Quality Assurance Manual

Environmental Health Division

Wisconsin State Laboratory of Hygiene
University of Wisconsin

This manual applies to accredited (1.1) methods within the following analytical departments:

Environmental Toxicology Department
Inorganic Chemistry Department
Trace Element Clean Lab
Metals Department
Organic Chemistry Department
Radiochemistry Department
Water Microbiology Department

This manual also includes the following support departments:

Operations (Shipping/Receiving
Horizon Data Management, Sample Receiving, & Customer Service)
Glassware/Media

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Revision 18 – June, 2020

Signature Page

Name	WSLH Position (applicable to this QA Manual)	[1]TNI Technical Manager?	Signature	Date
David Webb	Director, EHD Associate Director, WSLH	Yes		
Noel Stanton	Lab Mngr., Envir. Chemistry (incl. Organic, Inorganic Chem., Metals)	No		
Jocelyn Hemming	Mngr., Envir. Microbiology (incl. Water Micro., Envir. Tox., Glassware/Media)	No		
Graham Anderson	Chem. Supervisor, Inorganic Chemistry & Metals	No		
Dahman-Zaborske, Christa	Chem. Supervisor, Trace Element Clean Lab (TECL)	No		
Erin Mani	Chem. Supervisor, Organic Chemistry	No		
Martin Collins	Micro. Supervisor, Water Microbiology & Glassware/Media	No		
Dawn Perkins	Env. Health Supervisor, Envir. Tox.	No		
vacant	Chemistry Supervisor, Radio Chemistry	No		
Tracy Hanke	Mngr., EHD Operations (incl. Horizon Data Management, Sample Receiving, Customer Service, & Shipping/Receiving)	No		
Kathleen Dax-Klister	Supervisor, HDM, Sample Receiving, & Customer Service	No		
Barb Woehrl	Supervisor, Shipping/Receiving	No		
Susan Hill	QA Coordinator, Adv. Chemist	No		
Donna Johnsen	QA Coordinator, Senior Chemist	No		
Camille Danielson	QA Coordinator, Administrative Program Specialist	No		

[1]2009/2016 TNI 4.1.7.2—see section 1.1

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1. General Sections

1.1. Applicability

This Quality Assurance Manual is designed to meet requirements of TNI (NELAP), Wisconsin Department of Natural Resources (NR 149), and EPA. Testing conducted under any of these accreditations, at any WSLH facility (see 1.17.7), must meet the requirements set forth in this manual. Testing conducted under other accreditations or non-accredited testing may use this manual as is to meet requirements. If other accrediting agencies have additional requirements not covered in this manual, those must be met in ancillary documents.

Note: TNI (The NELAC Institute) was formed in 2006 when NELAC (National Environmental Laboratory Accreditation Conference) combined with INELA (Institute for National Environmental Laboratory Accreditation). NELAP (National Environmental Laboratory Accreditation Program) is one program operated by TNI. For more information, see https://nelac-institute.org/content//programs.php

1.2. Manual Organization and Maintenance

1.2.1. 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 NELAP 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 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 now Environmental Toxicology (Whole Effluent Toxicity), Inorganic Chemistry, Trace Element Clean Lab, Metals, Organic Chemistry, Radiochemistry, and Water Microbiology. Annual reviews resulted in minor revisions (designated as 3.1, 3.2, etc.)

Within revision 4.0, it was necessary to update the NELAC concordance to comply with

the new numbering scheme of the 2003 NELAC standards.

Revision 5.0 was completely reorganized so that each department would have a separate chapter devoted to it.

Revisions 6.0, 7.0, and 8.0 were reviewed by each department and changes implemented as necessary.

Revision 9.0 was re-written and a new concordance compiled to comply with the new TNI (The NELAC Institute) Standard EL-V1-2009, which is consistent with ISO/IEC 17025:2005 requirements that are relevant to the scope of environmental testing services.

Revisions 10.0 and 11 were reviewed by each department and changes implemented as necessary.

Revision 12 was completely reorganized by developing a template with required sections and content for each departmental chapter. The goal was to make the manual more concise, functional, and organized.

Revisions 13, 14, 15, 16, and 17 were reviewed by each department and changes implemented as necessary. Rev. 18 incorporated some updates and notes to correspond with 2016 TNI standards, and the concordance chart was updated for 2016 TNI.

1.2.2. Structure

Chapter one contains general information that applies to all departments covered by this manual. Each department then has a separate chapter that contains information specific to that department. These departmental chapters are electronically saved in separate files, which are linked through chapter one. Annually, analysts are required to review chapter one as well as the individual departmental chapter that applies to them.
The manual is currently constructed using Microsoft Word 2010. As software is updated the manual format may change as necessary.

1.2.3. Maintenance

Annually, each department will review and update its chapter of the manual. In addition, the Quality Assurance Officers will review and update the general chapter. Any necessary changes will be submitted to the QA team leader or designee, who will then finalize the manual and publish a new revision. Copies may be made available in hardcopy form to the various departments. The electronic version of the manual will reside on the shared server used by the EHD. Specifically, it will be located at O:\SOP\EHD\Division Wide\Final

Although the manual will be updated at a minimum on an annual basis, changes may occur at any time. Major changes will result in a new revision of the Manual. Each new revision will have a new revision number, and effective date. 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.

1.3. Quality Policy Statement

This quality policy statement describes the overall objectives of the Environmental Health Division's quality system. The complete quality system is documented in the remainder of this Quality Manual and in policies and procedures that are referenced in this manual. This quality system is based on the required elements contained in the 2009/2016 TNI (The NELAC Institute) Standard, Volume 1 "Management and Technical Requirements for Laboratories Performing Environmental Analysis." The management of the Environmental Health Division, under the authority of the division director, is committed to these quality objectives as a means of maintaining our status as a world class public and environmental health laboratory.

Objectives of the Quality System:

High accuracy of work

The Environmental Health Division's quality system ensures that data is of excellent and documented quality. The management and laboratory professionals are committed to following good professional practice as defined by our quality system and to compliance with applicable standards.

Data Integrity

