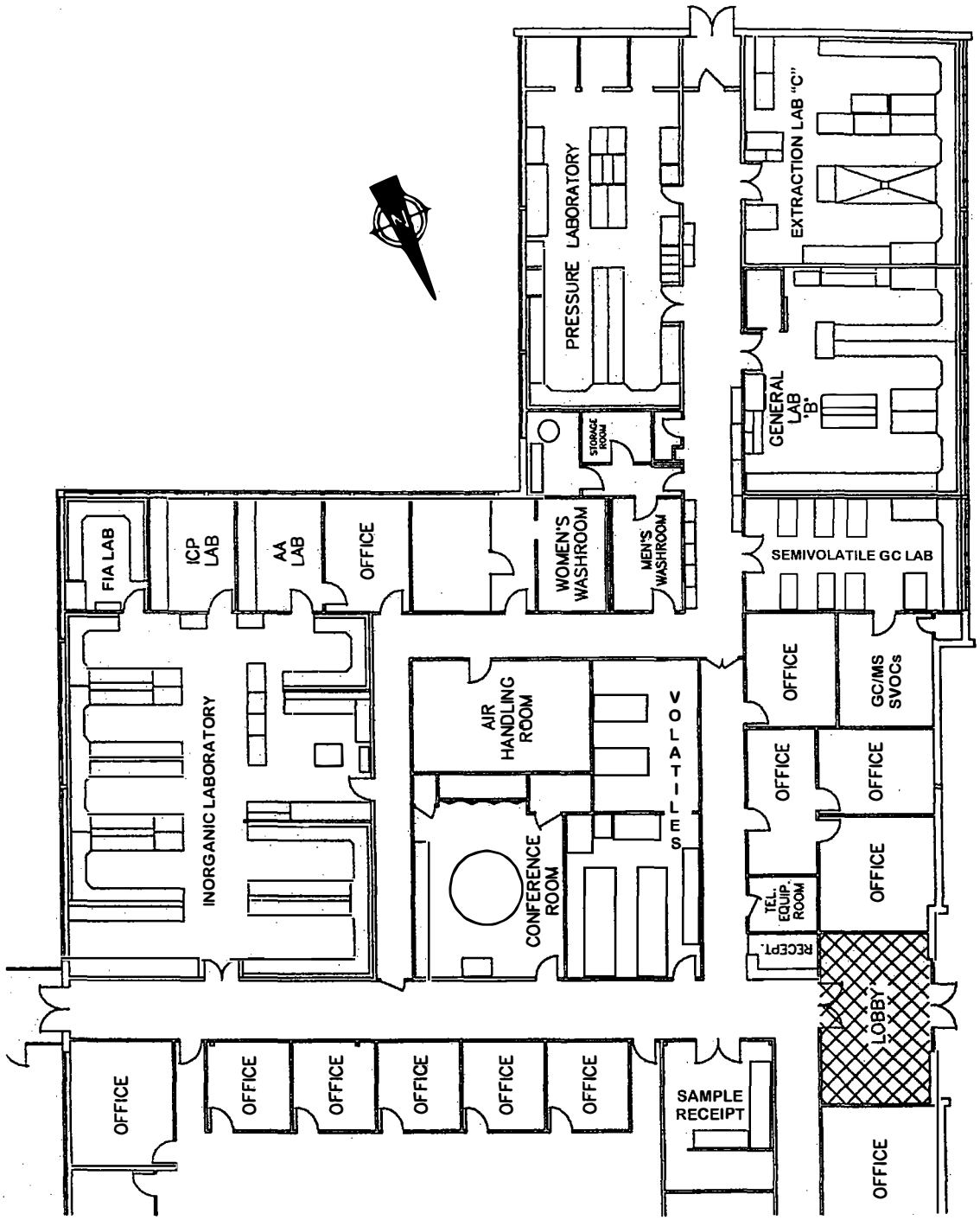


FIGURE 2.2 USFILTER, ENVIROSCAN SERVICES - ORGANIZATIONAL CHART

FIGURE 2.3 USFILTER, ENVIROSCAN SERVICES – LABORATORY STAFF - *2004*

Employee Name,Title	Hire Date	Education	Responsibilities/Duties
Administrative Services			
Jim Salkowski, Laboratory Director	09/76	M.S., Chemical Limnology	Oversees Laboratory operations
Lois Sirvio, Secretary	06/75	B.A., Education	Assists staff
Bruce Schertz, Technical Director	05/91	B.S., Chemistry	Inorganic/Organic Supervision, ICP
Marketing and Sales			
Sara Opper,Sales Manager	04/03	B.S. Biochemistry	Pursues new work
Susan Anderson, Sample Receiving	09/97		Sample custody and receipt
Jim Waelchli, Client Service Manager	2/04	B.S. Chemistry	Oversees Client Service
Quality Assurance			
Cindy Varga	07/90	B.S., Chemistry	Oversees QC program
Inorganic Laboratory			
Jay Hunger, Senior Chemist (Metals)	06/91	B.S., Chemistry	GFAAs, CVAAs Mercury
Dominic Bush, Senior Chemist (Metals)	11/90	B.S., Water Chemistry	Metal Preparation, ICP
Christina Foster, Analytical Chemist (Inorganic)	5/03	B.S. Chemistry	Anions by IC, GFAAs
Lynne Kamke, Group Leader	08/75		BOD, FIA,Titration,Autoanalyzer
Gloria Gerstl, Analytical Technician	03/94		FIA, ISE,Distillation
Joyce Patterson, Analytical Technician	04/00		TCLP, Carbon Analyzer,Titration
Barb Verhey, Analytical Technician	9/03	12 years analytical	Metal Preparation, Solids,Titration
Staci Kamke, Anlaytical Technican	5/03		Spectrophotometer, Solids, Titration
Organic Laboratory			
Jason McCann, Chemist (GC)	09/02	B.S. Biology	GC/ECD
Peg deLong, Senior Chemist (GC/MS)	08/93	B.S., Medical Technology	Semivolatile Organic Mass Spec.
Mike Melotik, Senior Chemist (GC/MS)	05/94	B.S., Biology	Volatile Mass Spec.
Laurie Pietrowski, Senior Chemist (GC)	06/93	B.S., Biology	Volatiles GC, GC/FID,NPD,HPLC
Eric Lorge, Senior Chemist (GC)	09/02	M.S. Organic Chemistry	GC/ECD
Elizabeth Ziebell, Analytical Technician	11/97		Volatiles Lab Technician
Deanna Oertel, Analytical Technician	10/99		Volatiles Lab Technician
Jennifer Gerstl, Analytical Technician	04/93		Extraction Tech, Sample Prep
Kathy Mundl, Analytical Technician	09/97		Extraction Tech
Mary Gritzmacher, Analytical Technician	02/98		Extraction Tech, Sample Prep
Julia Sirvio, Analytical Technician	11/01		Extraction Tech

SECTION 3.0: SAMPLE PROCEDURE

USFilter, Enviroscan Services is a full service certified environmental laboratory. The laboratory's services include providing sampling kits to our clients along with sampling instructions; and taking the necessary steps to maintain traceability of each sample throughout the laboratory's analytical process to the clients' final report.

3.1 Parcel - Receiving

USFilter, Enviroscan Services receives samples by commercial carrier (Federal Express, USPS, courier service, etc.) or by delivery in person.

3.1.1 Commercial Carriers (i.e. Federal Express, USPS, UPS, etc.)
Packages are received by the Shipping/Receiving Department personnel who are responsible for verifying the accuracy of the shipping form, examining the integrity of the container(s), notifying Log-in personnel of any discrepancy, and delivering the shipment to the log-in area as soon as possible.

3.1.2 Courier Service (i.e. Dunham, City Delivery, etc.) or in person.
Log-in area personnel located near the front entrance receive all packages. Chain of custody (COC) forms are signed and dated accepting transfer of samples.

3.2 Log-In Area (Figure 3.1: Sample Flow Chart)

The log-in technician is responsible for providing the client with sample kits for collection and for introducing samples from the client into the laboratory's analytical process. Log-in personnel sign the COC verifying the condition of each sample container for proper analyses. A unique sequential analytical number is assigned to each sample.

3.2.1 Sample Kit Requests, Form 3.1

USFilter, Enviroscan Services provides sample bottles with preservative, if needed, along with COCs or Analytical Request form(s). Requests for bottles may be made directly to the log-in area or via a customer service representative or appropriate personnel. The Sample Bottle Kit Request, Form 3.1, is properly completed and submitted to the Log-in technician to prepare the order for shipment. Standard items included in a sample kit are as follows:

a. Sample bottles with preservation identifier labels, if required.
b. Sample label for each bottle.
c. Return address label
d. Chain of custody form
e. Sampling and shipping instructions, if applicable.
f. Temperature blank
g. Trip blank for VOC analysis.

Custody seals and instructions for sampling are included with each shipment of supplies provided by USFilter. Instructions include all procedures for WATER sampling, SOIL sampling, Lead and Copper analysis, Preservation, and bottle types needed, see Appendix B for detailed sampling instructions. Each sample kit includes extra sample containers to be used for quality control procedures. This requirement is only applicable when the client is requesting semi-volatile extractable

analysis. If the QC requirements are not fulfilled for your sample set, USFilter, Enviroscan Services is obligated to qualify sample data as not meeting minimum QC requirements.

3.2.2 Chain of Custody (COC), Form 3.2

The COC and/or sample request sheet(s) provided by USFilter Enviroscan Services contain significant space to record the following information:

- Client's name, shipping address, and contact person
- Collection date and time
- Client's sample identification
- Number of containers
- Sample description (environmental matrix)
- Analysis requested for each sample
- Spaces to be signed as custody is transferred

3.2.3 Receiving Sample Containers

Sample container(s) along with a chain of custody (COC) are received by Log-in personnel to initiate the analytical testing process. All samples submitted to USFilter, Enviroscan Services must meet a set of minimum acceptance requirements before USFilter will take possession. To assure that your sample meets the minimum requirements, make sure that the sample shipment is accompanied by the appropriate information for the laboratory to assess at the time of receipt. At the time of receipt the laboratory will perform a documentation and preservation review that encompasses the following checks.

3.2.3.1 Documentation Review

1. Does a Chain of Custody document accompany the sample shipment? If not, is sufficient documentation included that will allow USFilter to accurately identify the analysis being requested?
2. Do any sample containers show signs of damage, breakage, leakage, contamination, or tampering?
3. Are all sample bottles clearly labeled allowing USFilter indisputable identification of the sample aliquot?
4. Is the date and time of collection clearly labeled on the sample bottle?
5. Can the bottles identified be traced back to the chain of custody?
6. Were the appropriate sample containers provided by the client for the tests requested?
7. Do the sample containers provided meet the preservation requirements as listed in the USFilter Sample Receipt Policy?
8. Is sufficient volume provided for USFilter to perform the requested analysis in compliance with method or agency specific requirements?
9. Is sufficient time allowed for USFilter to complete the analysis and adhere to the method specified holding times?

If any of the previous requirements are not met and the deviation will have an affect on the final results, USFilter, Enviroscan Services will attempt to obtain the necessary information from the client contact as listed on the chain of custody. If a satisfactory resolution is not achieved, the requested analyses will be canceled. If the client consents to the analysis, it is the legal responsibility of USFilter, Enviroscan Services too unambiguously flag all data associated with the failing criteria. The log-in representative is responsible for recording all

the deviations on a sample receipt report and including it with the final hard-copy report. These steps shall be taken by USFilter to sufficiently document and report the acceptance criteria variances.

3.2.3.2 Preservation Review

To determine if a sample meets the minimum receipt requirements, the laboratory must verify that thermal and chemical preservation, when required, are met. The laboratory will demonstrate this by performing the following checks and record all relevant information:

1) If thermal preservation is required, the sample receipt temperature must be between 0 and 4°C (WI) or 0 and 6°C (NELAP). If hand delivered the samples must be noted as "received on ice" and the receipt temperature recorded.

   The temperature of the sample container will be measured using an infrared thermometer gun and recorded on the associated paperwork by performing one of the following:

   a. Samples hand delivered within 15 minutes of collection shall be noted as such.
   b. Measurement of the Temperature Blank, if provided.
   c. Measurement of one of the sample containers. The sample container selected for measurement must contain at least 50ml of liquid.

   If the previous thermal preservation requirements are not met, USFilter, Enviroscan Services will attempt to contact the client for further instructions. Ultimately, it is the clients' decision as to whether or not the laboratory is to proceed with analysis. However, it is the legal responsibility of USFilter, Enviroscan Services too unambiguously flag all data associated with the failing criteria. A sample receipt report must be prepared clearly explaining the deviations.

   Drinking water samples that arrive at USFilter, Enviroscan Services that do not meet the thermal preservation requirements will be rejected. It is then the client's choice to continue the analyses outside of the regulated parameters. This approval will be noted on the Sample Receipt Report, see 3.2.5.

2) The pH of all preserved bottles provided for aqueous testing must be measured and recorded using the appropriate pH paper or calibrated pH probe in the following manner:

   a. The pH of all VOC vials will be measured on one of the partially consumed VOC vials after analysis is completed. If the vial pH is not <2, the measurement must be relayed to the customer via hard copy report.
   b. The pH of all nitric acid preserved sample aliquots will be measured prior to shelving. If the pH is not <2, the pH will be adjusted using nitric acid and the bottle allowed to stand 18 hours prior to performing sample analysis. The sample does not require client notification.
   c. The pH of all Sulfuric Acid preserved sample aliquots will be measured prior to refrigerated storage. If the pH is not <2, the pH will be adjusted using sulfuric acid and the initial pH relayed to the customer via hard copy report.

d. The pH of all Sodium Hydroxide preserved sample aliquots will be measured prior to refrigerated storage. If the pH is not >12, the pH will be adjusted using sodium hydroxide and the initial pH relayed to the customer via hard copy report.

e. The pH of all 1-L amber jars that require chemical preservation will be taken prior to extraction. If the pH is not <2, the pH will be adjusted using sulfuric acid or hydrochloric acid and the initial pH relayed to the customer via hard copy report.

The pH of all 1-L amber jars that require a specific pH range for the extraction will be taken prior to the extraction. If the pH is not in the designated range, the pH will be adjusted using hydrochloric acid or sodium hydroxide.

CHAIN OF CUSTODY SHALL BE SIGNED ALONG WITH THE DATE AND TIME OF RECEIPT BY THE ATTENDING LOG-IN TECHNICIAN

3.2.4. Sample Receipt Report, Form 3.3

The sample receipt report is completed when samples deviate from EPA, NELAP or WDNR protocol and/or are received in unregulated containers from the client. The report is filled out and attached to the COC and faxed to the client upon receipt of the samples. All appropriate information is stored in the analytical report folder.

3.2.5 Laboratory Information Management System (LIMS)

LIMS is a computer software system, which maintains traceability of a sample from the date of receipt throughout the analysis process to the final report. The log-in technician, after reviewing all parameters, enters information of each sample into the computer by analytical number, customer code, analyses codes, and sample precautions.

3.2.5.1 Analytical Number

A sample is identified by an eight digit, sequentially generated, analytical number which is recorded on the sample jar (self-adhesive label) and COC or Analytical Request form. The first two digits identify the sample location in the refrigerator or other storage area. The last six identify the unique analytical number of the sample. Sample bottle aliquot identification is written on the bottle using an aliquot identification code. The aliquot code is assigned based on preservation type, bottle type, and method specific requirements. The aliquot code assigned to each bottle must match the code that is brought into the LIMS system with the analyte of interest. If the proper aliquot was not provided, the laboratory must make note of this on the sample narrative. Listed below is the table of aliquots used to identify bottles:

Aliquot	B	C		D	E	F	G	U	P	V
Pres.	HNO_3	NONE		H_2SO_4	NAOH	NAOH + ZnOA	H_2SO_4	NONE	HCL	HCL
Analysis	METALS	ACID	NO_3-N	NH3-N	CN	SULFIDE	GREASE	HERB	DRO	VOC
	HARD	ALK	NO_2-N	NPOC			H-GREASE	PEST		GRO
		BROMIDE	OP	COD			G-GREASE	PCBs		
		T. BOD	PH	MCOD			HEM	PAHs		
		CBOD	SO_4-S	TKN			SGT-HEM	SVOCs		
		DIC	S	PHENOLS						
		S.CL	R-CN	PHOS.						
		COLOR	R-S	NO_3+NO_2-N						
		COND	SS & SA							
		CR+6	TDS							
		S.FL	TS & TA							
		HACS	SO_3-S							
			TURB							

3.2.5.2 Customer Code

A customer code, assigned by the Accounting Department, is entered with a sample or group of samples for invoicing purposes.

3.2.5.3 Analysis Code

All analyses requested are converted to a letter code which identify the analyte or group of analytes. This code may represent the analysis, EPA method number, matrix or instrument used to complete the analysis.

3.2.5.4 Special Precautions

Samples with special precautionary measures shall be noted in the LIMS system. All precautions are checked on the worklist for all employees. The Laboratory Officer will also notify the analysts during the weekly laboratory meeting. Special precautions on samples may include:

non hazardous	hazardous	refrigerate
limited sample	alkaline	cancer causing agent
acidic	toxic	do in order
do in hood	cyanide present	flammable

3.2.5.5 Computer-Generated Work Lists

The computer generates work lists from the LIMS system with all the necessary information and analysis requested from the client. The work lists are generated over night and the information is distributed to each analyst the following work day. The work list includes sample date, analytical number, test, project number, client's requested due date, sample type, and precautions.

3.2.5.6 Sample Delivery to Lab Areas

The log-in technician places each sample and/or aliquot on a cart for delivery to the appropriate refrigerated areas for storage. These samples are taken to the appropriate storage area approximately every 30 minutes. If a prolonged amount of time is spent in log-in due to

high sample volume, samples must be stored in the log-in refrigerator until they can be delivered to the appropriate storage area. Once in the proper storage area, it is the analyst's responsibility to retrieve each sample for analysis in a timely manner. See Figure 3.1: USFilter, Enviroscan Services Laboratory - Sample Flow Chart

3.3 Subcontracted Laboratories

Analysis requests, which cannot be completed at USFilter, Enviroscan Services will be subcontracted, with the permission of our client, to a NELAP certified laboratory if necessary. If a NELAP certified laboratory is not available, the analysis will be identified as a non-NELAP accredited analysis.

USFilter, Enviroscan Services investigates the credentials of potential subcontract laboratories. The subcontracting laboratory we choose must be competent to perform the requested analysis in a manner that is consistent with USFilter's requirements and any regulatory requirements that may apply. In the event that a subcontract laboratory is required, the client will be notified in writing of our intentions to subcontract the work.

USFilter performs the following steps to evaluate the competence of the subcontract laboratory. An employee will request the following information from the chosen laboratory:

1) A list of the subcontract laboratories current certifications/accreditation's.
2) A copy of the subcontract laboratories Quality Assurance Documentation.
3) Example tests report or certificate for the tests we intend to subcontract.
4) Price quotation for the tests to be subcontracted.

It is up to the discretion of the USFilter Employee as to the requirement of the following:

5) Copy of the subcontractor's procedure(s) for the work in question (i.e., sample amounts, preservation, sample bottle type, custody requirements, methodology).
6) A copy of the training records for the personnel responsible for performing the subcontracted work.

When the subcontract laboratory is accepted to do work for the clients of USFilter, Enviroscan Services, a file will be created. This file should contain the previously mentioned information and notification attesting to the fact that we have reviewed the information and accept said laboratory to perform the work in question.

At this time a subcontract laboratory specific analytical request sheet should be made up and stored in sample log-in.

3.4 Customer Complaints

USFilter, Enviroscan Services responds to all customer complaints in a professional and timely manner. Any complaint made against a USFilter result or activity will be resolved in a fashion that is acceptable to both the client and USFilter.

To resolve complaints that we receive via oral or written communication a record of the request is made initially by noting the following information from the client:

1. What company the complaint was received from
2. Who at the company placed the complaint
3. Who at USFilter received the complaint
4. Date/Time the complaint was received

5. The content/basis of the complaint.

The receiving person needs to make a decision as to the nature of the complaint and how it needs to be handled. If the complaint warrants compensation from USFilter, the complaint needs to be transferred to the Laboratory Director. However, if the complaint is viewed as being manageable, the receiving individual should continue investigating the nature of the complaint.

Once the initial information has been gathered, the complaint can be assigned to the individual that is most capable of handling the complaint. In most situations, the individual receiving the complaint should handle the complaint in its entirety. This will help to ensure that the client receives a prompt and mutually satisfactory resolution to the problem. If the complaint exceeds the scope of the receiving person's capabilities, the appropriate person must be sought out and provided all the available information at that time.

All evidence of the complaint and its resolution is filed by the receiving individual and retained in the client's file for future reference. A phone complaint taken by customer service that is handled immediately does not need to be logged into the client's folder.

If the nature of the complaint raises concern with USFilter's ability to comply with its written policies and procedures, a full Quality Systems Audit will be performed and corrective action considered.

FORM 3.1: SAMPLE BOTTLE KIT REQUEST

ORDERED BY: SMA
DATE: ____/____/____

Client: _____ **Required by:**____ /____/____
Address: _____
_____
_____

Attn: _____

Quantity	Bottle Type	H2SO4	HNO3	HCl	NaOH	None
Water Containers						
_____	40ml vials	_____	_____	_____	_____	_____
_____	1L amber glass	_____	_____	_____	_____	_____
_____	500ml amber	_____	_____	_____	_____	_____
_____	250ml amber	_____	_____	_____	_____	_____
_____	125ml plastic	_____	_____	_____	_____	_____
_____	250ml plastic	_____	_____	_____	_____	_____
_____	500ml plastic	_____	_____	_____	_____	_____
_____	1000ml plastic	_____	_____	_____	_____	_____
_____	Water trip blanks (GRO/PVOC/VOC samples only)					

Soil Containers

_____ 4 oz. jar
_____ 9 oz. jar
_____ 60 ml jar with no preservative for SOILDRO
_____ 60 ml jar with no preservative for SOILGRO
_____ No. of methanol vials
_____ TS cups
_____ MEOH trip blanks (GRO/PVOC/VOC samples only)

Requested Analyses

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

Miscellaneous Supplies

_____ Tedlar bags
_____ Syringe
_____ Methanol Impingers
_____ C-Tubes
_____ Temperature Blank

Bubble Wrap Bags - Small Number of:_____
- Large Number of:_____

_____ Gallons of water
_____ Liters of water

Sampling Instructions

Shipping Information

Sent: ____/____/____ **Cooler ID:** _____
Via: _____ **Returned:** ____/____/____
By: _____

COMMENTS: _____

FORM 3.2: USFILTER, ENVIROSCAN SERVICES - CHAIN OF CUSTODY (COC)

REQUEST FOR SERVICES USFilter

ENVIROSCAN SERVICES 301 W. MILITARY RD. ROTHSCHILD, WI 54474 1-800-338-SCAN

REPORT TO:
Name: _____
Company: _____
Address: _____
_____
Phone: (____) _____
P. O. # _____
Project # _____ Quote # _____
Location _____

BILL TO: (if different from Report To info)
Name: _____
Company: _____
Address: _____
_____
Phone: (____) _____

Sample Type
(Check all that apply)
- ☐ Groundwater
- ☐ Wastewater
- ☐ Soil/Solid
- ☐ Drinking Water
- ☐ Oil
- ☐ Vapor
- ☐ Other

Turnaround Time
- ☐ Normal
- ☐ Rush (Pre-approved by Lab)

Date Needed _____
Approved By _____

ANALYTICAL REQUESTS
(use separate sheet if necessary)

LAB USE ONLY	DATE	TIME	No. of Containers COMP	No. of Containers GRAB	SAMPLE ID						REMARKS

Delv. Hand Comm
Ship. Cont. OK Y N N/A
Samples leaking? Y N N/A
Seals OK? Y N N/A
Rec'd on ice? Y N N/A °C

CHAIN OF CUSTODY RECORD

SAMPLERS: *(Signature)*

RELINQUISHED BY: *(Signature)*	DATE/TIME	RECEIVED BY: *(Signature)*
RELINQUISHED BY: *(Signature)*	DATE/TIME	RECEIVED BY: *(Signature)*
RELINQUISHED BY: *(Signature)*	DATE/TIME	RECEIVED FOR LABORATORY BY: *(Signature)* DATE/TIME

Comments: _____
_____
_____
_____
_____

FORM 3.3: USFILTER, ENVIROSCAN SERVICES - SAMPLE RECEIPT REPORT

Client: _____ **Date Received:** _____/_____/_____

Analytical Number: _____ **through** _____

Check all deviations from the EPA or WDNR sample protocol.

[ ] Sample(s) received at _____°C which is above the EPA and WDNR limit of 4°C.

[ ] VOC vial(s) received with headspace.

[ ] Sample(s) received in bottles not furnished by USFilter, Enviroscan Services. The preservation method, if used, is unknown.

[ ] Sample(s) were not properly preserved per EPA or WDNR protocol for the following analyses:
- _____

[ ] Sample(s) were received beyond the EPA/WDNR holding time for the following analyses:
- _____

[ ] Sample date/time not supplied by client. Actual holding time is unknown.

[ ] GRO / PVOC / VOC / DRO (circle) sample(s) are <19.5 grams. This report is the qualifier flag for that QC failure. The client has been contacted for further instructions. Analytical number(s) of the sample(s) under weight are:
- _____

[ ] GRO / PVOC / VOC (circle) sample(s) were between 26.4 and 35.4 grams. Methanol was added in a 1:1 ratio in the lab. Analytical number(s) of the sample(s) affected are:
- _____

[ ] GRO / PVOC / VOC / DRO (circle) sample(s) are >35.4 grams and are required to be rejected. This report is the qualifier flag for that QC failure. The client has been contacted for further instructions. Analytical number(s) of the sample(s) affected are:
- _____

[ ] Other problems:
- _____

Client contacted concerning the above deviations:

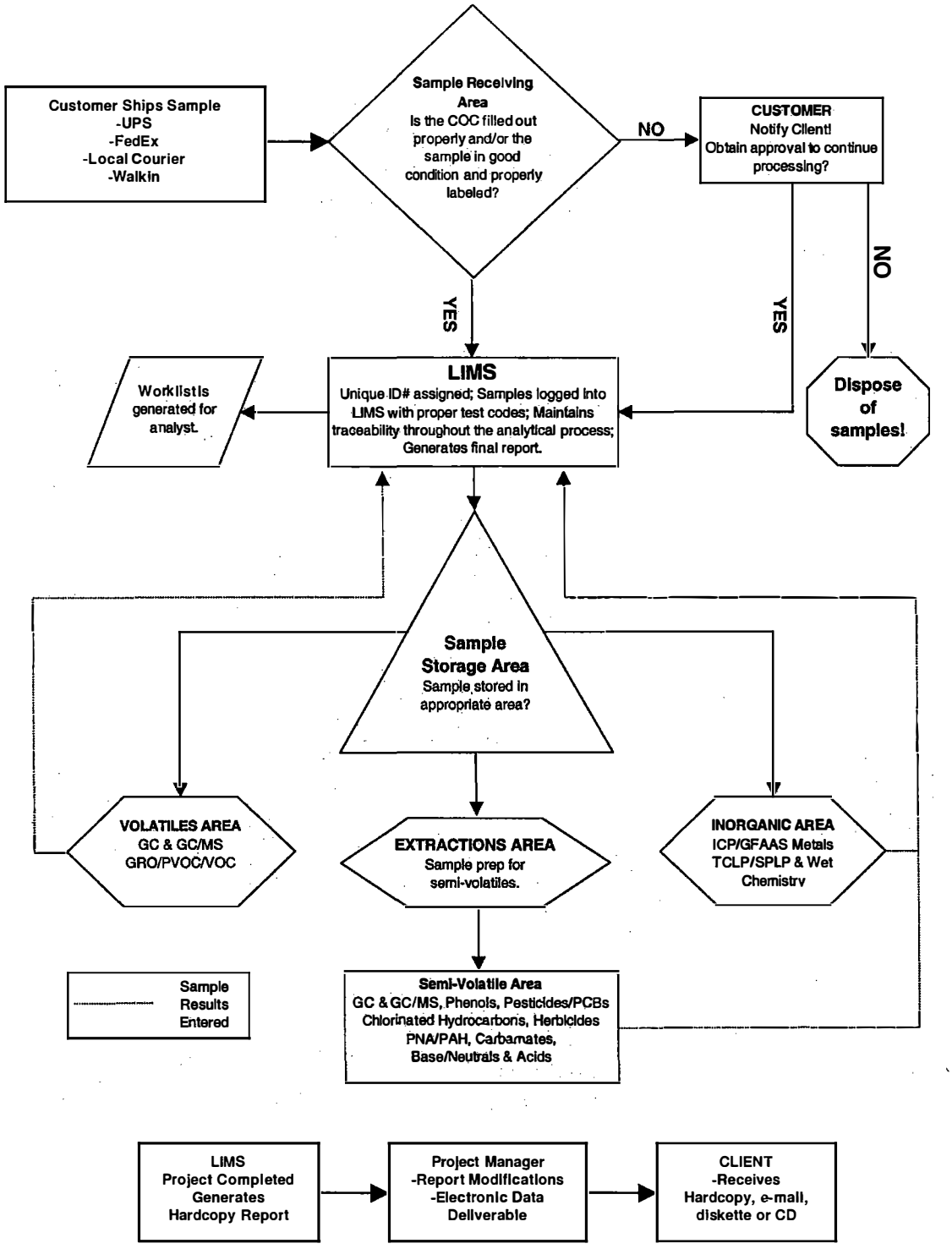
_____ notified of the above deviation(s) on _____/_____/_____ @
contact name

_____:_____ am/pm by _____ and the client ordered the following:
initial

[ ] Proceed with analyses as ordered.
[ ] Proceed with analyses after taking the following corrective action:
- _____

[ ] Do NOT proceed with analyses.

FIGURE 3.1: USFILTER, ENVIROSCAN SERVICES SAMPLE FLOW CHART



SECTION 4.0: LABORATORY EQUIPMENT AND SUPPLIES

USFilter, Enviroscan Services laboratory equipment and supplies are continually monitored and well maintained to meet the standards for high quality work. Equipment is checked against certified NIST traceable standards where available during calibration and throughout the analysis process. Critical supplies are received with a certification of compliance to be contaminant-free.

4.1 Laboratory Equipment

The laboratory equipment plan consists of scheduled checks for continuous monitoring. All critical equipment logbooks shall be kept current and corrective/preventive action taken to maintain the equipment. See Table 4.1: Laboratory Equipment Maintenance Schedule for a listing of the routine checks and the quality control limits.

4.1.1 Equipment Monitoring

All records are maintained and properly stored for critical equipment. The following records are completed and submitted for review and storage:

a. Temperature records for refrigerators, freezers and incubators are recorded daily and submitted to QA monthly for review.
b. Balance Checks are performed daily just prior to useage. Results are not submitted to QA until scale log-book is full.
c. Water Quality is checked weekly for Anions, Conductivity and pH. The results are submitted to QA immediately for review.
d. Water Hardness is checked bi-weekly by ICP and submitted to QA and Building Maintenance for review.
e. VOC levels in stored water are checked twice per week using EPA 8021 analysis. The data is tabulated and submitted to QA monthly.

4.1.2 Equipment - Corrective/Preventive Action

Corrective/preventive action reports are completed as necessary and submitted to the immediate supervisor as soon as possible for appropriate course of action when equipment needs maintenance. See the steps below for troubleshooting:

4.1.2.1 Refrigerator/Freezer:

a. Check power source to unit.
b. Check if unit is "iced up".
c. Adjust temperature and recheck in one hour.

4.1.2.2 Incubator/Oven:

a. Check power source to unit.
b. Check to see if the oven is in use; move samples to appropriate oven.
c. Adjust the temperature and recheck in one hour.

4.1.2.3 Laboratory Water:

a. Take a new aliquot and repeat check.
b. Check conductivity meter for malfunction and recheck sample.
c. Wait approximately one hour and recheck sample. If value decreases continue monitoring.
d. Duplicate values or increasing values require anions and pH check.

e. Detectable quantity of anions require a completed CPAR.

4.1.2.4 Balances:
A maintenance contract is held by an independent firm to clean and maintain calibration yearly or sooner, if necessary. A certificate is issued and kept on file in the quality assurance department.

4.1.2.5 Pipettes:
A weekly check is performed on all automatic dispensing pipettes. The weight recovery is compared to set quality control limits. The instrument is calibrated by the QA Manager if necessary.

4.2 Laboratory Supplies

Glassware, reagents, solvents, standards, gases, and other materials are carefully selected to meet specifications given in USEPA and Wisconsin DNR approved methodologies. Certificates for critical supplies are stored in the quality assurance department.

4.2.1 Glassware
Only Class A volumetric glassware is purchased for laboratory use. All glassware is cleaned thoroughly prior to use to help prevent contamination. Glassware may be reused (i.e. extraction separatory funnels, graduated cylinders, etc.) after proper cleaning and drying.

4.2.2 Reagents, Solvents, and Standards
All standards, reagents, and solvents are labeled with the date of receipt and stored properly. Reagent and solvent purity is chosen based on sample analysis. In general, USFilter, Enviroscan Services uses certified grades that meet ACS standards. Each certificate shall contain ingredient information, purity, lot number, expiration date and shall be stored in the quality assurance department.

4.2.3 Gas Supply
Ultra high purity grade gas is used for laboratory applications. Tanks used for the semi-volatile and volatile labs are hooked up in series and piped into each lab for analysis needs. All lines are equipped with line purifiers. Compressed air and hydrogen are piped to all labs that require their use.

4.2.4 Purity Check
Standards with new lot numbers shall be compared to existing lot numbers. Multiple vendors are used for this purpose. Standards shall be within an acceptable range or rejected. Reagents and solvents are checked by preparing and processing a blank through the entire analytical process. The check for reagents and solvents is completed with each new lot received. Reagents or solvents found to contain any interfering substances cannot be used for analysis.

4.2.5 Sample Bottles
A supply of sample bottles including soil jars, VOC vials, plastics, and liter ambers are kept at the laboratory for client use. Glass containers including VOC vials, liter ambers, and soil jars are purchased pre-cleaned and certified contaminant-free from our supplier. Certificates are filed in the quality assurance department. The laboratory pre-weighs each jar for VOC, GRO and DRO soils analyses before shipping them to our clients.

TABLE 4.1: LABORATORY SUPPORT EQUIPMENT MAINTENANCE SCHEDULE

Equipment:	Model/ID:	Location:	Temperature Check		Maintenance Schedule:
Refrigerator		All areas	Daily	0-4°C	Clean at least once per year
Freezer		All areas	Daily	-10 to -20°C	Defrost at least once per year
Incubators		Inorganic Lab	Daily	19-21°C	Clean at least once per year
Oven-VWR Scientific	1350FM (A)	Inorganic Lab	Daily	148-152°C	Keep clean
Oven-Precision Scientific	114A/(B)	Inorganic Lab	Daily	103-105°C	Keep clean
Oven-Precision Scientific	104A/(C)	Inorganic Lab	Daily	178-182°C	Keep clean
Laboratory Water	Methods IV	InorganicLab	Daily	pH;Cond	Weekly; Nitrate; Chloride; Sulfate, Conductivity/Resistivity
USFilter Modulab	MAX 149	Inorganic Lab	Daily	pH;Cond	Weekly; Nitrate; Chloride; Sulfate; Conductivity/Resistivity
Balance-Fisher	A160	Inorganic Lab	Daily	10.0±.0010g	Full calibration check monthly
Balance-Mettler Toledo	PB 1502	Inorganic Lab	Daily	10.0 ± 0.1g	Full calibration check monthly
Balance-Sartorius	B310S	VOC Lab	Daily	10.0± 0.1g	Full calibration check monthly
Balance-Sartorius	BA1105	Lab C	Daily	10.0 ± 0.001g	Full calibration check monthly
Balance-Sartorius	BP612	Lab C	Daily	10.0 ± 0.1g	Full calibration check monthly
Balance-Sartorius	BA210	Sample Receipt	Daily	20.0 ± 1.0g	Full calibration check monthly
Thermometers	NA	All Areas	Check before use		Calibration check against a NIST certified thermometer once per year
Auto-Pipettes	NA	Various Areas	Check before use		Check yearly; See SOP097.
Laboratory Glassware	NA	Inorganic	Check before use		Wash w/ detergent except for phosphorus, orthophosphate, ammonia & TKN analyses. Rinse w/ tap water; and rinse w/D-water three times; Air dry; Store properly
Laboratory Glassware	NA	Semivolatiles	Check before use		Wash w/ detergent; Rinse w/D-Water; Air Dry; Place into kiln and heat to 400°C for 100 minutes; Cool and Store properly.
Laboratory Glassware	NA	Volatiles	Check before use		Rinse with D-water; Dry in 130°C oven for 30 minutes; Cool and Store properly.

SECTION 5.0: METHODS AND PROCEDURES

USFilter, Enviroscan Services follows approved analytical methods from the Wisconsin Department of Natural Resources (DNR) and the United States Environmental Protection Agency (USEPA) protocols for environmental sample analysis. USFilter, Enviroscan Services shall use Standard Operating Procedures (SOP) for each analysis to explicitly dictate laboratory operations.

5.1 Standard Operating Procedures

USFilter, Enviroscan Services utilizes standard operating procedures to address administrative and technical procedures. The administrative SOP identifies the comment management procedures used by USFilter. A technical SOP addresses criterion that may include specifics about the instrument settings, calibration, glassware, method detection limits, cleaning instructions, and quality control requirements. These standard operating procedures are written to assist the analyst in performing each analysis consistently to reduce the potential for error and to increase quality of the final result. These procedures have a specific "SOP" code with a three-digit number, for example, (SOP179-Total Solids) was developed from the guidelines specified in USEPA method 160.3, "Methods for Chemical Analysis of Water and Wastes."

A USFilter, Enviroscan Services Technical SOP must addresses the following components per NELAC Chapter 5 Quality Systems:

Identification of the test method
Applicable matrix or matrices
Method detection limit study
Scope and application, including list of analytes
Summary of the test method
Definitions
Interferences
Equipment and supplies
Glassware cleaning procedure
Reagents and standards
Sample colletction, preservation, shipment and storage
Quality control requirements
Calibration and standardization requirements
Detailed Procedure (step by step instructions to complete analysis)
Calculations
Method performance (precision and accuracy data generation procedure)
MS/MSD selection procedure
Data assessment and acceptance criteria for quality control measures
Corrective actions for out-of-control data
Contingencies for handling out-of-control or unacceptable data
Waste management
Pollution prevention
Safety
References
SOP approval by the analyst, technical director, and QA/QC officer
Revision date, Revision number, Effective date

5.1.1 Tracking

SOPs are tracked by the QA officer for determining the yearly review schedule for active SOPs. The QA officer is also in charge of monitoring revision dates, active revision numbers and procedure archiving. USFilter utilizes electronic file tracking software to keep track of active and archived revisions. Previous versions of the SOP can be recalled and viewed with all changes shown in red and underlined.

5.1.2 Storage

Controlled copies of the USFilter, Enviroscan Services methods are stored in the quality assurance department master file cabinet. The QA officer shall maintain complete files and inventory of all methods. A copy of each procedure is kept in the appropriate areas of the lab for reference. When a copy of the standard operating procedure leaves the boundaries of this facility, the procedure is to be marked "UNCONTROLLED". At this time, it is no longer USFilter, Enviroscan Services responsibility to update this procedure.

5.1.3 Updating

USFilter, Enviroscan Services methods are reviewed as part of an internal audit and QA/QC reporting schedule on a yearly basis. The analyst along with the technical director and QA officer shall make the necessary updates. New methods and revisions are to be reviewed and approved by the analyst, technical director, and QA officer prior to their use. Additional notes may be made on the methods by the analysts to aid in their analysis such as appropriate sample sizes per project, etc. These notes should become additions to the SOP (not project dependent) at the next revision. At the time a new revision is put into effect, all old revisions must be recovered by the QA officer. The QA officer will then close out the effective period for the SOP and distribute the new revision to the appropriate laboratory personnel. The following distribution locations shall be used for the new controlled SOPs.

Inorganic Laboratory

a. Technical Director's Office.
b. South End binder.
c. North End binder.
d. Area Specific binder

Organic Laboratory

a. Technical Director's Office.
b. Analytical Technician's Laboratory.
c. Analytical Chemist's Office.

5.2 Initial Demonstration of Capability (IDC)

Initial demonstration of capabilities consists of a method validation, method detection limit study, and/or linear dynamic range study. The IDC is performed within the required precision and accuracy as defined by each individual approved method. An IDC study must be completed by each new analyst and when a new instrument is purchased, a significant instrument modification has occurred (i.e. different type of column, detector, trap, etc.), or substantial method changes have taken place. Data from the completed validation is recorded and stored in the quality assurance department analyts's personnel file, the analysis file and in the analysis area. When completed, a copy of the NELAC Demonstration of Capability Certification Statement (modified) must be completed, signed by the appropriate parties and attached to the data generated by the IDC. The QA Manager has a file called DEMOFCAP-1 that must be used for certification, see Figure 5.1 for the certification statement.

See Table 5.2: USFilter, Enviroscan Services - Minimum QC Requirements, for the initial demonstration of capability recovery and deviation limits.

5.2.1 Inorganic Area (IDC)

The initial demonstration for inorganic parameters and metals include a method validation study, linear dynamic range determination and a method detection limit study (see section 5.3 for MDL information).

5.2.1.1 Method Validation Study(VAL)/Demonstration of Capability (DoC)

The method validation study is performed by preparing and analyzing five aliquots of a known QC Standard from an outside source. If not available, the QC Standards may be prepared using a 2nd source stock standard. Calculate the % Recovery (%R) and the Standard Deviation (SD). Compare the %R and SD to the method acceptance criteria as stated in the standard operating procedures or the EPA method.

The method validation can be further substantiated through the analysis of a Blind QC sample. As part of our program, the QA manager will administer this sample if and when available.

5.2.1.2 Linear Range Determination (LDR)

The linear dynamic range is determined by analyzing two standards that increase in concentration starting beyond the highest level standard in the calibration curve. These points are then calculated for percent recovery. The calibration range of the instrument is determined to exist within the instrument 's response if the standards analyzed have a minimum recovery greater than 90.0%. If a nonlinear response (<90.0% recovery) occurs or the response is overrange, i.e. top of peak is flat, the calibration range must be verified by recovering greater than 90.0% on a standard analyzed at the highest calibration level. These standards are analyzed according to the method used including all preparation steps (i.e.,extraction, digestion, etc.).

5.2.1.3 Method Detection Limit Study

See Section 5.3 for information on performing an MDL study.

5.2.2 Organics Area (IDC)

The initial demonstration for organic parameters include a method validation study and a method detection limit study. The method validation study must be performed prior to running the MDL study. An instrument may be validated once for similar methods using the most stringent criteria. For example, methods 502.2 and 8021 are validated together. Use the appropriate number of standards, five standards must be used. Use the lowest concentration between the methods. If the concentration is not applicable to the laboratory's calibration range (i.e. pesticides/PCB) the analyst shall use a mid-range standard and the Method Accuracy and Precision conversion table for single analyte precision to evaluate the data. The analyst shall convert the given acceptance criteria to a percentage and evaluate. Use the strictest limits when evaluating the data for each compound from each method.

5.2.2.1 Method Validation Study(VAL)/Demonstration of Capability (DoC)

The method validation study is performed by prepare and analyze five aliquots of a known QC Standard from an outside source. If not available, the QC Standards may be prepared using a 2nd source stock standard. Calculate the % Recovery (%R) and the Standard Deviation (SD). Compare the %R and SD to the method acceptance criteria as stated in the standard operating procedures or the EPA method.

The method validation can be further substantiated through the analysis of a Blind QC sample. As part of our program, the QA manager will administer this sample if and when available.

- Use the appropriate number of standards. For example, the 600 series lists four minimum standards; the 500 series lists five minimum standards; therefore, five standards must be used.

- Use the lowest concentration between the methods. For example, if the concentration is not applicable to the laboratory's calibration range (i.e. pesticides/PCB) the analyst shall use a midrange standard and the Method Accuracy and Precision conversion table for single analyte precision to evaluate the data. The analyst shall convert the given acceptance criteria to a percentage and evaluate.

- Use the strictest limits when evaluating the data for each compound from each method.

5.2.3 Validation Report Criteria

The following information shall be included on each validation report: Instrument identification, date of validation, data file, method reference, standard concentrations, compound(s), analyst name, resulting concentrations, average, pass/fail notification.

5.3 Reporting Limits

There are four types of reporting limits that can be used during the evaluation of sample data

a. Limit of Detection (LOD)/Method Detection Limit (MDL)
b. Limit of Quantitation (LOQ)
c. Practical Quantitation Limit (PQL) or Reporting Limit (RL)
d. Gravimetric and titrimetric limits.

Current limits used throughout the laboratory are listed in the USFilter, Enviroscan Services's Environmental Guidance: Preservation, Reporting & Regulatory Limits.

5.3.1 Limit of Detection (LOD)/Method Detection Limit (MDL)

A method detection limit study is completed as described in method 40CFR Part 136, Appendix B following Enviroscan Services SOP093-MDL. A minimum of eight aliquots of spiked reagent water at a concentration between one and five times the calculated method detection limit is analyzed. The standard deviation of the eight results is determined and multiplied by the student's "t" factor at 99% confidence interval and one degree of freedom. The equation is as follows:

$$MDL = t_{(n-1,1-\mu=0.99)}\, s$$

Most areas use this method of detection in reporting analytical data. Methods that do not have a realistic MDL or methods that are not conducive to performing MDL studies use a PQL. Additionally, analytes that are not technically sound to be reported at this level may default to the PQL.

5.3.2 Limit of Quantitation (LOQ)

The LOQ is calculated as 10/3 times the LOD/MDL as obtained in the method detection limit study.

5.3.3 Practical Quantitation Limit (PQL)

The practical quantitation limit (PQL) is the lowest standard used in the calibration curve generated by the instrument to quantitate data. The PQL reflects a level that can be measured by good laboratory practices under normal operating conditions within specified limits of precision and accuracy. The low standard is seen on the chromatogram, chart paper, or digital readout as a response greater than noise which is determined to be reliable. A positive confirmation of an analyte exists in the sample if the response for the analyte is equal to or greater than the low standard. Areas, which use practical quantitation limits, are determined by the ability to report technically sound data for the analyte(s) as well as the need for low level reporting. The following analytes have been classified by the Wisconsin Department of Natural Resources as compounds of concern and must be reported down to the LOD.

INORGANICS	**Phthalates & Adipates**	**Volatiles**
Metals	Di(2-ethylhexyl)phthalate	1,1,2,2-Tetrachloroethane
Antimony	**Chlorinated Hydrocarbons**	1,1,2-Trichloroethane
Beryllium	Hexachlorobenzene	1,3-Dichloropropene (cis/trans)
Cadmium	**Dioxins/Furans**	Bromodichloromethane
Lead	Dioxin	Bromoform
Thallium	**PCBs**	Bromomethane
Mercury	Polychlorinated biphenyls	Chloroform
Chromium (Hexavalent)	**Chlorinated Pesticides**	Chloromethane
	DDT and Metabolites	Methyl tert-butyl ether (MTBE)
ORGANICS	Heptachlor	Methylene Chloride
Acids/Phenols	Heptachlor epoxide	Vinyl Chloride
Pentachlorophenol (PCP)	Lindane	Dibromochloropropane (DBCP)
Benzidines	Toxaphene	Ethylene dibromide (EDB)
Benzidine	**Carbamate Pesticides**	
Haloethers	Aldicarb	
Bis(chloromethyl)ether	**Nitrogen Pesticides**	
Nitroaromatics	Alachlor	
2,4-Dinitrotoluene	Dimethoate	
2,6-Dinitrotoluene	Parathion	
PAHs	Trifluralin	
Benzo(a)pyrene		

5.3.4 Gravimetric and Titrimetric Detection Limits

Analyses completed by gravimetric and titrimetric methods have detection levels based on a minimum weight or volume. This is based on the current normality and sample volume for each analysis. Modifications are made based on the sensitivity of the balance, buret, or normality used. These limits are referred to as LOQs. When an LOQ is assigned to an analysis that has either a

minimum weigh-back or minimum titration amount as the limiting factor, the laboratory must clearly establish that the level chosen can be achieved through our standard procedure. The accuracy of the instrumentation may not be the limiting step. The demonstration of capability can be done by performing a method detection limit study or analyzing a know standard at the minimum level.

5.3.4.1 Gravimetric Limits
A weight no less than 0.0010 grams shall be weighed for a sample to be at a detection level for gravimetric analyses.

5.3.4.2 Titrimetric Limits
A volume no less than 0.50 ml of titrant shall be measured for a sample to be at a detection level for titrimetric analyses.

5.4 Guidelines for Good Laboratory Practices Index

USFilter, Enviroscan Services makes use of a number of standard operating procedures to clearly identify the daily tasks undertaken by the laboratory. Many administrative procedures are standardized as a SOP. Listed below are the proceduralized follows:

SOP#	Title/Description:	SOP#	Title/Description:
070	NELAC Standard Review	085	Reporting Subcontracted Data
071	Tracking Controlled Documents	086	Client Confidentiality
072	NIST Traceability	087	Sample Disposal
073	Work Review	090	Oven, Refrigerator, Incubator Temp
074	Data Anomolies	091	Log-In
075	Complaint	092	Limit Calculations
076	Employee Experience	093	Method Detection Limit
077	Education/Training	094	Hood
078	Internal Audits	095	Chain of Custody
079	Management Review	096	E-mail report
080	QC Criteria - Unregulated	097	Pipette
081	IDC/DOC	098	Data
082	Data/Peer Review	099	Outlier
083	QC Review	100	SOP
084	Purchase, Reception, Storage	101	Corrective/Preventative Action Report

5.5 Analytical Methods

All methods used in the laboratory are based on the United States Environmental Protection Agency (USEPA), the State of Wisconsin Department of Natural Resources, and various other approved methods. Copies of these and other source methods are located in the library for reference. The QA Officer shall maintain these references. Method sources include:

- Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Environmental Monitoring and Support Laboratory, 26 West Martin Luther King Drive, Cincinnati, Ohio 45268, Revised 1983, including EPA-600/4-84-017, March, 1984.

- Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, December 1996.

- Standard Methods for the Examination of Water and Wastewater, 19th Edition, American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, 1995.

- 1991 Annual Book of ASTM Standards, Section 11.01, 11.02, and 11.04, Water and Environmental Technology, American Society for Testing and Materials, 916 Race Street, Philadelphia, PA 19103.

- Code of Federal Regulations Title 40, Part 136, Appendices A and B, U.S. Government Printing Office, Washington, D.C. 20402, 1991.

- Methods for the Determination of Organic Compounds in Drinking Water, EPA/600/4-88/039 and EPA/600/4-90/020, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268.

- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, Office of Research and Development, June 1991.

- Method for Determining Gasoline Ranges Organics, Wisconsin Department of Natural Resources, Office of Technical Service, P.O. Box 7921, Madison, Wisconsin 53707, PUBL-SW-140.

- Method for Determining Diesel Range Organics, Wisconsin Department of Natural Resources, Office of Technical Service, P.O. Box 7921, Madison, Wisconsin 53707, PUBL-SW-141.

- Total Recoverable Petroleum Hydrocarbons, Wisconsin Department of Natural Resources, Office of Technical Service, P.O. Box 7921, Madison, Wisconsin 53707, PUBL-SW-143.

Figure 5.1 Demonstration of Capability Certification Statement

USFilter

USFILTER, ENVIROSCAN SERVICES
301 West Military Road
Rothschild, WI 54474

Demonstration of Capability Certification Statement

Analyst Name: _____

Trainer(s) Name: _____

Work Area: _____

Analyte/Class of Analytes: _____

Method #: _____

SOP # and Revision Date: _____

Demonstration Performed: ☒ - IDC (5 aliquots) ☐ - Blind

Matrix: ☒ - Reagent Water ☐ - Ottowa Sand

The undersigned, CERTIFY that:

☒ Y ☐ N The study was performed by the person identified above.

☒ Y ☐ N The analyst identified above has met the predetermined precision and accuracy requirements.

☒ Y ☐ N A copy of the most recent test method and the latest revision of the SOP is available and was read prior to performing this study.

☒ Y ☐ N The data associated with the demonstration of capability is true, accurate, complete and self-explanatory.

☒ Y ☐ N All raw data necessary to support this analyses have been retained at the facility, and the associated information is well organized and available for review by authorized inspectors.

_____	_____	____/____/____
Analyst Name	Signature	Date
_____	_____	____/____/____
Area Supervisor's Name	Signature	Date
Eric P. Martin	_____	____/____/____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

Table 5.1: USFilter, Enviroscan Services – Analytical SOP/Method Reference Summary

			EPA Method Number					Standard Methods
SOP #	Analysis	Technique	Drinking Water	Ground Water	Waste Water	Hazardous Waste	Soil/Solid	20th Edition
102	COD, Macro	Titrimetric				410.1,410.2	410.1,410.2	5220C
105	Sulfate	Gravimetric		375.3	375.3	375.3	375.3	4500SO4 D
106	Sulfur, Total	Gravimetric		375.3	375.3			
107	pH	Electrometric	150.1	150.1	150.1	9040	9045	4500H B
110	Acidity	Titrimetric		305.1	305.1			2310
111	Sulfate	Turbidimetric	375.4	375.4		9038		4500SO4 E
112	Alkalinity	Titrimetric	310.1	310.1	310.1			2320
120	Carbon(NPOC,POC,DIC)	Persulfate Oxidation		415.1	415.1		9060	5310C
126	Hexavalent Chromium	Colorimetric			7196		3660	3500 Cr D
128	Color	Color-Spec.	110.2	110.2	110.2			2120
129	Conductivity	Conductivity Bridge	120.1	120.1	120.1	9050		2510
134	Cyanide (AA)	Pyridine-Barbituric	335.3	335.3	335.3	9012	9012	4500 CN E
135	Cyanide	Flow Injection	335.3	335.3	335.3	9012	9012	4500 CN E
138	Fluoride	ISE	340.2	340.2	340.2			4500 F C
141	Grease & Oil	Gravimetric		413.1	413.1	9071	9070	5520
143	Hardness	Calculation	200.7	200.7	200.7	6010	6010	2340B
158	NO3+NO2,NO3-N	Colorimetric	353.1	353.1	353.1	9056	9056	
161	Nitrogen, TKN	Semi-Micro Kjeldahl	351.3	351.3	351.3	351.3	351.3	4500N C
166	COD, Mini	Colorimetric		410.4	410.4			5220D
168	pH	Electrometric	150.1	150.1	150.1	9040	9045	4500H B
170	Phenols	Colorimetric	420.2	420.2	420.2	9066	9066	
177	Solids, Settleable	Volumetric		160.5	160.5	160.5		2540F
178	Ash, Suspended	Gravimetric		160.4	160.4			2540 E
178	Solids, Suspended	Gravimetric	160.2	160.2	160.2	160.2		2540D
179	Solids, Total	Gravimetric	160.3	160.3	160.3	160.3	160.3	2540B
180	Specific Gravity	Hydrometer						2710F
185	Sulfide, Sulfite/Thiosulfate	Iodimetric		376.1	376.1	9030		4500S2 E
187	Thiocyanate	Colorimetric						4500CN M
189	Turbidity	Nephelometric	180.1	180.1	180.1			2130
190	Volatile Acids	Titrimetric		5560C				
193	BOD	5 Day, 20C	405.1	5210B	5210B			5210B
198	Ignitability	Pensky-Martens				1010	1030	
199	Corrosivity	Electrometric				1110	1110	
200	Cyanide, Reactive	Gas Evolution				7.3.3.2	7.3.3.2	
200	Sulfide, Reactive	Gas Evolution				7.3.4.1	7.3.4.1	
201	Extraction, EPTOX	Extraction				1310	1310	
202	Grease & Oil	IR Freon Extraction		413.2				5520C
203	Bromide	Ion Chromatography	300.0	300.0	300.0	9056	9056	4110
203	Chloride	Ion Chromatography	300.0	300.0	300.0	9056	9056	4110
203	Fluoride	Ion Chromatography	300.0	300.0	300.0	9056	9056	4110
203	Nitrogen, Nitrate	Ion Chromatography	300.0	300.0	300.0	9056	9056	4110
203	Nitrogen, Nitrite	Ion Chromatography	300.0	300.0	300.0	9056	9056	4110
203	Sulfate	Ion Chromatography	300.0	300.0	300.0	9056	9056	4110
205	Chlorophyll a	Spectrometric						10200H
207	Cyanide, Amenable	Pretreatment	335.1	335.1	335.1	9012	9012	4500 CN G
208	Solids, Dissolved	Gravimetric	160.1	160.1	160.1	160.1		2540C
212	Volatile Solids	Calculation		160.4	160.4	160.4	160.4	2540E
227	Extraction, TCLP	Extraction				1311	1311	
236	Extraction, SPLP	Extraction				1312	1312	
251	Sulfite	Calculation	377.1	377.1	377.1			Calculation
261	TKN	Flow Injection	351.2	351.2	351.2			
262	Phosphorus	Flow Injection	365.4	365.4	365.4			
263	Orthophasphate	Flow Injection	365.1	365.1	365.1			
264	NH3-N	Flow Injection	350.1	350.1	350.1			

Table 5.1: USFilter, Enviroscan Services – Analytical SOP/Method Reference Summary

			EPA Method Number					Standard Methods
SOP #	Analysis	Technique	Drinking Water	Ground Water	Waste Water	Hazardous Waste	Soil/Solid	20th Edition
265	Chloride	Flow Injection	325.2	325.2	325.2			
266	NPOC	Tekmar/Dohrmann	415.1	415.1	415.1	9060	9060	5310 C
267	Phenols	Flow Injection	420.2	420.2	420.2	9066	9066	
268	AS & AI Sulfides	Digestion				9030	9030	
269	Sulfides	Titrimetric				9034	9034	
270	TOC/NPOC	Tekmar/Dohrmann	5310 B	415.1	415.1	9060	9060	5310 B
271	NO3+NO2-N (FIA-Lachat)	Flow Injection	353.2	353.2	353.2			
272	Soluble Salts	Calculation					MOSA	
273	Chloride (ISE)	Ion Selective Electrode						
370	Silica & Silicate	Colorimetric	370.1					
420	Extraction, SM6630B	Extraction				3510,50	3510,50	
427	Extraction, Formaldehyde	Extraction				3510,50	3510,50	
428	Cleanup, CarboPrep	Cleanup						
430	Cleanup, Florisil	Extraction				3620B	3620B	
431	SPE HEM Oil and Grease	Gravimetric		1664A	1664A	1664A		
432	Sulfur Cleanup	Cleanup				3660	3660	
433	Sulfuric Acid Cleanup	Cleanup				3665	3665	
500	Acrolein/Acrylonitrile	GC/MS		8260	8260	8260	8260	
500	Volatiles	GC/MS	524.2	624	624	8021/8260	8021/8260	6000
501	SemiVolatiles	GC/MS	525.1	625	625	8270	8270	6410
502	Pesticides/PCB	Gas Chromatography	508	608	608	8081	8081	6630B
503	Phenols	Gas Chromatography		604	604	8041/8270	8041/8270	6420B
504	TPH (California)	Gas Chromatography						9073
505	Volatiles	Gas Chromatography	502.2	601/602	601/602	8021	8021	6000
507	Pesticides (GC/ECD)	Gas Chromatography				8081	8081	
508	PAH	HPLC	550.1	610	610	8270/8310	8270/8310	6440B
512	Diesel Range Organics	Gas Chromatography		WIDNR	WIDNR/8015			
513	Gasoline Range Organics	Gas Chromatography	Wisconsin Department of Natural Resources					
514	TRPH	IR Spectrophotometer		418.1	418.1	418.1	418.1	
515	Chlorinated Hydrocarbons	Gas Chromatography		612	612	8120A	8120A	
516	Herbicides	Gas Chromatography	515.1	8150	8150	8150	8150	6640
518	Volatiles/Carbon Tubes	Gas Chromatography		1003	NIOSH1501			
519	Carbamates	HPLC	531.1					
525	PVOC (GC/PID)	Gas Chromatography						
527	VOC miscellaneous	Gas Chromatography						
529	Glycols	Gas Chromatography				8015	8015	
530	EDB/DBCP	Gas Chromatography	504	504	8021		8021	
531	Pesticides, Organochlorine	Gas Chromatography	508	608	608	8081	8081	6630B
537	PCBs in Oil (GC/ECD)	Gas Chromatography		EPA 600 RD				
539	PCBs (GC/ECD)	Gas Chromatography	508	608	608	8082	8082	
540	OP Pesticides (NPD)	Gas Chromatography				8141	8141	
541	Organic Acids by HPLC	HPLC						
913	Mercury	A2S, Cold Vapor	245.1	245.1	245.1	7470	7471	3500HG B
950	Aluminum	ICP		200.7	200.7	6010	6010	3120
950	Arsenic	ICP			200.7	6010	6010	3120
950	Barium	ICP	200.7	200.7	200.7	6010	6010	3120
950	Berylium	ICP		200.7	200.7	6010	6010	3120
950	Bismuth	ICP		200.7	200.7			3120
950	Boron	ICP		200.7	200.7			3120
950	Cadmium	ICP			200.7	6010	6010	3120
950	Calcium	ICP	200.7	200.7	200.7	6010	6010	3120
950	Chromium, Total	ICP	200.7	200.7	200.7	6010	6010	3120
950	Cobalt	ICP		200.7	200.7	6010	6010	3120
950	Copper	ICP	200.7	200.7	200.7	6010	6010	3120

Table 5.1: USFilter, Enviroscan Services – Analytical SOP/Method Reference Summary

			EPA Method Number					Standard Methods
SOP #	Analysis	Technique	Drinking Water	Ground Water	Waste Water	Hazardous Waste	Soil/Solid	20th Edition
950	Iron	ICP	200.7	200.7	200.7	6010	6010	3120
950	Lead	ICP			200.7	6010	6010	3120
950	Magnesium	ICP	200.7	200.7	200.7	6010	6010	3120
950	Manganese	ICP	200.7	200.7	200.7	6010	6010	3120
950	Molybdenum	ICP		200.7	200.7	6010	6010	3120
950	Nickel	ICP		200.7	200.7	6010	6010	3120
950	Potassium	ICP		200.7	200.7	6010	6010	3120
950	Selenium	ICP			200.7	6010	6010	3120
950	Silicon	ICP		200.7	200.7	200.7	200.7	3120
950	Silver	ICP	200.7	200.7	200.7	6010	6010	3120
950	Sodium	ICP	200.7	200.7	200.7	6010	6010	3120
950	Strontium	ICP	200.7	200.7	200.7	200.7	200.7	3120
950	Thallium	ICP			200.7	6010	6010	3120
950	Tin	ICP		200.7	200.7	6010	6010	3120
950	Titanium	ICP		200.7	200.7			3120
950	Vanadium	ICP		200.7	200.7	6010	6010	3120
950	Zinc	ICP	200.7	200.7	200.7	6010	6010	3120
960	Antimony	ICP			200.7	6010	6010	3120
960	Antimony	GFAAS	200.9	204.2	204.2	7041	7041	3113
960	Arsenic	GFAAS	200.9	206.2	206.2	7060	7060	3113
960	Cadmium	GFAAS	200.9	213.2	213.2	7131	7131	3113
960	Chromium, Total	GFAAS	200.9	218.2	218.2	7191	7191	
960	Copper	GFAAS	200.9	220.2	220.2	7211	7211	3110
960	Lead	GFAAS	200.9	239.2	239.2	7421	7421	3110
960	Selenium	GFAAS	200.9	270.2	270.2	7740	7740	3110
960	Silver	GFAAS		272.2	272.2	7761	7761	3110
960	Thallium	GFAAS	200.9	279.2	279.2	7841	7841	3110
970	Metals Preparation (Liquid)	Digestion	200.7,200.9	3010		3010		
980	Metal Preparation (Solid)	Digestion					3050	
103, 219	Chloride	Ferricyanide	325.2	325.2	325.2		9251	4500CL E
104, 222	Phosphorus, Ortho	Colorimetric	365.1	365.1	365.1			4500P F
158, 216	Nitrogen, Nitrate	Hydrazine Reduction	353.1	353.1	353.1			4500 NO3 H
160, 215	Nitrogen, Nitrite	Colorimetric	354.1	354.1	354.1			4500 NO2 H
162, 220	Nitrogen, Ammonia	Salicylate	350.1	350.1	350.1			
401,12	Extraction, Phenols	Extraction				3510,50	3510,50	
402,06,09	Extraction, Pesticides	Extraction				3510,50	3510,50	
403,23	Extraction, PNAs	Extraction				3510,50	3510,50	
404,05,08	Extraction, SVOCs	Extraction				3510,50	3510,50	
407,13	Extraction, Hydrocarbons	Extraction				3510,50	3510,50	
410,11,24	Extraction, Herbicides	Extraction				3510,50	3510,50	
414,15	Extraction, DRO	Extraction				3510,50	3510,50	
416,17	Extraction, TPH DRO	Extraction				3510,50	3510,50	
418,19	Extraction, TPH	Extraction				3510,50	3510,50	
421,22	Extraction, Pesticides	Extraction				3510,50	3510,50	
425,26	Extraction, PCBs	Extraction				3510,50	3510,50	

Table 5.2 Minimum QC Requirements							
SOP #	Analysis	Technique	TYPE	MIN %R	MAX %R	RSD	LEVEL
102	COD, Macro	Titrimetric	LCS	90	110	<10	2000
105	Sulfate	Gravimetric	LCS	90	110	<10	1374
106	Sulfur, Total	Gravimetric	LCS	90	110	<10	1374
107	pH	Electrometric	PROC	95	105	<5	4,10
110	Acidity	Titrimetric	PROC	95	105	<5	1225
111	Sulfate	Turbidimetric	PROC	90	110	<10	10
112	Alkalinity	Titrimetric	PROC	95	105	<5	1000
126	Hexavalent Chromium	Colorimetric	PROC	90	110	<10	0.2
128	Color	Color-Spec.	PROC	90	110	<10	100
129	Conductivity	Conductivity Bridge	PROC	90	110	<10	1408
134	Cyanide (AA)	Pyridine-Barbituric	PROC	90	110	<10	0.3
135	Cyanide	Flow Injection	LCS	90	110	<10	0.3
138	Fluoride	ISE	PROC	90	110	<10	1
141	Grease & Oil	Gravimetric	LCS	90	110	<10	40.1
143	Hardness	Calculation	PROC	90	110	<10	5
158	NO3+NO2,NO3-N	Colorimetric	PROC	90	110	<10	1
161	Nitrogen, TKN	Semi-Micro Kjeldahl	LCS	90	110	<10	5.0
166	COD, Mini	Colorimetric	PROC	90	110	<10	750
189	Turbidity	Nephelometric	PROC	90	110	<10	18
193	BOD	5 Day, 20C	LCS	167.5	228.5	<10	198
203	Anions	Ion Chromatography	PROC	85	115	<15	Varied
208	Solids	Gravimetric	LCS	90	110	<10	100
261	TKN	Flow Injection	LCS	90	110	<10	5.0
262	Phosphorus	Flow Injection	LCS	90	110	<10	3.0
263	Orthophasphate	Flow Injection	LCS	90	110	<10	3.0
264	NH3-N	Flow Injection	LCS	90	110	<10	5.0
265	Chloride	Flow Injection	LCS	90	110	<10	50.0
267	Phenols	Flow Injection	LCS	90	110	<10	0.1
269	Sulfides	Titrimetric	QCS	90	110	<10	Uk
270	TOC/NPOC	Tekmar/Dohrmann	PROC	90	110	<10	10
271	NO3+NO2-N (FIA-Lachat)	Flow Injection	PROC	90	110	<10	0.5
273	Chloride (ISE)	Ion Selective Electrode	PROC	90	110	<10	50
500	Volatiles	GC/MS	PROC	80	120	20	5
501	Semivolatiles	GC/MS	LCS	50	150	30	50
503	Phenols	Gas Chromatography	LCS	70	130	20	10 x MDL
505	Volatiles	Gas Chromatography	PROC	80	120	20	20
507	Pesticides (GC/ECD)	Gas Chromatography	LCS	70	130	20	10 x MDL
508	PAH	HPLC	LCS	70	130	20	10 x MDL
512	Diesel Range Organics	Gas Chromatography	LCS	75	115	20	100/10
513	Gasoline Range Organics	Gas Chromatography	PROC/LCS	60	160	20	100/10
515	Chlorinated Hydrocarbons	Gas Chromatography	LCS	70	130	30	10 x MDL
516	Herbicides	Gas Chromatography	LCS	70	130	30	10 x MDL
519	Carbamates	HPLC	LCS	80	120	20	10 x MDL
525	PVOC (GC/PID)	Gas Chromatography	PROC/LCS	80	120	20	20/0.1
530	EDB/DBCP	Gas Chromatography	LCS	70	130	20	10 x MDL
531	Pesticides, Organochlorine	Gas Chromatography	LCS	70	130	20	10 x MDL
537	PCBs in Oil (GC/ECD)	Gas Chromatography	LCS	70	130	20	10 x MDL
539	PCBs (GC/ECD)	Gas Chromatography	LCS	70	130	20	10 x MDL
540	OP Pesticides (NPD)	Gas Chromatography	LCS	70	130	20	10 x MDL
541	Organic Acids by HPLC	HPLC	LCS	70	130	20	mid-point
913	Mercury	A2S, Cold Vapor	LCS	90	110	10	mid-point
950	Metals	ICP	LCS	95	105	10	mid-level
960	Metals	GFAAS	LCS	90	110	10	mid-point

SECTION 6.0: INSTRUMENTATION AND CALIBRATION

USFilter, Enviroscan Services maintains the integrity of all laboratory instrumentation to ensure the highest quality analyses.

6.1 Instrumentation

It is the responsibility of the analyst to maintain instruments in working condition. If the analyst performing the testing is not technically qualified to perform instrument maintenance, the technical director is responsible for the instrumentation. Each instrument is cleaned on a regular basis and repaired when required. Each instrument purchased is assigned a unique code, which is used to identify it within the Laboratory Information Management System (LIMS). Laboratory instruments, manufacturer, model, instrument code, lab location, primary analysis and description are found in Table 6.1: Laboratory Instrumentation Description.

6.2 Maintenance

USFilter, Enviroscan Services has maintenance agreements with the manufacturer to complete preventive maintenance (PM) checks and/or emergency services of designated instruments. The analyst shall fill out a corrective/preventive action report (CPAR) as soon as possible when additional steps need to be taken to correct an instrument problem. The CPAR is submitted to the Laboratory Officer for corrective action.

6.2.1 Replacement Parts

To ensure that down time is kept to a minimum, an adequate supply of spare parts are kept in inventory by each analyst. Used instrument parts are to be disposed of properly.

6.2.2 Maintenance Log Book

Records of instrument repair and maintenance are updated in a bound journal located near the specific instrument and shall include the following information:

- Date of maintenance check
- Description of problem or reason for service
- Description of maintenance performed on instrument
- Verification of proper instrument function
- Analyst's initials

All affected analytical data shall be reanalyzed, flagged, and/or a Corrective/Preventive Action Report shall be filed by the analyst, see Section 8.

6.3 Calibration Procedure

Analysts shall monitor and evaluate a calibration curve daily to ensure the highest quality data. Calibration is the correlation of an instrument response to a known physical or chemical set of standards. A known concentration of target analyte is analyzed and the instrument response is recorded. A mathematical relationship between the instrument's response to the target analyte at different levels and the original known concentrations is then processed creating a multi-point calibration curve. Calibration equations used in the laboratory may employ one of the following models:

- Linear Regression: $y = mx + b$ (Beer's Law)
  Linear not forced through the origin (Zero can be used)
  Second Order Curve
  2^{nd} Order: $y = ax^2 + bx + c + d$
  3^{rd} Order $y = ax^3 + bx^2 + cx + d$

$R^2 > 0.995$

- External Standard Calibration
  Average Calibration Factors: CF = Response/Concentration
  %RSD < 10%
- Internal Standard Calibration
  $Rf = \frac{\text{Area TA x Conc IS}}{\text{Area IS x Conc TA}}$

Look to Table 6.2: Calibration Requirements, for the exact calibration technique employed on each method

6.3.1 Calibration - Inorganic Area
A minimum of four standards are used each working day to establish a linear calibration curve with a correlation coefficient of at least 0.995 or an average calibration factor with less than 10%RSD. Seven standards and a blank are required for 2nd or 3rd order curves with a minimum correlation coefficient of 0.999. As standard practice, the percent variance of each standard is reviewed by measuring the calculated value to the true. A ≤10% variance is acceptable in most cases. Variance should not exceed 20%. Standards for calibration are prepared and stored as specified in each method. Calibration curves are verified using initial and continuing calibration check standards immediately following calibration, prior to sample analysis, and following a batch and/or every 10 or 20 samples. The anion analysis by IC is verified after a batch and/or ten samples. The check standard response shall be within ± 10% of the true value.

6.3.2 Calibration - Metals Area

6.3.2.1 Graphite Furnace/Atomic Absorption Spectrometer (GFAAS)
A minimum of three calibration standards and a blank are used each working day to establish a linear calibration curve with a correlation coefficient of 0.995 for all metals analyses by atomic absorption. Working standards for calibrations completed on the GFAAS are prepared according to the appropriate methodology. Calibration curves are verified using initial and continuing calibration check standards immediately following calibration, prior to sample analysis, and following a batch and/or every 20 samples. The initial calibration verification must be within ±5% of the true value. Continuing calibration check standard response must be within ±10% of the true value.

6.3.2.2 Inductively Coupled Plasma Spectrometer (ICP)
A minimum of three calibration standards and a blank are used each working day to establish a linear calibration curve with a correlation coefficient of 0.995 for all metals analyses by ICP. Calibration curves are verified using initial and continuing calibration check standards immediately following calibration, prior to sample analysis, and following a batch and/or every 20 samples. The initial calibration verification must be within ±5% of the true value. Continuing calibration check standard response must be within ±10% of the true value. Standards for calibration shall be prepared according to the appropriate methodology.

6.3.3 Calibration - Organics Area
Three to five calibration standards are required to establish a linear curve with a correlation coefficient of 0.99, and a quadratic curve requires a minimum of seven standards with a correlation of 0.999. Standards are

prepared as needed, with a maximum holding time in a freezer of six months. Organic calibration procedures are specific for each compound group, see Table 6.2 for the following information:

- Compound group name
- EPA method
- Minimum number of Calibration Standards
- Frequency of calibration
- Verification of standard limits

TABLE 6.1: LABORATORY INSTRUMENTATION DESCRIPTION

Instrument	Manufacturer	Model	Instrument Code	Lab Location	Primary Analysis	
Analytical Balance	Fisher	A160	I1	Inorganics	Metal Prep	Fisher Analytical
Analytical Balance	Fisher	A160	I2	Inorganics	TS, SS, TDS	Fisher Analytical
Analytical Balance	Sartorius	BA1105	E1	Extracions	Oil and Grease	Sartorius Analyti
Top-Loader Balance	Mettler	PB1502	I3	Inorganics	Reagent Preparation	Mettler Top-Loa
Top-Loader Balance	Sartorius	BA210	L1	Log-In Lab	Vial,Sample	Sartorius Top-Lo
Top-Loader Balance	Sartorius	BP612	E2	Extractions	Reagent, Sample	Sartorius Top-Lo
Top-Loader Balance	Sartorius	B310S	V1	VOCs	Reagent, Sample	Sartorius Top-Lo
Refrigerators (9)				All Areas	Sample Storage	
Walk-In Refr.(1)				Inorganics	Sample Storage	
Freezers (2)				Ext, VOCs	Reagent Storage	
Incubators (3)			A,B,C	Inorganics	BOD	
Oven	VWR	1350FM	A	Inorganics	COD	Oven
Oven	Precision	114A	B	Inorganics	TS	Oven
Oven	Precision	104A	C	Inorganics	TDS	Oven
Furnace (3)	Lindberg	51441	A,B,C	Inorganics	Ash, Fusion	Furnace
Atomic Absorption	Perkin-Elmer	4100ZL	AA3	AA Lab	Metals and Mercury	Zeeman backgro printer, and Dec Flow injection A
Atomic Absorption	Perkin-Elmer	4100ZL	AA2	AA Lab	Metals	Zeeman backgro printer, and Dec
Atomic Absorption	Perkin-Elmer	FIMS100	FIMS1	Inorganics	Mercury	Perkin Elmer FI 810C printer, De monitor, and AS
ICP	SA Jobin Yvon	Ultrace 138	ICP2	ICP Lab	Metals	Gilson Autosam
Flow Injection Analysis System	LACHAT	QC8000	LACH1	Inorganic Lab	TKN, NH3, P, OP, CL, Phenols, Cyanide	XYZ Autosampl
Flow Injection Analysis System	LACHAT	QC8000	LACH2	Inorganic Lab	TKN, NH3, P, OP, CL, Phenols, Cyanide	XYZ Autosampl
Carbon Analyzer	Tekmar/ Dohrmann	Apollo	TOC2	Inorganic Lab	NPOC, DIC, TOC	XYZ Autosampl
FTIR Spec.	Perkin-Elmer	1600	FTIR	Inorganic Lab	Grease, TRPH	Equipped with c
Ion Chromatograph	Dionex	DX-120	IC	Inorganic Lab	Bromide, Chloride, Sulfate, Nitrate, Nitrite, Fluoride	SP4270 integrato
pH Meter	Thermo Orion	410A+	PH1	Inorganic Lab	pH	-
pH Meter	Orion	SA701	PH2	Inorganic Lab	BOD, CBOD	-
pH Meter	Orion	SA601A	PH3	Inorganic Lab	TCLP	-
pH Meter	Orion	SA720A	PH5	Inorganic Lab	Potentiometric Analyses	-

TABLE 6.1: LABORATORY INSTRUMENTATION DESCRIPTION

Instrument	Manufacturer	Model	Instrument Code	Lab Location	Primary Analysis	
Turbidimeter	Hach	18900	TURB	Inorganic Lab	Turbidity	Digital reading f Standardization
Spectrometer	Hitachi	U-1100	SPEC3	Inorganic Lab	Color, COD, and Hexavalent Chromium	Digital Reading
Conductivity Meter	Orion	126	COND	Inorganic Lab	Conductivity	Digital Reading
Flashpoint Tester	Pensky-Martens	-	FLASH	AA Lab	Flashpoint	Closed cup appa
Gas Chromatograph	Varian	3400	GC1	VOC Lab	Volatiles	Dynatech PTA-3 PID/Hall detect
Gas Chromatograph	Varian	3400	GC3	VOC Lab	Gas Phase Analysis	Packed column;
Gas Chromatograph	Varian	3400	GC5	SemiVOC Lab	Phenols & DRO	Two FID detect columns.
Gas Chromatograph	Varian	3400	GC6	SemiVOC Lab	PCBs	Two ECD detec columns.
Gas Chromatograph	Hewlett-Packard	5890	GC7	SemiVOC Lab	DRO, TPH	FID detectors; t capillary column
Gas Chromatograph	Varian	3400	GC10	VOC Lab	Volatiles	Capillary column
Gas Chromatograph	Varian	3400	GC11	SemiVOC Lab	Carbon Tubes,Volatile Acids,& miscellaneous VOCs	Equipped with 8 column.
Gas Chromatograph	Varian	3400	GC12	VOC Lab	Volatiles	Capillary column concentrator; PI
Gas Chromatograph	Finnegan	9001	GC14	VOC Lab	PVOC/GRO	Capillary column concentrator, an
Gas Chromatograph	Finnegan	9001	GC15	VOC Lab	PVOC/GRO	Capillary column concentrator; an
Gas Chromatograph	Varian	3400	GC16	SemiVoc Lab	N/P Pesticides	Two phase nitro Varian 8200 CX
Gas Chromatograph	Varian	3400	GC17	SemiVoc Lab	Herbicides, EDB, Chlorinated Hydrocarbons	Two ECD detec columns.;
Gas Chromatograph	Varian	3400	GC18	SemiVoc Lab	Pesticides	Two ECD detec columns.;
HPLC	Waters	-	HPLC	SemiVOC Lab	Carbamates and PAHs	Hitachi AS4000 programmable U Detector.
HPLC	Waters	-	HPLC2	SemiVOC Lab	PAHs	Waters 717+ aut 996 Photodiode Detector.
GC/MS	Finnegan	INCOS 50	GC/MS2	Lab A-1	Semivolatiles	Quadrapole with SUPERINCOS; software.
ICPMS	Perkin-Elmer SCIEX	ELAN	ICP-Ms	ICPMS Lab	Metals	Gilson Autosam
GC/MS	Finnegan	ITS 40	GC/MS3	VOC Lab	Volatiles	Ion-trap; Capilla version 2.1; AUT

TABLE 6.1: LABORATORY INSTRUMENTATION DESCRIPTION

Instrument	Manufacturer	Model	Instrument Code	Lab Location	Primary Analysis	
						software.
GC/MS	Finnegan	ITS 40	GC/MS5	VOC Lab	Volatiles	Ion trap; Capillar 2.0 software
GC/MS	Finnegan	Voyager	GC/MS6	VOC Lab	Volatiles	Ion trap; Capillar 2.0 software
GC/MS	Finnegan	Voyager	GC/MS7	Lab A-1	Semivolatiles	Quadrapole with SUPERINCOS; software.

Table 6.2: **Calibration Criteria**

ANALYSIS	Method	SOP Code	# of Stds	Blank in Curve	L, 2nd CF or RF	CC/R^2/RSD	Verification Frequency	Recovery Limits
Alkalinity	310.1	112	1	N	na	na	5%	±10%
Ammonia	350.1	264	4	Y	L	0.995	5%	±10%
Ammonia, Distillation	350.2	155	1	na	na	na	5%	±10%
BOD	SM5210 B	193	1	na	na	na	5%	167.5 - 228.5
CBOD	SM5210 B	193	1	na	na	na	5%	167.5 - 228.5
Bromide	300.0	203	4	Y	L	0.995	5%	±10%
Carbon(NPOC,TOC,POC)	415.1/SM5310 B	120	5	Y	L	0.995	5	±10%
Chloride	325.2	265	4,7	Y	2nd	0.995	5%	±10%
Chloride	300.0	203	4	Y	L	0.995	5%	±10%
COD, Macro	410.1	102	1	na	na	na	Daily	±10%
COD, Mini	410.4	166	4,7	Y	CF	<10%	5%	±10%
Color	110.2	128	4	Y	L	0.995	5%	±10%
Conductivity	120.1	129	1	na	na	na	5%	±1%
Cyanide (Total,Amenable,Free)	335.3/9012	135	4	Y	L	0.995	5%	±10%
Fluoride	340.2/SM4500 F C	138	3	Y	L	0.995	5%	±10%
Ignitability	1010	198	1	na	na	na	5%	± 1°C
Nitrogen, Nitrate	300.0	203	4	Y	L	0.995	5%	±10%
Nitrogen, Nitrite	354.1	160	5	Y	2nd	0.9995	5%	±10%
Nitrogen, Nitrate	353.1	158	5	Y	2nd	0.9995	5%	±10%
Nitrogen, Nitrite	300.0	203	4	Y	L	0.995	5%	±10%
Nitrogen, TKN	351.2	261	5	Y	L	0.995	5%	±10%
pH	150.1	168	2	N	Logarithmic	na	5%	±0.05 SU
Phenols	420.2/9066	267	4,7	Y	L 1/x	0.995	5%	±10%
Phosphorus, Ortho	365.1	263	4	Y	L	0.995	5%	±10%
Phosphorus, Total	365.4	262	4	Y	L	0.995	5%	±10%
Solids, Total Dissolved	160.1	208	2	N	na	3%,1%	5%	±10%
Solids, Total	160.3	179	2	N	na	3%,1%	5%	±10%
Solids, Total Suspended	160.2	178	2	N	na	3%,1%	5%	±10%
Sulfate	375.3	105	4	na	na	3%,1%	5%	±10%
Sulfate	375.4	111	3	Y	L	0.995	5%	±10%
Sulfate	300.0	203	4	Y	L	0.995	5%	±10%
Sulfide	376.1	188	na	na	na	na	5%	±10%
Sulfide	SM4500-S2- G	187	4	N	Logarithmic	0.995	5%	±10%
Sulfur, Total	375.3	106	4	na	na	3%,1%	5%	±10%
Turbidity	180.1	189	2	Y	L	1.0	5%	± 5%
ORGANIC								
VOC by GC	502.2/8021	505	5	N	RF	20%	5%	± 15%
Phenols by GC	604/8041	503	5	N	L	0.99	5%	± 15%
Pesticides by GC	508/608/8081	502/507	5	N	L	0.99	5%	± 15%

Table 6.2: Calibration Criteria								
ANALYSIS	**Method**	**SOP Code**	**# of Stds**	**Blank in Curve**	**L, 2nd CF or RF**	**CC/R^2/RSD**	**Verification Frequency**	**Recovery Limits**
EDB/DBCP	504.1	530	7	N	L	0.99	5%	± 20%
PCBs by GC	608/8082	539	5	N	CF	<10%	5%	± 15%
Nitrogen/Triazine Pesticides	8141	507	5	N	L	0.99	5%	± 15%
Chlorinated Hydrocarbons-GC	612/8121	515	5	N	L	0.99	5%	± 15%
Herbicides by GC	515.1/8151	516	5	N	L	0.99	5%	± 15%
Carbamates by HPLC	531.1	519	4	N	L	0.99	5%	± 20%
PAH/PNA by HPLC	550.1/610/8310	508	5	N	L	0.99	5%	± 15%
Semivolatile by GC/MS	8270	501	5	N	RF	<30,15%	12hr	± 15%
Volatiles by GC/MS	8260	500	5	N	RF	<30,15%	12hr	± 15%
TPH gas and Diesel	8015(DRO)	504	3	N	L	0.99	5%	± 20%
GRO	WI DNR	513	3	N	L	0.995	5%	± 20%
DRO	WI DNR	512	3	N	L	0.995	5%	± 20%
Oil & Grease by IR	413.2	202	5	N	L	0.999	5%	± 10%
TRPH by IR	418.2	514	5	N	L	0.999	5%	± 10%
LUST TRPH by IR	418.2	514	5	N	L	0.999	5%	± 15%
METALS								
Metals by ICP	200.7	950	3	Y	L	0.995	10%	±5/10
Metals by GFAAS	200.9	960	3	Y	L	0.995	10%	±5/10
Mercury by Cold Vapor AA	245.1	913	5	Y	L	0.995	5%	±10
Hexavalent Chromium	218.5	126	4	Y	L	0.995	5%	±10

SECTION 7.0: QUALITY CONTROL DATA EVALUATION

USFilter, Enviroscan Services evaluates quality control data based on regulatory laws, approved method control limits and historical laboratory data, to identify a number of different batch acceptance criteria. These most common acceptance criteria applied to analytical data are upper and lower control limits and warning limits, which are defined as follows:

Control Limit: A statistically determined value established at ±3 standard deviations from the average of 20 or more quality control data points.

Warning Limit: A statistically determined value established at ±2 standard deviations from the average of 20 or more quality control data points.

Data points outside the warning limit may indicate a developing problem. Three consecutive points between the warning and control limits shall be investigated. Data points beyond the control limit indicates the method is out of control and the problem must be addressed.

Numerous quality control samples are evaluated each day for each analysis against a number of set and calculated accuracy and precision control limits. This information is tabulated and maintained on benchsheets and/or in QC binders for each analysis area. The QC binders include the following information: Standard Operating Procedures; Method Validation; Method Detection Limit Studies, Check Standards; Laboratory Control Samples, Duplicates; Matrix spikes; Matrix spike duplicates; Surrogates; and other Miscellaneous information (i.e , QA/QC Reports, Audit replies, etc.).

Benchsheets and QC data shall include all pertinent information for easy access and quality control review. Analysis completed using the ACCESS*CHROM chromatography software system, utilizes an automatic computation of the quality control data, for volatile and semivolatile analysis by GC. The data is processed and completed using an RGEN extraction program linked to Microsoft Excel. Trends are observed by the amount of key analytes within the warning limits.

7.1 Data Review

The analysts are responsible for reviewing all quality control data prior to entering sample results into the LIMS database. Analysts are also responsible for keeping data quality control limits updated and organized for periodic review by the Technical Director and/or the QA Officer. A typical batch of samples analyzed by USFilter, Enviroscan Services must be evaluated using the following fundamental criteria (see SOP-083 for more detail):

Instrument – Performance All GC and GC/MS methods require Instrument Performance Checks to be performed on a daily basis prior to and immediately following sample analysis.

a. Initial 5-Point Calibration Curve (when analyzed)
b. Initial Calibration Verification (when analyzed)
c. Continuing Calibration Verification
d. Instrument Degradation Check (Pesticide Analysis)
e. Internal Standard Area Check
f. Retention Time Check
g. Instrument Blank

Sample Prep – Sample preparation QC checks are performed on a daily basis to confirm that the entire analytical process is in control.

a. Method Blank
b. Laboratory Control Sample (LCS)

c. Matrix Spike/Matrix Spike Duplicates
d. Surrogate Spikes
e. Cleanup Blanks (when cleanup procedures are performed)

7.2 Data Evaluation

Method or program specific quality control limits are used directly from the most recent method publication. These limits are typically only available for calibration criteria and calibration verification standards. Some methods do establish default control limits for some matrices. For most quality control parameters, in-house limits are established according to matrix type or expected analyte concentrations. Limits based on different matrices are completed for all analysis areas. These limits are established according to the following matrix types: drinking water, ground water, wastewater, soil/solid/sludge, and TCLP, if applicable. Limits based on various concentration levels are completed for inorganic and some metals analyses (ex. influent vs. effluent control limits for BOD_5). The concentration levels are established for each analysis depending upon sample matrices. New limits are calculated once per year or when twenty data points are accumulated for infrequent analysis. Default limits may be used when an adequate amount of data points cannot be recorded. Default limits can also be used when calculated limits are unreasonable (i.e. a negative percent recovery, 3% RPD, etc.). When developing in-house limits, the analyst is encouraged to use their professional judgment along with a Q-test to eliminate any obvious outlier points. The QA Manager, prior to instituting new control limits into the analytical area, reviews in-house control limits. At this time, the QA Manager is responsible for making sure that the control limits meet method specific requirements.

7.2.1 Initial/Continuing Calibration Verification (ICV and CCV)

An ICV check standard is prepared from a different lot number and/or vendor when available, and is used to confirm the accuracy of the initial calibration curve. The CCV, which can be of the same lot, is used as an ongoing daily check of the calibration stability. The concentration of both should be at or near the middle of the working calibration range of the method or the instrument. However, on a routine basis, the level of the CCV must be varied to verify that the entire working range of the calibration curve is functioning properly. The standard is analyzed immediately after the calibration curve. The ICV check standards are calculated as percent recovery:

$$\frac{\text{Measured Concentration of Standard}}{\text{Theoretical Concentration of Standard}} \times 100 = \text{Percent Recovery}$$

7.2.2 Laboratory Control Sample (LCS)

A LCS is a laboratory blank that is spiked with a known amount of standard. The spike is then brought through any digestion or preparation method in the same manner as a sample. A LCS must be analyzed with every batch of samples that undergo a preparation procedure. This sample allows us to evaluate our ability to perform the analysis on a sample that is free of matrix interference. Acceptance limits vary with each method. The LCS is calculated as percent recovery:

$$\frac{\text{Measured Concentration of Standard}}{\text{Theoretical Concentration of Standard}} \times 100 = \text{Percent Recovery}$$

7.2.3 Duplicate

A duplicate is prepared by dividing a sample into two identical aliquots and analyzing them within the same batch. Duplicates are only used for those analytes that do not lend themselves to matrix spiking. Duplicates are completed for each matrix at a set frequency of 5%. A minimum of one duplicate shall be completed per analytical batch for all areas of the laboratory and calculated using relative percent difference rather than absolute values due to the wide range of concentrations analyzed. Duplicate results are evaluated as follows:

$$\frac{\text{(Duplicate 1 - Duplicate 2)}}{\text{(Duplicate 1 + Duplicate 2)/2}} \times 100 = \text{Percent Difference*}$$

*Zero percent difference may be used in the calculation of limits if both results are greater than the detection limit. The default percent difference for all analyses is 25%.

7.2.4 Matrix Spike (MS)

A matrix spike is a sample prepared prior to analysis with a predetermined quantity of stock standard and should be in the middle to upper half of the calibration range. A matrix spike assesses recovery of the analytical system on actual sample matrices. Matrix spikes are analyzed for each matrix at a set frequency. A minimum of one spike shall be analyzed with each analytical batch. Matrix spikes are evaluated at follows:

$$\frac{\text{(Spiked sample concentration - Sample concentration)}}{\text{Theoretical spike concentration}} \times 100 = \text{Percent Recovery}$$

7.2.5 Matrix Spike/Matrix Spike Duplicate

Samples, identically spiked, are referred to as a matrix spike/matrix spike duplicate (MS/MSD.) A matrix spike duplicate ensures that both precision and recovery are monitored for all requested compounds in each analytical batch. Matrix spike duplicates are used in most areas and are evaluated as follows:

Recovery Evaluation:

MS: $\frac{\text{(Spiked sample concentration - sample concentration)}}{\text{Theoretical spike concentration}} \times 100 = \text{Percent Recovery (A)}$

MSD: $\frac{\text{(Spiked sample concentration - sample concentration)}}{\text{Theoretical spike concentration}} \times 100 = \text{Percent Recovery (B)}$

Precision Evaluation: Relative Percent Difference

$$\frac{\left|\text{Concentration of Spike (A)} - \text{Concentration of Spike (B)}\right|}{\frac{\text{Concentration of Spike (A)} + \text{Concentration of Spike (B)}}{2}} = \text{Percent Difference}$$

Note: Matrix spike duplicates follow the same protocol concerning matrix types and daily evaluation as given in previous sections.

7.2.6 Surrogate

A surrogate is a compound similar in chemical composition and chromatographic response as the analytes of interest. A surrogate is spiked into every sample including standards, blanks, and quality

control samples. Surrogates are used to monitor the entire analytical system including the extraction, digestion, and analysis process. Surrogates are typically used in the following areas: Semivolatiles by GC and GC/MS, Volatiles by GC and GC/MS, Polynuclear Aromatic Hydrocarbons by HPLC, and Wisconsin Modified Diesel and Gasoline Range Organics. Surrogates are evaluated as follows:

$$\frac{\text{Measured Concentration of Surrogate}}{\text{Theoretical Concentration of Surrogate}} \times 100 = \text{Percent Recovery*}$$

7.2.7 Internal Standards

An Internal Standard is a compound similar in chemical composition and chromatographic response as the analytes of interest. An internal standard is spiked into every sample including standards, blanks, and quality control samples. Internal Standards are used to monitor the entire analytical system including the extraction, digestion, and analysis process. Data results are adjusted based on the internal standard result. Internal Standards are used in the following areas: Semivolatiles GC/MS and Volatiles by GC and GC/MS.

Internal standards evaluated under EPA 502.2 for volatiles by GC use the area counts obtained in the internal standards analyzed in the same analytical batch. The internal standard area of each sample is acceptable if within ± 3 standard deviations of the internal standard area counts and between -50 to 100 percent with a retention time ± 30 seconds from the last calibration.

7.2.8 Default Limits

Default limits are a guideline used until a sufficient number of data points are established for each analyte, or the calculated limits are greater than the default limits established by an approved method or by a regulatory agency (Wisconsin Administrative Code NR149.) Default limits are typically established according to actual laboratory performance on a particular matrix.

Most EPA methods for organic analyses list a default limit for check standards and spikes but not duplicate limits. The EPA inorganic methods list default limits for check standards on some analytes. Refer to the following guidelines to determine which default limits to use:

1) EPA method limits take precedence.
2) Use the most stringent limits for identical methods.
3) when EPA or other method limits do not exist, USFilter, Enviroscan Services has established the following limits, based on laboratory experience:

Check Standards, Inorganic	true ± 10%;	Duplicates, Organic	zero + 25%
Check Standards, Organic	true ± 20%;	Spikes, Inorganic	true ± 25%
Duplicates, Inorganic	zero + 25%;	Spikes, Organic	true ± 50%

7.2.8.1 Default Limit Rules

- Data exceeding default limits shall be flagged and corrective action taken immediately.
- Default limits shall be used to evaluate data when check standard values are beyond default limits.
- Default limits shall be used if either the upper or lower control limit is beyond the default limit.

SECTION 8.0: CORRECTIVE/PREVENTIVE ACTION REPORT

Reporting data of the highest quality is of primary importance to USFilter, Enviroscan Services, but because quality for the laboratory is based on mathematical statistics of the norm, occasionally quality control data exceeds established control limits. A Corrective/Preventive Action Report (CPAR) is completed by the appropriate personnel to document corrective action taken for a noncompliance. Any deviation from the following must be approved by management. In the event that an employee observes unusual data or anomalies for which there is no predetermined corrective action, the employee must bring it to the attention of the Supervisor and QA manager.

8.1 Determination of Out-of-Control Data and Corrective Action

A method shall be developed for each type of matrices (i.e. waste water, drinking water, solid waste, etc.) for lab use in analysis of samples to reduce matrix interference problems. Samples are randomly selected for analysis as duplicates, spikes, matrix spikes, internal standards or surrogates and are analyzed to meet method protocol. A sample which exhibits abnormal characteristics when analyzed by the appropriate method is deemed to be out-of-control. The following corrective action steps shall be taken for data points exceeding pre-established control limits:

8.1.1 Calibration Standard

1. Find possible causes for the problem (i.e. instrument wear, solvent or standard contamination, standard degradation. Etc.) and reanalyze the standard.
2. Recalibrate. A non-linear fit may be used if method allows and a sufficient number of standards have been analyzed.
3. Reanalyze all effected samples, when available, or qualify the results by flagging the data and documenting appropriately on bench sheets to maintain traceability, see Qualifier Description.
4. File a Corrective/Preventive Action Report, Form 8.1 if calibration remains out-of-control and data needs to be reported.

8.1.2 Check Standard

1. Prepare a fresh standard and rerun effected samples.
2. Recalibrate the instrument and rerun the check standard.
3. Find possible causes for the problem (i.e. instrument wear, solvent and standard preparation or degradation.)

NO ANALYSIS MAY BE COMPLETED UNTIL PROBLEM IS SOLVED.

If problem cannot be solved, report it to your Supervisor immediately and file a CPAR.

8.1.3 Duplicate; Matrix Spike; Matrix Spike Duplicate

1. Reanalyze the quality control (QC) parameter, if sample quantities allow.
2. Prepare the QC parameter and rerun. Continue to analyze samples if within limits.
3. Find possible causes for the failure.
4. Qualify data by flagging and documenting appropriately on bench sheets and client's reports to maintain traceability.

8.1.4 Surrogate or Internal Standard

1. Find possible causes for failure in various areas (i.e., extraction log, and original sample description), and document.
2. The areas of analysis and extraction shall be notified immediately and the problem identified and corrected when numerous surrogates are out-of-control within the same run, file CPAR.
3. Check instrument operation and/or investigate the matrix when the internal standard is out-of-control.
4. Qualify out-of-control data by flagging and documenting appropriately on bench sheets and client's report to maintain traceability, see Section 8.3.
5. Do the following checks for information on the instrumentation malfunction affecting analytical data.
   a. Refer to the owner's manual and appropriate instrument log to determine the cause of the malfunction.
   b. Document the repair in the instrument log book.
   c. Check all instrument systems and recalibrate, if necessary.
   d. Reanalyze samples effected by the malfunction, if possible.
   e. Qualify out-of-control data by flagging and documenting appropriately on bench sheets and customer's report.
   f. Contact your supervisor immediately, and file a CPAR if you are unable to properly document on the benchsheet or log book.

8.2 Corrective/Preventive Action Reports - Review/Approval Process

Any employee of USFilter, Enviroscan Services may initiate a corrective/preventive action request. The following personnel are responsible for Corrective/Preventive Action Reports review/approval process.

8.2.1 Analyst

The analysts are responsible for investigating and initiating corrective actions in the laboratory to rectify any out-of-control situation, detailed above. The Analysts shall complete a CPAR, if necessary, and submit it to their Supervisor or Laboratory Officer for review/approval as soon as possible.

8.2.2 Supervisor/Technical Director

The Supervisor or Technical Director shall review/approve any immediate corrective action taken by the analyst and submit the form to the QA Officer.

8.2.3 Quality Assurance Officer

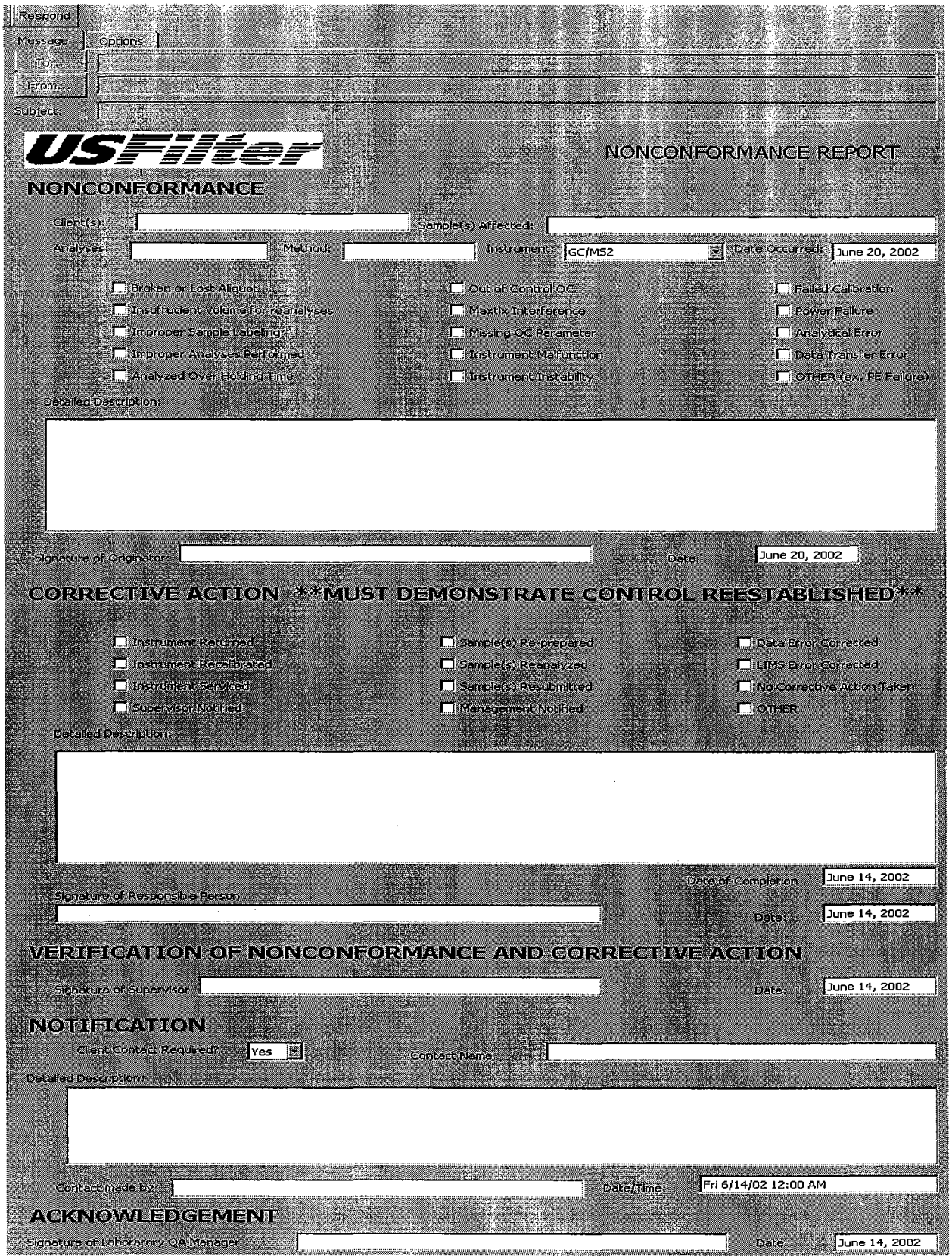
The QA Officer shall assign a number to the CPAR, review requests and implementations of corrective actions. Also, the QA Officer shall initiate a CPAR for non-compliances found during internal audits. The completed CPAR shall be stored in the quality assurance department.

8.3 Qualifier Description

Qualifiers are abbreviations describing the reasons why data may be out-of-control and are used to flag data on bench sheets and client's reports. Examples of qualifiers are listed below. For a full list of qualifiers, see Appendix D.

CSH =Check Standard high
CSL = Check Standard low
RB = Detect in reagent blank
S1H = First spike of MS/MSD high
S1L = First spike of MS/MSD low
IB = Detect in instrument blank

Form 8.1: Corrective Action Form



SECTION 9.0: PEFORMANCE EVALUATION STUDY PROGRAMS

USFilter, Enviroscan Services participates in performance evaluation studies (PE) from external organizations and contracted suppliers. Also, an internal study is completed at a minimum of three times per year. The laboratory receives samples of unknown results and analyzes them according to the appropriate methodology. The results are submitted and statistically evaluated by the provider based on the true value and values obtained by other participating labs to ensure continued performance at an acceptable competency level.

9.1 Performance Evaluations (PE) - External

To meet NELAC Chapter 5 program policy requirements, external performance evaluation studies consisting of 2 - Water Supply (Potable), 2-Water Pollution (Non-Potable), 2 Solid Matrix and 2 Underground Storage Tank studies are performed per year. Upon satisfactory completion of the performance evaluation programs NELAC grants the laboratory certification for the acceptable analytes. Certification is issued by the state of Wisconsin when an acceptable PE result is received for each category that the laboratory is currently holding certification for. Typically, successful completion of one WP and one WS study will fulfill the Wisconsin certification requirements.

9.1.1 Performance Evaluation Sample Distribution

USFilter, Enviroscan Services receives PE samples for analyses from an NIST approved supplier noted in NR 149. The QA Officer shall introduce the samples into the analytical process, provide the necessary instructions to the analysts, review results, and complete the report. The analyst shall prepare the sample, if required, analyze and interpret the data, and report the results in a timely manner.

9.1.2 Performance Results

USFilter, Enviroscan Services receives the PE evaluations from an NIST approved supplier. All participating laboratory personnel shall be given a copy of the results. A corrective/ preventive action report (CPAR) shall be completed and submitted to the QA Officer for each analyte that was not in the acceptable range. The Laboratory Officer shall review and complete each CPAR, if appropriate. A copy of the CPAR is sent to the external organization providing the samples, if required. USFilter, Enviroscan Services's performance results may be obtained upon request.

9.2 Potable Water (WS) Laboratory Performance Evaluation Study

USFilter, Enviroscan Services maintains certification in the Safe Drinking Water Program. Regulated and unregulated compounds are analyzed twice per year using the appropriate drinking water methods. Certification is granted through the State of Wisconsin under the provision of chapter NR149 of the Wisconsin Administrative Code. For an updated list of regulated compounds see NR 809.

9.3 Non-potable Water (WP) Laboratory Performance Evaluation Study

USFilter, Enviroscan Services participates in two Water Pollution Laboratory Performance Evaluation Studies per year. The program involves samples similar in concentration to plant discharges and other waste waters.

9.4 Solid Matrix (SM) Laboratory Performance Evaluation Study

USFilter, Enviroscan Services participates in two Solid Matrix Laboratory Performance Evaluation Studies per year. The program involves samples similar in analyte concentration and matrix effects of soil samples analyzed by the laboratory.

9.5 Environmental Reference Sample Program

USFilter, Enviroscan Services shall maintain certification in the State of Wisconsin by successfully completing one set of WP reference samples as previously mentioned. Proficiency samples are submitted to the laboratory and shall be completed accurately for each test category in which certification is desired. Categories are defined in chapter NR149 of the Wisconsin Administrative Code. USFilter, Enviroscan Services is certified in the following categories, see Table 9.1: Wisconsin Environmental Reference Sample Program.

9.6 Performance Evaluations - Internal

Internal (intralaboratory) performance studies consist of periodic assessments of the laboratory's and individual analyst's performance. USFilter, Enviroscan Services laboratory personnel analyze certified standards from an external source in which the concentration of the analyte is unknown to the analyst but is known to quality management. These studies, termed "blinds," are completed at least three times per year. A blind may also be introduced to any area for quality checks due to training, cross-training, instrumental or analysis quality issues.

The QA Officer introduces all samples for the blind program into the analytical process. Analysts shall treat the blind samples the same as routine samples to measure the overall quality of the laboratory's performance. The analyst shall receive the true value and range of each analyte upon completing the analysis. A CPAR shall be completed for the analytes that are not within the certified control limits. The CPAR shall be submitted to the QA Officer and Lab Officer for review and approval as soon as possible to address and correct any problems.

The analysts shall use the following checks to assist them in finding the problem:

- Sample preparation
- Reagents and standards; Check expiration dates and preparation
- Instrumentation; Check log
- Benchsheets; Check data transfer and calculations
- Control Limits; Check QC data
- Re-analyze sample

A new blind shall be introduced to any area with unacceptable results below 80% for an analyte group or when an acceptable reason for failing the study is not determined. This process will be repeated until an acceptable result can be obtained. The QA Officer shall discuss with management any trends and possible improvements to ensure lab quality. Quality records are maintained by the QA Officer. See Table 9.1: Wisconsin Environmental Reference Sample Program, for a full list of parameters:

Table 9.1: Wisconsin Environmental Reference Sample Program

Category Number:	Parameters:
1 - Oxygen Utilization	BOD and CBOD
2 - Nitrogen	Nitrate, Nitrite, Ammonia, and TKN
3 - Phosphorus	Orthophosphate, Phosphorus
4 - Physical	Total Solids, Dissolved Solids, Volatile Solids, Total Suspended Solids, Oil/Grease
5 - General I	Alkalinity/Acidity, Bromide, Chlorophyll a, Color, Hardness, Silica, Silicate, Sulfide, Sulfite, Surfactants
6 - General II	COD, Chloride, Cyanide, Fluoride, Sulfate, Total Phenolics
7 - General III	EP Toxicity, Ignitability, Reactivity, Waste Fingerprinting Analyses, TCLP, Total Organic Carbon, Total Organic Halide
8 - Metals I	Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead , Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, and Vanadium.
9 - Metals II	Bismuth, Gold, Lithium, Rhodium, Silicon, Titanium, Tungsten, Zirconium
10 - VOC by GC & GC/MS	Purgeable Halocarbons, Purgeable Aromatics
11- Base/Neutral	Benzidine, Phthalate Esters, Nitrosamines, Nitroaromatics, Isophrone, PAH, Haloethers, by GC/MS Nonpurgeable Chlorinated Hydrocarbons
12 - Acid Extractables by GC & GC/MS	Phenolic Compounds
13 - Extractables by HPLC	Polynuclear Aromatic Hydrocarbons and Carbamates
14 - Pesticides	Acid Extractable 2,4-D, 2,4,5-T, Dinoseb, Picloram, etc.
15 - Petroleum Hydrocarbons	GRO, DRO, PVOC, TRPH
16 - Organochlorine Compounds	Polychlorinated Biphenyls and Organochlorine Pesticides
17 - Dioxin	Not Certified
18 - Safe Drinking Water	See WS parameters

SECTION 10.0: LABORATORY AUDITS

USFilter, Enviroscan Services perform several different types of audit procedures. Some audits are directed at the administrative operations of the laboratory and others are geared more towards the daily technical processes. Both are key parts to smooth daily operations. Outlined in this section are USFilter's audit procedures.

10.1 Internal Audits

USFilter, Enviroscan Services conducts three types of internal audits; process, system, and training. The process and system audits are performed on the same project set due to the overlapping of information. The report is in two sections, process and data validation. Audits are completed by the QA Officer and typically take 4-5 days, depending upon the analyses area. A report is generated by the auditor detailing observations and findings with recommendations for corrective action. The report is distributed to the Laboratory Director, Technical Director, Area Analyst, Technicians and all other appropriate personnel. It is the responsibility of management to find the root cause of any problems and take corrective action. The QA Officer will do a follow-up audit to ensure that any corrective actions are being implemented.

10.1.1 Process Audit (see attached audit checklist for 10.1)

A process audit is an evaluation of our analytical method and how well it adheres to EPA or Standard Method procedures. Data is reviewed and the analyst is interviewed and observed. All personnel and their designated areas to be audited shall be notified in writing at least one week prior to the audit. A checklist shall be used by the Quality Assurance Officer to conduct the audit and shall be prepared prior to the audit. The following areas shall be evaluated:

10.1.1.1 Control Charts and Tables

Check standards, duplicates, matrix spikes, matrix spike duplicates, and surrogates are reviewed and evaluated for the following parameters to ensure;

- Control limits are properly updated.
- Quality control samples are within the control limits.
- Standard limits are within default or need to be recalculated.
- Duplicates, matrix spikes, and matrix spike duplicates are analyzed and updated according to specific requirements.
- Quality control data is updated, properly recorded, and easily retrievable.
- Trends, which signify potential problems in quality control, are addressed.

10.1.1.2 Standard Operating Procedures

Standard Operating Procedures are designated SOP with a three digit number (e.i., SOP110-Acidity) and are updated on a yearly basis or as needed. Procedures shall be followed in accordance with government regulations. See section 5 for specific SOP requirements. The SOPs are reviewed by the analyst and QA prior to the internal audit. Inconsistencies are noted and the SOP or testing process is revised after the audit is completed.

10.1.1.3 Standard and Reagent Log Book

Standards and reagents shall be certified and traceable to NIST standards and recorded in the appropriate standard logbook for each area. The logbook shall contain the following information:

- Name of reagent or standard

- Name of vendor
- Lot number
- Received date
- Expiration date
- Date of disposal
- Initial weight or volume of stock
- Final volume
- Solvent
- Calculated final concentration
- Signature of the person preparing the solution, and date prepared.

Labels on the reagent and standard bottles shall be coded for reactivity, flammability, and health hazards (see MSDS) and cross-referenced to the appropriate logbook. During the internal audit a clear trail for NIST traceability must be established and detailed in the audit report.

10.1.1.4 Instrument Maintenance Log Book
Review instrument maintenance logs to determine the following:

- Instrument is well maintained; records are updated properly.
- Instrument replacement parts are on hand to minimize downtime.
- Instrument calibration reviewed for accuracy.

10.1.1.5 Bench Sheets and Data
Review bench sheets and data for organization, completeness, and proper identification of information for each analysis, including the following:

- Method referenced
- Analyst's identification
- Date of analysis
- Sample identification number; Holding times
- Calculations (i.e., dilution factors, measured responses, and recoveries.)
- Method blank, check standard, duplicate, matrix spike and matrix spike duplicate analysis.
- Calibration checked for sufficient number of standards to calculate a good curve fit.
- Data was transferred into LIMS within a reasonable amount of time.
- Identify improper cross-outs.
- Evidence of peer review for data that is not automatically transferred into LIMs.

10.1.1.6 Final Reports
USFilter, Enviroscan Services shall report to the customer all pertinent information accurately and concisely. The reports shall be submitted to the customer by the designated due date, or the customer shall be notified. When reviewing the final report during the internal audit, QA checks for the following:

- Consistent Sample Identification throughout the analytical process.
- Report recipient consistency.

- Consistent and accurate reporting of analytical results.
- Date and time correspondance.
- Holding time requirements met.
- Appropriate reporting of data qualifiers.
- Results reported are clear and comprehensible.
- Data anomalies and qualifiers are explicit.

10.1.2 System Audit (see attached checklist Form 10.1)
A system audit is an assessment of the overall operation of the laboratory with the written plan for quality assurance/quality control. A customer report is selected at random and followed through the system from receipt of samples to report generation. The audit focuses on areas needing improvement and determines the accuracy of the final product. Analyte data is checked per batch for receipt of each sample, designated holding time, quality control checks, and final report generation. This audit is performed in tandem with the process audit due to the similarities of the data being reviewed.

10.1.3 Training and Cross-training Audits
Training and cross-training audits are designed to review a new analyst in an area of the laboratory. Predetermined questions relating to the area of interest are presented to the new analyst shortly after the analyst has completed a demonstration of capability study.

10.2 Audit Report
The QA Officer shall document all observations and findings and submit an audit report to the appropriate areas and to management. The report shall include the following:

- Significant observations and findings of the audited areas.
- Corrective Action Requests shall include specific noncompliance issues with suggestions on corrective action.

10.3 Audit Records
The Quality Assurance Officer shall be responsible for maintaining all audit records. These records shall include, but are not limited to, checklists used during the audit, documentation of observations and findings of the audit, as well as all corrective action reports.

10.4 Corrective/Preventive Action Report (CPAR)
Corrective/Preventive action reports from previous audits shall be reviewed by the Quality Assurance Officer prior to the audit. The CPARs may assist the auditor in reviewing the corrective action and implementation of any problem area. All audit corrective/ preventive action requests shall be signed and dated by the QA Officer along with recommendations for corrections and submitted to the Laboratory Officer for review. The CPARs shall be included in the audit report along with the tentative due date for completion.

10.5 External Audits
External audits are completed approximately every two years by the State of Wisconsin Department of Natural Resources and every two years by the National Environmental Laboratory Accreditation Program. The audits are administrative and technical systems audits which are typically conducted over a period of 3 days. The results of the audit are evaluated according to state regulation NR149 to determine laboratory

compliance or the NELAC Chapter 5 Quality Systems Manual. Other external audits including those from the Environmental Protection Agency, ENOVIS and potential clients are performed upon request.

10.6 New Work Acceptability Audit.

USFilter, Enviroscan Services is frequently involved in reviewing the potential for new projects. During this period, several factors are considered prior to acceptance of any new work or client. These factors include, but are not limited to:

- Is the waste compatible with our facilities? Samples that are extremely hazardous (radioactive, biological hazard, carcinogen, etc.) will not be accepted if we have any doubts about our ability to handle the material safely.
- Are the tests requested within our capabilities, and if not, do we have an acceptable subcontractor for the tests we cannot complete.
- Does the testing require specific certifications or accreditation, and if so, do we have current status with the certifying or accrediting authority.
- Does the client have adequate credit.
- Any client specific contract requirements are reviewed and assessed.
  a. Contract reviews are performed initially by the Sales Department personnel.
  b. Differences between the client and USFilter are documented (if necessary, the USFilter legal department will evaluate the contract).
  c. If the contract is agreed upon, it will be signed, copied, and kept on file.
  d. Project management is then notified of the requirements.

Any work request or sample, which does not meet our acceptance policy, will be rejected by the Log-In Technician. Requests of a questionable nature shall be brought to the attention of management for a decision on acceptance. The Laboratory Director, Technical Sales Manager or Customer Service Representative reserves the right to reject any sample.

10.7 Management Review

The quality system of USFilter, Enviroscan Services is reviewed by Supervisors, Managers and the Laboratory Director, as information is available. The information routinely reviewed includes, but is not limited to:

- All audit reports issued by regulatory agencies and clients
- All performance standard results
- All internal blind standard results
- All analyst *Demonstration of Capability Study* forms
- All internal audit reports.
- Any Corrective Action reports that the QC Manager deems important; recurring, indicating a systematic problem; or unusual in nature.
- Internal water quality results
- TCLP blank results
- All justifications for new equipment or procedures

A monthly report, which includes a Quality Assurance section, is also prepared by the Laboratory Director for the General Manager of the local business center.

Audits are performed periodically to evaluate the quality assurance program of USFilter, Enviroscan Services for compliance with Wisconsin Department of Natural Resources 149, the United States Environmental Protection Agency, National Environmental Laboratory Accreditation Program and any other regulatory agencies. It is the goal of the quality assurance department to complete a process or system audit for each analysis area on a rotational basis per year. Training audits shall be completed on an as needed basis. If problems are found in a particular area or method, additional follow up audits and/or inspections will occur until the problem has been resolved.

Form 10.1 Quality Audit Checklist

Date:_____ **Auditor:**_____

Report Date:_____ **Report Writer:**_____

Analyst:_____ **Analytical #'s:**_____ - _____

Analysis:_____ **Method ID:**_____

Matrix:_____ **Instrument:**_____

Client: _____ **Batch:** _____

Method Specifics (to be filled out by auditor prior to audit):

*Applicability:*_____

*Number of analytes:*_____

*Method Validation:*_____

*QC Check Standards/Samples:*_____

*Method Detection Limit:*_____

*Standard Solution Expiration:*_____

*Initial Calibration:*_____

*Continuing Calibration:*_____

*Surrogate Standards:*_____

*Internal Standards:*_____

*Accuracy/Precision:*_____

*Blanks:*_____

*Preservation/Storage:*_____

*Holding Time:*_____

*Field Sample Amount:*_____

*Amount for Extraction:*_____

*Other Criteria (Method Specific):*_____

*Comments:*_____

Category I: Control Requirements

A: Method

1. Is the analyst applying the most recent version of a valid EPA method: ☐ Y ☐ N
2. Is an SOP method written for this procedure: ☐ Y ☐ N
3. What is the SOP identification #: _____
4. Copy of the SOP accessible to all personnel: ☐ Y ☐ N
5. Is the SOP up to date and accurate: ☐ Y ☐ N
6. Effective date of the SOP: ____/____/____
7. Date of Last Revision: ____/____/____
8. Does the SOP addresses the following Components?
   - ☐ Identification of the test method
   - ☐ Applicable matrix or matrices
   - ☐ Method detection limit
   - ☐ Scope and application, including components to be analyzed
   - ☐ Summary of the test method
   - ☐ Definitions
   - ☐ Interferences
   - ☐ Safety
   - ☐ Equipment and supplies
   - ☐ Reagents and standards
   - ☐ Sample colletction, preservation, shipment and storage
   - ☐ Quality control requirements
   - ☐ Calibration and standardization requirements
   - ☐ Detailed Procedure
   - ☐ Calculations
   - ☐ Method performance
   - ☐ Pollution prevention
   - ☐ Data assessment and acceptance criteria for quality control measures
   - ☐ Corrective actions for out-of-control data
   - ☐ Contingencies for handling out-of-control or unacceptable data
   - ☐ Waste management
   - ☐ References; and
   - ☐ Any tables, diagrams, flowcharts, and validation data.
9. Was the SOP approved by the analyst and QA/QC Manager: ☐ Y ☐ N

Comments:_____

B: Method Validation

1. Was the cited method validated by the current analyst: □ Y □ N ____/____/____
2. Is the analyst training file up to date and contain the following documentation:
   - □ Certification that the analyst has read, understood, and agreed to perform the most recent version of the test method.
   - □ Documentation of continued acceptable proficiency. (Single Blind, DoC, 4 acceptable LCS samples)
3. Is a valid MDL study available: □ Y □ N ____/____/____
4. Is a current LDR study available (when required): □ Y □ N ____/____/____
5. Is the LDR and MDL data acceptable and easily retrieved: □ Y □ N

Comments:_____

C: Instrument Calibration

1. Was the calibration performed using an average calibration factor, response factor, linear calibration or quadratic regression: _____
2. Is the following information easily obtained from the calibration sheet: □ Y □ N
   - □ Standard Concentrations
   - □ Instrument response
   - □ Slope
   - □ Intercept
   - □ Correlation coefficient
   - □ Response Factor
   - □ %RSD
   - □ Curve identification
3. What is the minimum Correlation Coefficient or %RSD requirement: _____
4. Was the minimum criteria met for all analytes: □ Y □ N
5. What is the minimum number of standards allowed for initial calibration: _____
6. Was the minimum criteria met for all analytes: □ Y □ N
7. Are corrective actions performed if the results of the initial calibration are outside of established acceptance criteria: □ Y □ N
8. Is data associated with unacceptable instrument calibration reported: □ Y □ N

9. What is the calibration range of the instrument: _____

10. Was the lowest calibration level above the detection limit of the instrument: ☐ Y ☐ N

11. Was the calibration verified with a standard from a second source: ☐ Y ☐ N

12. Were all calculations performed correctly: ☐ Y ☐ N

13. Is the following raw data retained to allow for calibration reconstruction: ☐ Y ☐ N

- ☐ Analyst's Name
- ☐ Calibration Date
- ☐ Test Method
- ☐ Instrument I.D.
- ☐ Analysis Date
- ☐ Each analyte name
- ☐ Analyte Concentration
- ☐ Analyte Response
- ☐ Curve Correlation Coefficient or Response Factor

14. Were manual integrations performed on the calibration data: ☐ Y ☐ N

15. Was the following information documented for the manual integration: ☐ Y ☐ N

- ☐ Raw data output of the final integration (i.e., chromatogram of peak).
- ☐ Notation of rationale.
- ☐ Data.
- ☐ Signature/Initials of person performing manual integration.

16. Were data points removed from the initial calibration: ☐ Y ☐ N

17. What was the rationale for the calibration data elimination: _____

18. Is this an acceptable explanation: ☐ Y ☐ N

19. Was all data quantitated from the initial instrument calibration : ☐ Y ☐ N

20. If continuing instrument calibration verification criteria were used to quantitate the sample result, what was the reasoning behind this decision: _____

21. Was the client notified of the variance: ☐ Y ☐ N

Comments:_____

_____

_____

D: Calibration Verification Standards

1. Is the continuing instrument calibration verification used to confirm the continued validity of the initial calibration? ☐ Y ☐ N

2. Is continuing instrument calibration verification repeated at the beginning and end of each analytical batch? ☐ Y ☐ N

3. Are check standard table/charts up to date: ☐ Y ☐ N

4. Are limits calculated or default (C/D): ☐ C ☐ D

If, calculated, date of latest limits: ____/____/____

5. What is the acceptable recovery range: _____

6. What is the required frequency for calibration verification: _____

7. Was the frequency of calibration verification acceptable: ☐ Y ☐ N

8. What is the source of the check standards (reference #): _____

9. Were the checks associated with this batch acceptable: ☐ Y ☐ N

10. If no, was the data qualified and an explanation available for the client: ☐ Y ☐ N

Comments:_____

_____

_____

E: Blanks (Negative Controls)

1. Was a method blank analyzed at a frequency of one per preparatory batch of samples per matrix type per sample extraction or preparation method: ☐ Y ☐ N
2. Is the result of blank analysis used to assess batch acceptance: ☐ Y ☐ N
3. Were any positive detects found in the blank above the reporting limit: ☐ Y ☐ N
4. Was this analyte also found to be in the samples above the reporting limit: ☐ Y ☐ N
5. If there is contamination, is the source of contamination investigated and corrective action taken to correct, minimize or eliminate the problem: ☐ Y ☐ N
6. Did the blank concentration exceed a concentration > 1/10th the measured concentration of any sample in the batch: ☐ Y ☐ N
7. Did the blank concentration exceed the concentration present in the samples and is greater than 1/10th the specified regulatory limit: ☐ Y ☐ N
8. Were the samples associated with the contaminated blank reprocessed or reported with the appropriate qualifier for all samples with positive detects: ☐ Y ☐ N

F: Laboratory Control Sample

1. Was an LCS performed at a minimum frequency of 1 per 20 samples per matrix type, per sample extraction or preparation method: ☐ Y ☐ N
2. Is the LCS used for determining batch acceptance: ☐ Y ☐ N
3. Was the % recovery within control limits: ☐ Y ☐ N
4. Are limits calculated or default (C/D): ☐ C ☐ D

   If, calculated, date of latest limits: ____/____/____
5. List compound(s)/analyte(s) without up to date limits: Compound/Number of points available at this time:

   _____

6. Estimated completion date for new limits: ____/____/____

7. If one of the above criteria was not met, was the data appropriately qualified and approved by the immediate supervisor or management: □ Y □ N

G: Duplicates - _____

1. What is the required frequency for duplicates: _____
2. Was the required frequency met for this batch of samples: □ Y □ N
3. Was the duplicate within control limits: □ Y □ N
4. Was the RPD calculated correctly: □ Y □ N
5. Are duplicate table/charts up to date? □ Y □ N
6. Is data being evaluated with the established/default limits: □ Y □ N
7. Are limits calculated or default (C/D): □ C □ D

   If, calculated, date of latest limits: ____/____/____
8. Estimated completion date for new limits: ____/____/____
9. List compound(s)/analyte(s) without up to date limits: Compound/Number of points available at this time.

   _____

10. If one of the above criteria was not met, was the data appropriately qualified and approved by the immediate supervisor or management: □ Y □ N

Comments:_____

_____

_____

H. Spikes - _____

1. What is the required frequency for spiked samples: _____
2. Was the required frequency met for this batch of samples: □ Y □ N
3. Was the % recovery within control limits: □ Y □ N
4. Was the % recovery calculated correctly: □ Y □ N
5. Are spike table/charts up to date? □ Y □ N
6. Is data being evaluated with the established/default limits: □ Y □ N
7. Are limits calculated or default (C/D): □ C □ D

   If, calculated, date of latest limits: ____/____/____
8. Estimated completion date for new limits: ____/____/____
9. List compound(s)/analyte(s) without up to date limits: Compound/Number of points available at this time.

   _____

10. If one of the above criteria was not met, was the data appropriately qualified

and approved by the immediate supervisor or management. ☐ Y ☐ N

Comments:_____

_____

_____

I. Matrix Spike/Matrix Spike Duplicate - _____

1. What is the required frequency for spiked samples: _____
2. Was the required frequency met for this batch of samples: ☐ Y ☐ N
3. Was the % recovery within control limits: ☐ Y ☐ N
4. Was the % recovery calculated correctly: ☐ Y ☐ N
5. Was the RPD within control limits: ☐ Y ☐ N
6. Was the RPD calculated correctly: ☐ Y ☐ N
7. Are MS/MSD spike table/charts up to date: ☐ Y ☐ N
8. Is data being evaluated with the established/default limits: ☐ Y ☐ N
9. Is the sample selected as the matrix spike rotated among clients: ☐ Y ☐ N
10. Are all reported analytes being spiked: ☐ Y ☐ N
11. Date of Duplicate/RSD Limits: ____/____/____
12. Date of Spike Limits: ____/____/____
13. If one of the above criteria was not met, was the data appropriately qualified and approved by the immediate supervisor or management. ☐ Y ☐ N
14. List compound(s)/analyte(s) without up to date limits: Compound/Number of points available at this time.

_____

Comments:_____

_____

_____

J. Surrogate and/or Internal Standards

1. Are surrogate standards being used for this analysis: ☐ Y ☐ N
2. Is surrogate standard data recorded and monitored appropriately: ☐ Y ☐ N
3. Are there calculated limits for surrogate in liquid matrices: ☐ Y ☐ N ____/____/____
4. Are there calculated limits for surrogate in solid matrices: ☐ Y ☐ N ____/____/____
5. Are poor surrogate recoveries reported to the client: ☐ Y ☐ N
6. Are internal standards being used for this analysis: ☐ Y ☐ N
7. Is internal standard data recorded and monitored appropriately: ☐ Y ☐ N
8. What are the monitoring requirements for this analyses: _____
9. Are internal standard recovery limits monitored on a daily basis: ☐ Y ☐ N

Comments:_____

_____

_____

K. Instrumentation

1. Is there an instrument log book: ☐ Y ☐ N
2. Are there regular entries documented in the log book: ☐ Y ☐ N
3. Is corrective action taken when instruments are out of control: ☐ Y ☐ N
4. Are spare parts in inventory for routine maintenance: ☐ Y ☐ N
5. Used parts labeled properly: ☐ Y ☐ N

Comments:_____

_____

_____

L. Reagents and Standards

1. Check logbook for completeness: _____
2. Check reagent and standard bottles for:
   - Log Book Number: _____
   - Proper labeling: _____
   - Expiration date: _____
   - Lot Number: _____

Comments:_____

_____

_____

M. Miscellaneous

1. Is there a copy of NR 149 available: ☐ Y ☐ N
2. QA/QC Manual available: ☐ Y ☐ N
3. Records and work area organized: ☐ Y ☐ N
4. CPAR completed when necessary: ☐ Y ☐ N

Comments:_____

_____

_____

N. Additional questions and/or comments:

Category II. Systems audit - Data Evaluation

A. Paper Trail

Sample Receipt

1. Was the date and time of receipt noted: ☐ Y ☐ N
2. Was there a signature on the COC as being "received by laboratory": ☐ Y ☐ N
3. Was a temperature or "received on ice", statement noted on the COC: ☐ Y ☐ N
4. Was the sample receipt form completed contain sufficient information: ☐ Y ☐ N
5. Were all bottles received and in acceptable shape: ☐ Y ☐ N
6. If receipt of DRO or GRO soils, was the weight, extraction volume, date & time recorded and calculated correctly: ☐ Y ☐ N
7. Were the samples assigned the correct analytical # according to the COC and sample label: ☐ Y ☐ N
8. Did the data in LIMS correspond to the data on the COC: ☐ Y ☐ N
   - ☐ Analyses entered
   - ☐ Date sampled
   - ☐ Matrix
   - ☐ Hazards
   - ☐ Customer sample ID

9. Was the overall documentation of Sample Receipt: ☐ Good ☐ Fair ☐ Poor

Comments:_____

_____

_____

Sample Preparation

1. Bench/Data sheet: **NOTES**
   - ☐ Identification of the analysis/compound: _____
   - ☐ Analysts Name (no initials): _____
   - ☐ Date of Analysis: _____
   - ☐ Analytical number(s): _____
   - ☐ Method Used: _____
   - ☐ Reference to the Calibration: _____
   - ☐ Measured Response: _____
   - ☐ Calculation: _____
   - ☐ Calculated Result: _____

- ☐ Dilution: _____
- ☐ Final Value Reported: _____

2. Calibration standards/curve traceable to the log book: ☐ Y ☐ N
3. Calibration standards certified by manufacturer: ☐ Y ☐ N
4. Certificate located and verified: ☐ Y ☐ N
5. Check standard used to verify calibration traceable to the logbook: ☐ Y ☐ N
6. Certificate located and verified: ☐ Y ☐ N
7. Check standards certified by manufacturer: ☐ Y ☐ N
8. Is NIST traceability established through the calibration standards: ☐ Y ☐ N
9. Can NIST traceability be estabilised through alternative means: ☐ Y ☐ N
10. Was QC recorded on a summary table/chart: ☐ Y ☐ N
11. Data was flagged properly: ☐ Y ☐ N
12. Was data for analytes of concern at low levels reported at the MDL: ☐ Y ☐ N
13. Corrective/preventive action filed and approved, if required: ☐ Y ☐ N

Comments:_____

_____

_____

Final Report

1. Did the report flag(s) give sufficient information to the client: ☐ Y ☐ N
2. Were all data results reported correctly: ☐ Y ☐ N
3. Were there the correct # of significant digits reported: ☐ Y ☐ N
4. Did the cover page cover all necessary information: ☐ Y ☐ N
5. Were the dates and methods listed correct: ☐ Y ☐ N
6. Was everything flagged that required a flag: ☐ Y ☐ N
7. Overall organization of data and presentation: ☐ Good ☐ Fair ☐ Poor

Comments:_____

_____

_____

B. Specific Sample Investigation #_____

1. Holding time met? ☐ Y ☐ N
2. Total number of samples in the batch: _____
3. Sufficient number of the following for the analytical batch:
   - ☐ Check Standards How many: _____
   - ☐ Duplicates How many: _____
   - ☐ Matrix Spikes How many: _____

☐ MS/MSD How many: _____

☐ Blanks How many: _____

4. Surrogate Standard added to all samples: ☐ Y ☐ N

5. Number of Calibration Standards: _____

Curve: _____ Standard #: _____

6. Calibration meet the minimum requirements for acceptance: ☐ Y ☐ N

Corr. Coeff.:_____ %RPD:_____ % Variance:_____

7. Were results, prior to dilution correction, within calibration range: ☐ Y ☐ N

8. Reported value in LIMS correct? ☐ Y ☐ N

9. Did all QC meet acceptance criteria or was the proper corrective action taken: ☐ Y ☐ N

Comments:_____

_____

_____

_____

The above audit was performed by the QA/QC manager and reviewed by the following:

Analyst's Signature: _____ ***Date:*** ____/____/____

Supervisor's Signature: _____ ***Date:*** ____/____/____

Laboratory Manager: _____ ***Date:*** ____/____/____

QA/QC Manager: _____ ***Date:*** ____/____/____

*Comments:*_____

_____

_____

_____

Any noted deficiencies shall be addressed immediately by all parties involved. A CAR form shall be filed immediately explaining all required modifications and a specific course of action.

SECTION 11.0: QUALITY ASSURANCE REPORT

USFilter, Enviroscan Services analysts shall submit to the QA Officer a report that updates the status of the laboratory's quality assurance program. Reports shall be completed and updates made by each analyst to the quality system at least once per year.

11.1 Report Content

Reports shall give a complete overview of the quality control status in each area of the laboratory. See example Form 11.1. Reports may include, but are not limited to, the status of:

- Applicable Instruments and Methodology
- Demonstration of Capability (DOC)
- Limit of Detection (LOD)
- Control Limits
- Update Standard Operating Procedures
- EPA Methodology Review
- Updating Quality Control data, including:
    - Calibrations
    - Check Standards
    - Matrix Spike
    - Matrix Spike Duplicates
    - Duplicates
    - Method Blanks
    - Internal Standards
    - Surrogates

11.1.1 Analyst Section

The report is divided into sections according to quality control parameters. It is the responsibility of the analyst to complete the report and have it approved by the QA Officer by the date indicated on the QA/QC report.

11.1.2 Quality Assurance Officer Section

The QA Officer shall complete the section, which addresses the Standard/Reagent Log and Instrument Maintenance Log. The QA Officer shall keep the Laboratory Officer updated on the status of the QA/QC reports.

Form 11.1 QA/QC Requirement Report

Area: _____ Date (Month/Day/Year): _____/_____/_____
Analyst: _____ Technicians: _____
_____

Following are the instruments and method(s) of analysis the above mentioned analyst(s) are responsible for:

Instrument I.D.	Method I.D.	Matrix Type

Demonstration of Capability (DOC): DOCs are a requirement for all analytical procedures. DOCs must be performed prior to using any test method, on a yearly basis, or any time there is a significant change in instrument type, personnel, or test method. Significant changes can be, but are not limited to, any change in matrix, instrumentation or test method change (e.g., a change in the detector, column, or method revision).

Instrument(s)	Method	Matrix	Validated (Y/N)			Date Performed
			XXX	XXX	XXX	

METHOD DETECTION LIMIT (MDL): The MDL study is completed whenever an instrument or method is initially established, for new analysts, when major modifications are made to the process or on a yearly basis. The MDL is completed and updated yearly for each matrix within the guidelines of 40 CFR part 136 Appendix B. Do you have a valid MDL study for all of the analyses listed in the QC Report?

Instrument(s)	Method	Date of Water MDL	Date of Soil MDL

CONTROL LIMITS: Control limits are established for spikes, duplicates, MS/MSDs, and surrogates using 15-20 data points and statistically obtaining limits. Control limits are established for each matrix, (if possible). In general those matrices are water/groundwater, wastewater, drinking water, methanol, soil/solid/sludge, and TCLP. Control limits are established each year OR when sufficient data is available. Do you have control limits for each matrix/compound/analysis for all of the analyses listed in the QC Report?

Method	Water/Liquid Groundwater	Wastewater	Drinking Water	Soil/Solid Sludge	TCLP

STANDARD OPERATING PROCEDURE(S): Please read the following SOP(s), and make a copy with the necessary revisions to update the procedure:

SOP #	Revision Date:	(X) Needs Revision/Updating

EPA METHODOLOGY: Please review the following methods to ensure that you are currently meeting all the requirements necessary to perform each analysis under the most recent version of the method:

QUALITY CONTROL:
Daily quality control includes calibrations, check standards, MS/MSDs, duplicates, method blanks, lab control samples, internal standards, and surrogates.

QC CHECKLIST FOR THE ANALYST:
Are you completing the daily quality control requirements for each matrix as required by the EPA or other approved source? YES NO

If no, what procedure(s) are you currently not performing, and what is the rationale behind the deviation?

_____

_____

Are you performing corrective action for all data, which does not meet quality control? YES NO

If no, why not and what is your current convention for dealing with unacceptable or out of control situations?

_____

_____

Are you filing Corrective/Preventive Action Reports when applicable? YES NO

Additional Comments:

_____

_____

_____

Below to be completed by Quality Assurance Manager:

Standard/Reagent Log Book up to date? YES NO
Instrument maintenance log book complete? YES NO
Quality Control Issues? YES NO
Last complete audit performed on ____/____/____.

Analyst Signature: _____ **Date:** ____/____/____
QA Manager Signature: _____ **Date:** ____/____/____

Please return to the Quality Assurance Manager's Office by ____/____/____.

SECTION 12.0: FINAL REPORTS

USFilter, Enviroscan Services shall review data and reports for completeness and accuracy before sending them to the customer. The reports shall consist of a cover letter, analytical results, sample receipt documents, and QA packages, if requested. See FIGURE 12.1 for an example of the standard completed hard copy report.

12.1 Cover letter

USFilter, Enviroscan Services reports shall begin with a cover letter, which includes:

- Report Date
- Report Identification #
- Customer identification:Client name, address, and contact person(s).
- Project/Purchase Order number
- Sample receipt date
- Approved method reference
- Reference to the chain of custody or sample receipt record which is attached.
- Closing comments along with the authors signature and a signature of approval.
- A sample summary page which puts the Lab ID#, Client Sample ID, Date Sampled and matrix type into a tabular format.
- A sample narrative/sample status section is also provided on the summary page that allows the laboratory to notify the client of potential problems or anomalies.
- The final report is also given an approval signature by the laboratory director, technical director or quality assurance officer.
- Confidentiality Statement
- Total Number of pages included in document.

12.2 Analytical Results

Analytical result pages follow the cover letter and may have one of two formats. 1) A number of parameters are included for one analytical number, or 2) A number of samples with the same parameter on one page. Each page shall include the following information:

- Customer Identification: Client name, address, and contact person(s)
- Project number, if available.
- Sampled by information
- Sample receipt date and analysis date
- Report date
- Report writer's initials
- Client's sample identification, matrix type, sample date and the laboratories analytical number.
- Calculated value of each analyte, units, level of detection, level of quantitaion, dilution and approved method
- Qualifier Descriptions, see Appendix D
- Analyst initials

12.3 Sample Receipt Documentation

Sample receipt documents are included in the last section of the report. These documents shall include:

- Chain of custody(s) and/or Analytical request form (s)
- Sample receipt form, if samples received deviated from EPA or WDNR protocol
- Wisconsin DNR form (s), if applicable

12.4 Quality Assurance Packages (QAP)

USFilter, Enviroscan Services shall include quality control data upon request from the client. The client shall notify the laboratory upon bid request for the individual project. The QA package request is handled on a case by case basis depending on the analytical data level requested. See Figure 12.2 for QAP levels.

12.5 Final Reports

A final report is generated by an analyst using data from a preliminary analytical report.

12.5.1 Preliminary Analytical Report

A preliminary report is generated after all analytical results are entered into LIMS from each set of samples. The preliminary report contains the following information:

- Analytical Number
- Analysis Requested
- Analytical Result
- Client Identification
- Sample Date
- Sample Type
- A summary page that includes comments, etc.

The Laboratory Officer or designee distributes the preliminary reports to the report writer for final report preparation. Report distribution is assigned according to the analytes requested. Final reports are completed on the same day or the day following distribution.

12.5.2 Analytical Data Transfer and Review

Final reports are completed by direct transfer of data from the LIMS system to the word processing program (Wordperfect or Word). The file is identified using the analytical numbers and the report format. All analysts completing their section of the report use the same file number to access the data. The report is printed on a laser printer and returned to the report writer. The writer reviews the report, signs the cover letter, and initials all data pages in the "Prepared By" section. The report shall be verified in LIMS after the report is completed and the samples are put into a holding area for a minimum of 30 days prior to disposal. The report file is submitted to the Lab Officer or designee for approval.

12.5.3 Report - Review and Approval

The final report with a copy of the COC, Analytical Request Form, and Sample Receipt Report, if applicable, are attached. The final report with attachments shall be reviewed by the Laboratory Director or other approved designee for final release. The following items are reviewed:

- Cover letter for clarity and completeness
- Analytical results for proper units and appropriate significant figures

- Qualifiers and descriptions
- Approved methods referenced
- Check results for reasonableness.

The report is revised, if necessary, or approved by the Laboratory Officer or designee and signed on the cover page of the report. The report and attachments are mailed and faxed, if requested, to the client and a copy put in the file folder for invoicing and storage.

12.5.4 Electronic Data Deliverables

Data can be submitted directly to the responsible party via diskette or electronic mail if requested. The "disk" request must be made visible on the chain of custody. Once the initial request is made the sample(s) are marked as needing electronic deliverables and are followed through the lab by the project officer. When the final report is complete and verified the electronic data is generated from the laboratories LIMS system.

12.5.5 Report Storage

USFilter, Enviroscan Services reports shall be filed in the Customer Service area by client name and analytical number. All files and sample information are kept for a minimum of three years. At all times the previous years analytical reports are kept in the customer services office. The year prior to that is stored on location in the warehouse.

12.5.6 Preliminary Final Reports

Analytical reports may be faxed as preliminary reports to the client, and shall be stamped "PRELIMINARY" to designate that they have not been approved by the Laboratory Officer or designee.

Figure 12.1: USFilter, Enviroscan Services – Sample Report

Sample Summary 098172.

Lab Id	Client Sample ID	Date/Time	Matrix
098172	CLOSURE	04/15/02 16:00	SOIL
098173	CLOSURE EXT	04/18/02	TCLP EXTRACT

Sample Narrative/Sample Status

LOGIN:

GENERAL:

ANALYSES:

QA/QC:

REPORTING:

Qualifier Descriptions:

SPL	Matrix spike recovery within analytical batch was low. Sample matrix appears similar to your sample; result may be biased low.

Definitions

LOD = Limit of Detection
LOQ = Limit of Quantitation
< = Less Than
COMP = Complete
SUBCON = Subcontracted analysis
mv = millivolts
pCi/l = picocurie per liter
µg/l = Micrograms per liter = parts per billion (ppb)
µg/kg = Micrograms per kilogram = parts per billion (ppb)
mg/l = Milligrams per liter = parts per million (ppm)
mg/kg = Milligrams per kilogram = parts per million (ppm)
NOT PRES = Not Present
ppth = Parts per thousand
(S) = Surrogate Compound

Apex Analytical Services
P.O. Box 001
1111 W. Summit Street
Zenith Tower, WI 55555

Attn: Tommy Highpoint

PROJECT NO.: PINNACLE-1
REPORT NO. : 098172.
DATE REC'D : 04/17/02
REPORT DATE: 05/01/02
PREPARED BY: TOP

Sample ID: CLOSURE **Matrix: SOIL** **Sample Date/Time: 04/15/02 16:00** **Lab No. 098172**

	Result	Units	LOD	LOQ	Dilution Factor	Qualifiers	Date Analyzed	Analyst
EPA 8082								
PCB-1016	<1.3	µg/kg	1.3	4.33	1		04/29/02	CKV
PCB-1221	<2.6	µg/kg	2.6	8.66	1		04/29/02	CKV
PCB-1232	<4.5	µg/kg	4.5	15.0	1		04/29/02	CKV
PCB-1242	<1.0	µg/kg	1.0	3.33	1		04/29/02	CKV
PCB-1248	<3.1	µg/kg	3.1	10.3	1		04/29/02	CKV
PCB-1254	<0.9	µg/kg	0.9	3.0	1		04/29/02	CKV
PCB-1260	<1.4	µg/kg	1.4	4.66	1		04/29/02	CKV
Tetrachloro-m-xylene (S)	107.	%	-	-	-		04/29/02	CKV
Decachlorobiphenyl (S)	95.	%	-	-	-		04/29/02	CKV
Method 3550 Ultrasonic Ext.	COMP		-	-	-		04/29/02	CKV
EPA 1010								
Flash Point	>140.	°F	-	-	1		04/30/02	DJB
EPA 1311								
TCLP Extraction	COMP		-	-	-		04/18/02	JJP
Zero Headspace Extraction	COMP		-	-	-		04/18/02	JJP
TCLP Phase Determination	COMP		-	-	-		04/17/02	JJP
EPA 300.0								
Diss. Chloride	127.	mg/kg	10.0	33.3	1		04/24/02	LTD
EPA 9045								
pH - Laboratory	9.50		-	-	-		04/18/02	JJP
EPA 9095								
Free Liquids	0.000	%	-	-	1		04/18/02	JJP
ES-180								
Sp. Gravity	2.09		-	-	-		04/18/02	JJP
MOSA21-2								
Total Solids	84.2	%	-	0.33	-		04/18/02	LMV
SW846 MET								
Reactive Cyanide	<0.0154	mg/kg	-	0.013	1		04/19/02	LCK
Reactive Sulfide	<28.4	mg/kg	-	50.0	1		04/19/02	JJP

All results calculated on a dry weight basis.

Apex Analytical Services
P.O. Box 001
1111 W. Summit Street
Zenith Tower, WI 55555

Attn: Tommy Highpoint

PROJECT NO.: PINNACLE-1
REPORT NO. : 098172.
DATE REC'D : 04/17/02
REPORT DATE: 05/01/02
PREPARED BY: TOP

Sample ID: CLOSURE EXT **Matrix: TCLP-EXT** **Sample Date/Time: 04/18/02** **Lab No. 098173**

	Result	Units	LOD	LOQ	Dilution Factor	Qualifiers	Date Analyzed	Analyst
EPA 245.1								
Mercury	<0.002	mg/l	0.0002	0.00067	10		04/22/02	JCH
EPA 420.2								
Phenols,colorimetric	<100.	mg/l	-	0.005	1	SPL	04/22/02	LCK
EPA 6010								
Arsenic	<0.016	mg/l	0.008	0.0266	2		04/26/02	BMS
Barium	0.85	mg/l	0.002	0.00666	2		04/26/02	BMS
Cadmium	<0.0022	mg/l	0.0011	0.00366	2		04/26/02	BMS
Chromium	<0.0032	mg/l	0.0016	0.00533	2		04/26/02	BMS
Copper	<0.008	mg/l	0.004	0.0133	2		04/26/02	BMS
Lead	<0.02	mg/l	0.01	0.0333	2		04/26/02	BMS
Nickel	0.028	mg/l	0.003	0.00999	2		04/26/02	BMS
Selenium	<0.036	mg/l	0.018	0.0599	2		04/26/02	BMS
Silver	<0.006	mg/l	0.003	0.00999	2		04/26/02	BMS
Zinc	0.329	mg/l	0.005	0.0167	2		04/26/02	BMS
EPA 8021								
Benzene	<6.20	µg/l	0.31	1.03	20		04/19/02	LMP
Carbon Tetrachloride	<11.8	µg/l	0.59	1.96	20		04/19/02	LMP
Chlorobenzene	<6.20	µg/l	0.31	1.03	20		04/19/02	LMP
Chloroform	<5.40	µg/l	0.27	0.899	20		04/19/02	LMP
1,4-Dichlorobenzene	<6.00	µg/l	0.3	0.999	20		04/19/02	LMP
1,2-Dichloroethane	<3.40	µg/l	0.17	0.566	20		04/19/02	LMP
1,1-Dichloroeth(yl)ene	<7.80	µg/l	0.39	1.3	20		04/19/02	LMP
Methyl Ethyl Ketone(MEK)	<40.0	µg/l	2.0	6.66	20		04/19/02	LMP
Tetrachloroeth(yl)ene	<6.40	µg/l	0.32	1.07	20		04/19/02	LMP
Trichloroeth(yl)ene	<7.20	µg/l	0.36	1.2	20		04/19/02	LMP
Vinyl Chloride	<4.00	µg/l	0.2	0.666	20		04/19/02	LMP
VOC Vial pH above 2	7.00		-	-	1		04/19/02	VOL
EPA 8081								
g-BHC (Lindane)	<0.013	µg/l	0.0013	0.00433	10		04/24/02	LTD
Chlordane	<0.15	µg/l	0.015	0.05	10		04/24/02	LTD
Endrin	<0.033	µg/l	0.0033	0.011	10		04/24/02	LTD
Heptachlor	<0.009	µg/l	0.0009	0.003	10		04/24/02	LTD
Heptachlor Epoxide	<0.014	µg/l	0.0014	0.00466	10		04/24/02	LTD
Methoxychlor	<0.119	µg/l	0.0119	0.0396	10		04/24/02	LTD
Toxaphene	<0.74	µg/l	0.074	0.246	10		04/24/02	LTD
Tetrachloro-m-xylene (S)	90.2	%	-	-	10		04/24/02	LTD
Decachlorobiphenyl (S)	113.	%	-	-	10		04/24/02	LTD
Method 3510 Liquid Ext.	COMP		-	-	-		04/19/02	CKV
EPA 8270								
o-Cresol	<43.0	µg/l	43.0	143.	1		04/29/02	MRD
m-&p-Cresol	<51.0	µg/l	51.0	170.	1		04/29/02	MRD
2,4-Dinitrotoluene	<11.0	µg/l	11.0	36.6	1		04/29/02	MRD
Hexachlorobenzene	<5.90	µg/l	5.9	19.6	1		04/29/02	MRD
Hexachlorobutadiene	<14.5	µg/l	14.5	48.3	1		04/29/02	MRD
Hexachloroethane	<12.0	µg/l	12.0	40.0	1		04/29/02	MRD
Nitrobenzene	<13.5	µg/l	13.5	45.0	1		04/29/02	MRD
Pentachlorophenol	<38.0	µg/l	38.0	127.	1		04/29/02	MRD
2,4,5-Trichlorophenol	<50.0	µg/l	50.0	167.	1		04/29/02	MRD
2,4,6-Trichlorophenol	<37.0	µg/l	37.0	123.	1		04/29/02	MRD
Pyridine	<11.0	µg/l	11.0	36.6	1		04/29/02	MRD
Method 3510 Liquid Ext.	COMP		-	-	-		04/25/02	CKV

Figure 12.2: USFilter, Enviroscan Services QAP Levels

QC Data Deliverables

Level 1

- Analytical summary w/ cover page and sample narrative.
- Analytical results.
- Chain of Custody (COC).

Level 2

- Level 1 +
- Surrogate recoveries* (when used).
- Continuing calibration verification.
- Method blank summary.
- LCS summary.
- MS/MSD summary.
- Replicate/duplicate summary.

Level 3

- Level 2 +
- GC/MS tune reports.
- Initial Instrument calibration.

Level 4

- Level 3 +
- Raw data:
- Instrument logs (bench sheets)
- Extraction logs
- Digestion logs
- Data sheets/Calculation Sheets
- Chromatograms (check, blank, LCS, MS/MSD, samples)
- Spectra
- Instrument sequences

SECTION 13.0: DOCUMENTATION AND STORAGE

USFilter, Enviroscan Services has an established program for documentation and storage of all analytical data and daily operations of the laboratory.

13.1 Documentation

Documentation of all the daily laboratory operations are completed through bench sheets, bound log books, computation books and computer software

13.1.1 Bench Sheets

Analytical data shall be recorded on established USFilter, Enviroscan Services bench sheet forms for the majority of the analyses performed in the laboratory. Infrequent analyses and sample preparation shall be documented in bound log books or computation books. Bench sheets are printed on standard 8½ x 11 paper using Microsoft® Excel. Bench sheets may be revised or updated with the approval of the QA Officer. Bench sheets shall include, but are not limited to, the following:

- USFilter, Enviroscan Services identification
- Analyst's name
- Name and Date of Analysis
- Analytical numbers and Batch ID
- Method reference
- Instrument information (calibration or last time calibrated)
- Raw data (i.e. peak height, absorbance, dilutions, etc.)
- Analytical results in proper units
- Quality Control data, i.e., CCV, LCS, MS/MSD, DUP, Surrogate recovery, etc..

Optional Elements:

- Project identification
- Comments area

13.1.1.2 Computer Sequence Sheets

The software program of ACCESS*CHROM automatically generates the analytical data sequence and required elements in report form. The following areas of the laboratory use this method for reporting data:

- VOCs by 8021
- Pesticides, Herbicieds, PCBs
- GRO/DRO by WI DNR

All elements listed in 13.1.1.1 above shall be included in each report or additional bench sheets shall be provided to manually add the necessary information.

13.1.1.3 Analyst's Responsibility

The following rules apply to the use of bench sheets

- A sufficient number of bench sheets shall be on hand for each analyte tested.
- A new bench sheet is required on each analysis day.
- Bench sheets shall be complete, legible, and written in ink, see

Section 10.1.1.5

- Errors shall be crossed out once and initialed by the analyst with corrections or comments near the site.
- Bench sheets are to be stored properly.

13.1.2 Bound Log Books and Computation Books

Log books and computation books shall be used in the laboratory to record reagent and standard preparation, instrument maintenance, and miscellaneous analyses requested infrequently. These books may be used by an entire department or an individual of the lab.

Note: A spiral notebook may be used for extractions in the organic laboratory.

13.1.2.1 Book Description

Log books are 6"x 9½" bound hard-covered books ; Computation books are 9"x 12" bound soft-covered books with numbered pages. Each book is assigned a specific number and used in a specific area of the laboratory. Books may be obtained from the lab secretary and/or QA Officer, upon request.

13.1.2.2 Analyst's Responsibility

Log books and computation books adhere to the same basic rules as bench sheets and require similar information to be recorded. The following additional rules apply to the use of log or computation books:

- Book description
- Date of log entry
- Refer to Section 6.2.2 for Maintenance Log information
- Refer to Section 10.1.1.3 for Standard/Reagent Log information.
- Both front and back pages shall be used, or an "X" shall be placed over the unused page.
- Store books properly, upon completion, and retained for a minimum of five years.

13.1.3 Computer System

USFilter, Enviroscan Services has a VAX network which is the center of documentation for the laboratory. The system organizes all the information from sample receipt through the analytical process to the final report. The VAX computer system is secure and a password shall be used to gain access into the system.

13.1.3.1 Laboratory Informational Management System (LIMS)

The LIMS system is a menu driven program connected to the VAX system with computer terminals throughout the laboratory areas. All USFilter, Enviroscan Services personnel have access to this program.

ATOAN is a calibration program which permits the analyst to enter calibration data and sample instrument response. The program calculates the best fit curve and the sample result before transferring the data into LIMS.

ACCESS*CHROM is a chromatography program designed to collect analytical data from all gas and HPLC chromatographs. Operational abilities include calibration curve determination and data

evaluation from the raw data. The program automatically transfers data results into the LIMS system. Data is printed out on laser printers and stored appropriately. Updates and maintenance for the computer system is completed by the Management Information Systems (MIS) department.

The primary software used by LIMS is Microsoft®Office, Word Perfect, and 20/20. LIMS handles all sample information, see Section 3.2.6. Microsoft® Office and WordPerfect are used to generate reports; 20/20 is used to evaluate data.

Data generated from the A^2S, ICP, GC/MS, and TRAACS instruments are not on the VAX system; therefore, data is transferred manually into the LIMS system. Each computer for the instrumentation above has the capability to determine the calibration curve, and evaluate data.

13.1.3.2 Data Storage
The VAX computer system creates a back up file for each days activities and stores data for approximately one year for ease of retrieval. Data is stored on computer tapes and microfiche for long term storage. See Form 13.1: Information Services: Programming Request.

13.2 Document Storage

USFilter, Enviroscan Services has established an active and formal storage program for all analytical data and related documents. Documents consist of three media; paper, microfiche, and computer tapes.

13.2.1 Active Storage
Active storage program refers to documents recently generated and readily accessible to complete data checks and generate reports.

13.2.2 Formal Storage
Formal storage program refers to the storing of documents in properly identified boxes (E-Year-Sequential number) measuring 15x12x10 and stored at a facility storage site. An itemized list shall be generated for each box; this list shall include: Box number; Total number of boxes sent to storage; Dates sent to storage; Tentative destroy date; Destruction confirmation with approval from Lab Officer; Box contents. A copy of the list shall be kept in the following areas:

- Office of personnel requesting boxes
- Inside the storage box
- Storage file binder located in the Lab Secretary's office
- Office of personnel requesting transfer to storage area

Formal storage boxes shall be accessible within a reasonable amount of time. This data shall be stored for a minimum of seven years.

13.2.3 Paper Storage
Paper documents are stored for a minimum of six years from the date of sample analysis. The data is in active storage for the first year, and the last five in formal storage. Documents, which are stored, include all areas previously discussed in Sections 13.1.1 and 13.1.2. All data is stored according to laboratory activity and date of analysis.

Paper documents are transferred from active storage to formal storage at the beginning of each calendar year, at a minimum. Areas, such as the analysis of compounds by gas chromatographs, generate a sizable amount of data and shall be stored more frequently.

13.2.3.1 Personnel Responsible:
Analysts shall organize and put data into storage boxes and inform the Lab Secretary of the content of each box. The Lab Secretary shall assign the number of each box. The box number is printed on the sides of each box . A copy of the list is put into the box, sealed, and ready for formal storage. Boxes shall be retrieved by submitting a request to the Lab Secretary. Data shall be reviewed and returned to the boxes for storage in a timely manner.

QC data accumulated for the year shall be bound, if appropriate, and submitted to the QA Officer for storage. The QA Officer shall retain three years of QC data in the Quality Assurance area, if practical.

13.2.4 Computer Data File Storage
Data shall be stored from the VAX system and include all files from ACCESS*CHROM, Client reports, and the same data as Section 13.2.4. Data is stored onto the hard drive of the VAX as it is entered into the data base. Each month this data is transferred onto tape. Tapes are stored for a minimum of three years but are typically kept indefinitely because of the small space it occupies. Storage of data onto the hard drive and magnetic tape is the responsibility of the Management Information System (MIS) department.

Retrieval of data off the hard disk is readily accessible through LIMS. Data contained on tapes may be obtained by requesting specific data from the MIS department.

13. 3 List of Active Storage Sites

Following is a list of locations where USFilter, Enviroscan Services currently store raw data prior to archiving.

Document Type	Location	Container Type
Inorganic Bench Sheets	Inorganic Area	File Cabinet
Log Books; Computation Books	Analyst Work Area	Not Specified
ATOAN Data Reports	Inorganic Area	File Cabinet
Sample Receipt Records	Log-in Area	Binders
QC Tables & Charts	Analyst Work Area	Binders
ACCESS*CHROM data	Analyst Office	File Cabinet
GC/MS Analysis Data	Analyst Office	File Cabinet
Extraction Data	Extraction Lab	File Cabinet
Instrument Log Books	Instrument Area	Not Specified
Standard/Reagent Log Books	Lab Area	Unspecified
Client Reports	Customer Service Office	File Cabinet (Active 2 years)

Appendix A

USFILTER, ENVIROSCAN SERVICES

PERSONNEL/POSITION

James R. Salkowski
Laboratory Director

RESPONSIBILITIES

Responsible for the overall management of USFilter, Enviroscan Services, Rothschild, WI.

EDUCATION

University of Wisconsin - B. S. Environmental Science, 1973
University of Wisconsin - M.E.A.S., Chemical Limnology, 1977
Additional Training
- Technicon AutoAnalyzer, 1977
- Supervision, 1980
- Emmission Spectroscopy (ICP), 1984
- Ion Chromatograph, 1985
- Management, 1980
- Leadership, 1996

EXPERIENCE SUMMARY

Twenty-three years experience in analytical chemistry and environmental assessments associated with lake studies, drinking water, groundwater, and hazardous wastes. Proficient in operation of AutoAnalyzer, AA, ICP, IC, and TOC instrumentation

PROFESSIONAL AFFILIATIONS

Wisconsin Environmental Labs Association
Analytical Laboratory Officers Association
President, 1989-91; Director, 1995 - 2000

USFILTER, ENVIROSCAN SERVICES

PERSONNEL/POSITION

Cindy K. Varga
Quality Assurance/Quality Control Officer

RESPONSIBILITIES

Development of Quality Assurance/Quality Control procedures. Surveillance of QA/QC programs which have been implemented. Direct and maintain laboratory certification programs and participation in performance evaluation studies. Work closely with clients and the environmental laboratory to maintain customer satisfaction.

EDUCATION

University of Wisconsin – Eau Claire; B.S. Chemistry-Business

EXPERIENCE SUMMARY

Over twelve years experience in the field of analytical chemistry. Experience in Gas Chromatography for analysis of semi-volatile organic compounds (SVOCs), HPLC for polynuclear aromatic Hydrocarbons (PAH) and Gas Cromatography for Diesel Range Organics (DRO) analyses.

USFILTER, ENVIROSCAN SERVICES

PERSONNEL/POSITION

Greg P. Flak
Sales Officer

RESPONSIBILITIES

Responsible for the generation of new business as well as supervision of current accounts. Performs marketing and forecasting functions. Serves as company representative to clients, potential clients, trade shows, and professional organizations.

EDUCATION

University of Wisconsin - Stevens Point; B.S. in Chemistry (ACS Certified)

EXPERIENCE SUMMARY

Over six years experience in the field of analytical chemistry. Experience in Atomic Absorption Spectrophotometry in metals analysis and Gas Chromatography for organic analyses. Functioned as the laboratory health and safety coordinator.

PROFESSIONAL AFFILIATIONS

Federation of Environmental Technologists
Wisconsin Groundwater Association
Wisconsin Wastewater Operator's Association
Marathon County Solid Waste Committee

USFILTER, ENVIROSCAN SERVICES

PERSONNEL/POSITION

Bruce Schertz
Technical Director

RESPONSIBILITIES

Responsible for overseeing the operations and supervision of personnel for the inorganic laboratory which includes wet chemistry, TRAACs, Ion Chromatography, and Carbon Analyzer. Responsibile for overseeing the operations and supervision of personnel for the organic laboratory. Responsible for Inductively Couples Plasma (ICP) spectrophotometry for metal analyses.

EDUCATION

University of Wisconsin - Stevens Point; B. S. Chemistry

EXPERIENCE SUMMARY

Over tenyears experience in the field of analytical chemistry. Proficient in the operation of Atomic Absorption spectrophotometry and Inductively Couples Plasma (ICP) spectrophotometry.

USFILTER, ENVIROSCAN SERVICES

PERSONNEL/POSITION

Sharon K. Maltbey
Customer Service Representative

RESPONSIBILITIES

Work as a liaison between our clients and the laboratory employees to ensure customer satisfaction. Responsible for supplying customers with analytical quotations, specific sampling procedures and practices, and sampling equipment needs when necessary. Provide preliminary sample results and sample status updates.

EDUCATION

University of Wisconsin - Eau Claire

EXPERIENCE SUMMARY

Twelve years experience in the laboratory as an Inorganic Lab Technician, MIS operator, Organic Laboratory Technician, and Sample Receipt Technician. The vast amount of experience is an essential tool when handling the questions and needs of clients.

Appendix B

SAMPLE CONTAINERS, PRESERVATION, and PROCEDURES

Soil Analyses

- **GRO/PVOC/VOC (preserved)**
  (1) - pre-weighed **60 ml amber glass jar**. Add 25 grams of soil. Add the 25 ml of pre-measured methanol from the 40 ml vial, provided. Keep at 4°C.

  CAUTION: Methanol is hazardous material.

  Note: GRO soil samples require a methanol trip blank. Transfer methanol to an empty 60 ml amber jar and label as TRIP BLANK.

- **VOC (non-preserved)**
  (1) - **4 oz. Clear glass** wide-mouthed soil jar - no headspace. Keep at 4°C.

- **DRO (non-preserved)**
  (1) - Preweighed 60 ml Amber jar. Add 25 grams of soil. Lab shall add methylene chloride. Keep at 4°C.

- **PNA, PCB, Herbicides, Pesticides, Semivolatiles (non-preserved)**
  (1) - 4 oz. Clear glass wide-mouthed soil jar. Keep at 4°C. Need 35 grams of sample per analyses. Any combination of three or more of the above listed analyses or a dry sample may require more than one jar. A 9 oz. jar may be provided for multiple analyses.

- **TCLP**
  (1) - 4 oz. Clear glass wide-mouthed soil jar with a minimum of 100 grams - no headspace.

- ***Total Solids, All Metal and Inorganic Analyses**
  (1) - **4 oz. plastic** or (1) - **4 oz. glass** wide-mouthed soil jar. Keep at 4°C.

  *** Total solids jar is required with each soil sample.**

SAMPLE CONTAINERS, PRESERVATION, and PROCEDURES

Water Analyses (*Organics*)

- **GRO/PVOC** (2) - 40 ml VOC vials with 0.5 ml 1:1 HCl. Keep at 4°C.
- **VOC** (3) - 40 ml VOC vials with 0.5 ml 1:1 HCl. Keep at 4°C.
- **GRO/VOC** (4) - 40 ml VOC vials with 0.5 ml 1:1 HCl. Keep at 4°C.

TRIP BLANKS ARE REQUIRED FOR EACH DAY OF SAMPLING!

Note: If sampling from a faucet, remove screen and allow the water to run for a several minutes or until cold before sampling. Slowly fill the vial to the top until you form a small crown with no headspace. Place cap squarely on vial and tighten. After the cap is secure, invert the vial to determine if any air bubbles remain inside. If bubbles are present, open the vial, slowly add more liquid, and recap. Label each vial immediately after sampling to clearly identify the sample using the peel-off labels provided.

- **DRO** (1) - Liter Amber bottle with 5.0 ml 1:1 HCl. Keep at 4°C.
- **PNA** (1) - Liter Amber bottle. Keep at 4°C. Per Analysis.
- **Pesticides** (1) - Liter Amber bottle. Keep at 4°C. Per Analysis.
- **Herbicides** (1) - Liter Amber bottle. Keep at 4°C. Per Analysis.
- **PCBs** (1) - Liter Amber bottle. Keep at 4°C. Per Analysis.
- **Semi-volatiles** (1) - Liter Amber bottle. Keep at 4°C. Per Analysis.
- **Oil & Grease** (1) - Liter Amber Bottle with 5.0 ml H_2SO_4. Keep at 4°C.
- **Total Phenolics** (1) - 250 ml Amber Bottle with 1 ml H_2SO_4. Keep at 4°C.

To avoid breakage, Fill all Amber bottles to the small of the neck!

Water Analyses (*Inorganics*)

- **Metals/Hardness** (1) - Plastic container with HNO_3 preservation. Keep at 4°C.
- **NH3-N, NPOC COD, MCOD, TKN,P, NO3+NO2-N** (1) - Plastic container with H_2SO_4 preservation. Keep at 4°C.
- **CN, Sulfide-S** (1) - Plastic container with NaOH preservation. Keep at 4°C.
- **All Other Inorganics** (1) - Plastic container non-preservered. Keep at 4°C

Plastic bottles may vary in size depending on the number of analysis requested.

Special Notes:

DO NOT RINSE OUT ANY OF THE VIALS or BOTTLES!

EXTRA QA/QC Bottles marked with pink labels "please fill this bottle for QC analysis" are included in each kit. These bottles are to be filled by the customer however, they are for lab use only. Testing performed on this sample will not be charged to you. If bottles are not returned to USFilter, the data may be subject to qualification

SHIPPING INSTRUCTIONS

DID YOU REMEMBER TO:

- ✓ Fill QC bottles. (Samples received without ample QC bottles may be subject to data qualification.)
- ✓ Pack all glass containers back in the bubble wrap pouches for protection during shipping.
- ✓ Pack wrapped glass bottles and plastic containers upright in the coolers secure without forcing the bottles in the cooler. If the containers can move around in cooler fill with bags of ice or packing material to prevent breakage.
- ✓ Place bags of ice on top of containers.
- ✓ Fill out Chain of Custody:
    1) **Company Name, Address, Project, P.O. and Quote Number.**
    2) **Bill To Information**
    3) **Check sample type and Turnaround time (**Rushes Must Be Pre-approved**)**
    4) **Fill out sample information: Date, Time, No. of Containers and Sample ID.**
    5) **Sign Chain**
- ✓ Seal chain of custody and any other paperwork in a plastic bag and tape to the inside of cooler lid.
- ✓ Tape cooler shut around the lid to prevent leaking.
- ✓ Place pre-printed label on the top of cooler and seal by taping completely around entire cooler.

- **Just a reminder if you are shipping from out of state, Fed-Ex will not ship if the cooler leaks.**

Thank you for using USFilter, Enviroscan Services. Please call Customer Service at 1-800-338-7226 if you have any questions.

Ship to: **USFILTER, ENVIROSCAN SERVICES**
301 W MILITARY ROAD
ROTHSCHILD WI 54474

SAMPLING INSTRUCTIONS

SAMPLING GUIDELINES FOR

Tedlar® Sample Bags

Sampling Instructions*:

1. Connect Teflon® tubing from the exhaust port of an air-sampling pump to the hose connection stem (protruding from the top of the hose/valve fitting.)

2. To unlock the valve, loosen the knurled thumbscrew on the side of the hose/valve fitting by turning counterclockwise.

3. Push the stem of the valve down to open the fitting for sampling.

4. To lock the valve open, re-tighten the knurled thumbscrew on the hose/valve fitting clockwise. Turn the sampling pump on to fill the bag.

5. When sampling is complete, turn the sampler off and loosen the knurled thumbscrew.

6. Close the shut-off valve by pulling the valve stem up (Note: be careful that the Teflon tube is not pulled off while pulling up on the valve stem.) Tighten the knurled screw to lock the valve closed.

Note: Tedlar® bags are not intended for storage of hazardous materials. If you suspect that your sample bags may contain hazardous materials in concentrations above safe levels defined by NIOSH, OSHA, or DOT, special federal and state packaging and transporting regulations may apply.

SHIPPING INSTRUCTIONS:

Samples must reach the lab as soon as possible after collection time. It is imperative that the lab be notified 24-48 hours before the sample is collected to ensure analyses.

Thank you for using USFilter, Enviroscan Services. Please call Customer Service at 1-800-338-7226 if you have any questions.

Ship to: **USFILTER, ENVIROSCAN SERVICES**
301 W MILITARY ROAD
ROTHSCHILD WI 54474

NOTICE: SHORT HOLDING TIMES

The following analyses have short holding times and may not be completed within the acceptable analyses time if USFILTER, ENVIROSCAN SERVICES is not notified prior to sample shipment.

Immediately:
Sulfite (SO3)

24 Hours:
Hex-Chrome (CR+6) water

48 Hours:
Biochemical Oxygen Demand (T.BOD,CBOD)
Color (True, Apparent)
Nitrate (NO3-N)
Nitrite (NO2-N)
Orthophosphate (OP)
Surfactants
Turbidity
VOCs in Tedlar Bags

Thank you for using USFilter, Enviroscan Services. Please call Customer Service at 1-800-338-7226 if you have any questions.

Ship to: **USFILTER, ENVIROSCAN SERVICES**
301 W MILITARY ROAD
ROTHSCHILD WI 54474

QC BOTTLE INSTRUCTIONS

AN ADDITIONAL 1 LITER AMBER BOTTLE MUST BE RETURNED TO USFILTER, ENVIROSCAN SERVICES "FILLED" FOR EACH OF THE FOLLOWING ANALYSES GROUPS. A PRELABELED BOTTLE WILL BE SHIPPED AND LABELED INDICATING THAT IT IS A "QC BOTTLE".

PAHs(8310)
PCBs(8082)
Pesticides(8081)
Semi-Volatiles(8270)
Herbicides(8151)
Phenols(604)

NOTE: 2 additional 1 liter amber bottles are required to be supplied per sample kit for Hexane Extractable Material (HEM-GREASE) by Method 1664A.

THE BOTTLES FOR QC ANALYSIS WILL BE PROVIDED TO YOU BY USFILTER, ENVIROSCAN SERVICES FREE OF CHARGE. IT IS THE RESPONSIBILITY OF THE SAMPLER TO FILL THE APPROPRIATE BOTTLES.

FAILURE TO FILL AND RETURN THESE BOTTLES TO USFILTER, ENVIROSCAN SERVICES WITH YOUR SAMPLE SET MAY SUBJECT YOUR SAMPLES TO DATA QUALIFICATION. DATA QUALIFICATION OF SAMPLES MAY LEAD TO REJECTION BY STATE REGULATORY AGENCIES.

Thank you for using USFilter, Enviroscan Services. Please call Customer Service at 1-800-338-7226 if you have any questions.

Ship to: **USFILTER, ENVIROSCAN SERVICES**
301 W MILITARY ROAD
ROTHSCHILD WI 54474

Appendix C

USFilter, Enviroscan Services Table of Analytical Qualifiers.

C	Confirmed. Compound found at the reported concentration and detected in the confirmation analysis above the specified limits.
CAL	Estimated concentration beyond the calibration range, but within the detector range of the instrument.
CC	Estimated concentration due to the calibration correlation coefficient not meeting the minimum requirements under Wisconsin NR149.
CE	Value for this compound may be elevated due to a compound(s) which shares retention time characteristics with the target compound.
CH	Confirmed HPLC compound. A positive spectral identification was confirmed on both wavelengths per photodiode array detectors.
CS	Confirmed Semivolatile Compound. A positive identification on both columns of a dual column GC system.
CSH	Check standard for this analyte exhibited a high bias. Sample results may also be biased high.
CSL	Check standard for this analyte exhibited a low bias. Sample results may also be biased low.
D1	The chromatogram is characteristic for a fuel oil/ diesel. (i.e. #1 or #2 Diesel, jet fuel, kerosene, aged or degraded diesel, etc.)
D2	The chromatogram is not characteristic for diesel. It has the characteristics of a product which has significant peaks within the DRO window.
D2A	The chromatogram is characteristic for a light petroleum product. (i.e. gasoline, aged or degraded gasoline, mineral spirits, etc.)
D2B	The chromatogram is characteristic for a heavier petroleum product other than diesel. (i.e. motor oil, hydraulic oil, etc.)
D3	The chromatogram is not characteristic for diesel or any single common petroleum product.
D4	The chromatogram contained significant peaks outside the DRO window.
D5	The chromatogram contained significant peaks and a raised baseline outside the DRO window.
DUP	Result of duplicate analysis in this quality assurance batch exceeds the limits for precision.
DWF	The dilution water dissolved oxygen depletion was above the method specific requirement for this sample.
G1	The chromatogram is characteristic for gasoline.
G2	The chromatogram has characteristics of an aged gasoline sample.
G3	The chromatogram in not characteristic for either gas or aged gas. It has a reportable concentration of peaks/area within the GRO window.
G4	The chromatogram contains a single compound which accounts for most of the GRO result.
G5	The chromatogram contains a significant number of peaks outside the GRO window.
G6	The chromatogram contains a significant number of peaks and a raised baseline outside the GRO window.
G7	The chromatogram is characteristic for gasoline, however either additional peaks are present or PVOC peaks are not proportional to gasoline, indicating the presence of additional compounds.
G8	The chromatogram is characteristic for aged gasoline, however either additional peaks are present or PVOC peaks are not propotrional to aged gasoline indicating the presence of additional compounds.
HT	Analyte analysis was completed outside of allowable holding time.
IB	Analyte observed in the instrument blank. Sample results may be biased high.
ISH	Internal standard recovery exceeds normal limits. Sample results may be biased low.
ISL	Internal standard recovery below normal limits. Sample results may be biased high.
J	Estimated concentration below laboratory quantitation level.
LBC	Analyte is a common laboratory solvent or chemical. Positive identification may be due to laboratory contamination. An additional qualifier of MB indicates this compound was also found in the Method Blank.
LCH	The laboratory control sample for this analyte exhibited a high bias. Sample results may also be biased high.
LCL	The laboratory control sample for this analyte exhibited a low bias. Sample results may also be biased low.
MB	Analyte observed in method blank. Sample results may be biased high.
MSH	Sample matrix spike recovery was high. Sample concentration may be biased high.
MSL	Sample matrix spike recovery was low. Sample concentration may be biased low.
NO	The compound was analyzed for confirmation but was not qualitatively confirmed.
OR	Instrument detector range below estimated concentration.
P	Sample aliquot was preserved at the time of sampling, but the preservative added was not sufficient to meet the preservation level required. Preservative was added in the lab prior to analysis.
RB	Analyte observed in reagent blank. Sample results may be biased high.
S1H	Sample matrix spike recovery was high. Sample result may be biased high.
S1L	Sample matrix spike recovery was low. Sample result may be biased low.
S2H	Sample matrix spike duplicate recovery was high. Sample result may be biased high.
S2L	Sample matrix spike duplicate recovery was low. Sample result may be biased low.
SCR	A low standard concentration equivalent to sample parameter was used.
SCF	The seed correction factor does not meet the method specific requirements for this sample.
SH	Surrogate recovery was high. Result for sample may be biased high.
SL	Surrogate recovery was low. Result for sample may be biased low.
SPH	Matrix spike recovery within analytical batch was high. Sample matrix appears similar to your sample; result may be biased high.
SPL	Matrix spike recovery within analytical batch was low. Sample matrix appears similar to your sample; result may be biased low.
UC	Unconfirmed. Compound initially found at the reported concentration, however confirmation of the analyte was below specified limits.
UCH	Unconfirmed HPLC Compound. A positive spectral identification was confirmed on fluorescence detector only.
UCS	Unconfirmed Semivolatile Compound. Compound was detected on one column of a dual column GC system.
XXX	Special qualifier.
YES	The compound was qualitatively confirmed.

Wisconsin Department of Natural Resources
STS Project No. 4-27393E

Appendix F

Field Audit Checklists

PRE-FIELD WORK PROCEDURES CHECKLIST - MONITORING WELLS

All the following procedures may not be necessary for each sampling event. Use those procedures applicable to your sampling plan or customize this list.

LOGISTICS

___ Arrange for site access with the land/home/facility owner and tenants.
___ Locate the nearest post office, UPS office, Fedex drop off spot, etc., if you will need to ship the samples from the field. (UPS has a 70 lb. restriction per container.)

LABORATORY ARRANGEMENTS

___ Select a qualified laboratory to perform the sample analysis. Check that the laboratory (and subcontracted lab) is certified to perform the required analysis.
___ Make sure you have sufficient numbers, types, and volumes of sample containers – **get extras!** Remember QA/QC sample containers and trip blanks.
___ Discuss sample preservation, holding time, shipping requirements, and QA/QC expectations with the laboratory.
___ Inform the laboratory of the date and number of samples you will send.
___ Familiarize yourself with chain of custody and other sample tracking procedures.

SITE HISTORY

___ Review past water quality data or SAP to determine the well sampling order.

EQUIPMENT AND FIELD PREPARATION

___ Review the sampling and analysis plan (SAP) and QA/QC plan.
___ Organize equipment (Equipment Checklist - Monitoring Well Sampling).
___ Check that equipment is in good working condition:

- ✓ Test and recharge/replace batteries as necessary.
- ✓ Test the equipment with tap water or calibration standards.
- ✓ Inspect the equipment for defects, loose bolts, frayed wiring, etc.
- ✓ Check the instruments' ability to calibrate and function properly.

___ Check that all equipment is properly decontaminated and stored for transport.
___ Fill out the Well Specific Field Sheet (WSFS) as much as possible before heading out to the field.

HEALTH AND SAFETY EQUIPMENT AND PREPARATION

___ If required, prepare and follow a health and safety plan (HSP).
___ Inform sub-contractors and other site personnel of contaminants and site hazards.

EQUIPMENT CHECKLIST - MONITORING WELL SAMPLING

All the following items may not be necessary for each sampling event. Check those items applicable to your sampling plan or customize this list.

GENERAL AND LOGISTICS

___ Permission/notification to land/home owner/tenant
___ Directions to the site and site access roads/site access keys
___ Contact names, addresses and phone numbers
___ Site map showing well locations, keys for well locks
___ Calculator and/or purge volume conversion tables
___ _____

DOCUMENTATION AND REFERENCE MATERIALS

___ *Groundwater Sampling Field Manual*
___ Sampling and analysis plan (SAP), QA/QC plan, and health and safety plan (HSP)
___ Well Specific Field Sheet (WSFS) and Field Procedures Documentation sheet
___ Well and boring logs
___ Field note book and waterproof pens
___ Clipboard with waterproof cover
___ Chain of custody forms and other sample tracking forms
___ Camera and film
___ _____

PURGING AND SAMPLING EQUIPMENT

___ Plastic sheet or equivalent ground cover
___ Purging pump or bailer and accessories (inert material)
___ Sampling pump or bailer and accessories (inert material)
___ Pump or bailer rope/cable (no cotton or cloth) and tripod
___ Pump sample tubing (inert material)
___ Pump power supply, air compressor, inert gas, etc.
___ Calibrated buckets or similar device for purge water
___ Waterproof grease markers or pens (Sharpies™ are a potential source of VOCs)
___ Sample containers (provided by lab) - **bring extra,** and water proof labels/tags
___ QA/QC sample bottles (VOC trip blanks filled by lab)
___ Sample transfer containers and wide mouth funnel
___ Filtering apparatus and all accessories
___ Filter membranes (0.45 micron) and pre-filters, or
___ Disposable in-line filters
___ 55 gallon drums for wastewater and drum labels
___ _____

FIELD MEASUREMENTS AND EQUIPMENT

___ Water level measuring instrument (0.01 foot increments) and backup device
___ Thermometer or temperature instrument
___ Conductivity meter and calibration standards (KCl)
___ pH meter, buffer solutions (pH 4, 7 and 10) and beakers
___ Dissolved oxygen meter and membrane replacement kit and/or Eh meter
___ Turbidity meter
___ All meters fully charged and operational; spare batteries
___ Closed flow through cell
___ Squirt bottles filled with reagent grade water
___ _____

DECONTAMINATION EQUIPMENT

___ Non-phosphate cleaner and scrub brushes
___ Wash and rinse tubs or buckets and wastewater containers
___ Laboratory reagent grade water (two gallons/well usually sufficient)
___ Clean containers to transport equipment
___ _____

SAMPLE PRESERVATION AND SHIPPING

___ Sample preservatives, transfer pipettes and pH paper
___ Coolers sufficiently large to hold all samples, including QA/QC samples
___ Crushed or cubed ice (frozen cold packs discouraged, need temp. blank)
___ Bubble wrap, Ziplock™ bags or equivalent to protect sample containers
___ Strapping tape, postage, Fedex or UPS shipping labels, COC forms, etc.,
___ _____

TOOLS AND MISCELLANEOUS

___ Extra locks, keys for wells, flashlight, rain gear, etc.
___ Propane torch for frozen locks and bolt cutters for corroded locks
___ Adjustable wrench, screw drivers, hammer, scissors, knife, duct tape, etc.
___ Plastic garbage bags for contaminated waste
___ Bailer retrieval device (e.g., weighted hook)
___ Drum bung wrench and racket socket set (typ. 15/16" socket for 55 gallon drums)
___ _____

PERSONAL PROTECTIVE EQUIPMENT

___ Respirators and cartridges (compatible for contaminants)
___ Safety glasses and/or splash shield
___ Inner and outer gloves (compatible for contaminants)
___ Hard hat and steel toed boots
___ Air monitoring equipment
___ First aid kit and eye wash kit
___ _____

WELL SPECIFIC FIELD SHEET - MONITORING WELLS (Sheet ___ of ___)

Facility/Project Name: _____ Date: _____
Section/Grid or Address: _____
License or Permit #: _____
Weather today and past weeks (precipitation): _____
Persons Sampling: _____

Well Name					
DNR Well ID No.					
Wis. Unique Well No. (WUWN)					
Damage to Well? (Y/N)					
Top of Casing or Reference Elevation (MSL)					
Depth to Water (to 0.01 ft)					
Groundwater Elevation (MSL)					
Depth to Well Bottom (ft)					
4 Well Volumes (gal. or liters)					
Purging Device; dedicated (D) or portable (PT)					
Purge Device Intake Depth (ft)					
Purging Time (start - stop)					
Average Purging Flow Rate (gpm or L/min)					
Volume Purged (gal. or liters)					
Purged Dry? (Y/N)					
Problems Purging? (Y/N)					
Sampling Device (D or PT)					
Sampler Intake Depth (ft)					
Average Sampling Flow Rate (gpm or L/min)					
Time Sample Collected					
Preservative (e.g., HCL)					
Field Temperature (°C)					
Field Specific Conductance @25°C (µMhos/cm)					
Time Measured					

Well Name					
Field pH (standard units)					
Time Measured					
Turbidity (NTUs or describe) (e.g. slight, moderate)					
Time Measured					
Dissolved Oxygen (mg/l)					
Time Measured					
Eh - redox potential (mv)					
Time Measured					
Color (describe - grey, etc)					
Odor (describe - pungent, etc)					
Sample Field Filtered? (Y/N)					
Time Samples Filtered					
Well Capped & Locked? (Y/N)					

Comments (Discuss well damage, purging or sampling problems, deviations from sampling plan, etc.):

Sheet Completed by _____ Date _____

GROUNDWATER SAMPLING FIELD PROCEDURES DOCUMENTATION

Facility/Project Name: _____ Date: _____

Section/Grid Location or Address: _____

Facility Type: _____ License/Permit #: _____

DNR Regulatory Program: _____

Weather (temp., cloudiness, bar. pres., wind): _____

_____

Persons Sampling and Title: _____

_____

_____

Water Level Equipment (type, model): _____

Purging Equipment (type, model, material): _____

_____

Purging Method (4 well vol. or stabilization): _____

How Purge Volume Measured? (eg., calibrated bucket): _____

Sample Collection Equipment (type, model, material): _____

_____

Method of Sample Withdrawal (bottom emptying device, low flow): _____

Type of Transfer Containers: _____

Filtering Equipment (type, material): _____

Filter Membrane (type, pore size): _____

When Were Samples Sent to Lab? _____

What Lab Were the Samples Sent to? _____

Were Enforcement Samples Sent? _____

How Were Samples Kept Cool (ice, other)? _____

Equipment Decontamination Procedures? _____

_____

Decontamination Water Disposal? _____

_____

pH Meter (type, model): _____

Person calibrating: _____

Frequency calibrated: _____

Calibration procedures (buffers used): _____

Problems with meter: _____

Conductivity Meter (type, model): _____

Person calibrating: _____

Frequency calibrated: _____

Calibration procedures: _____

Problems with meter: _____

Turbidity Equipment (type, model): _____

Person calibrating/set-up: _____

Frequency calibrated: _____

Calibration procedures: _____

Problems with meter: _____

Dissolved Oxygen Meter (type, model): _____

Person calibrating/set-up: _____

Frequency calibrated: _____

Calibration procedures: _____

Problems with meter: _____

When Were In-field Measurements Taken (immediately after collection or XX minutes after collection)?: _____

Comments (difficulties, questionable data, deviations from sampling plan, etc): _____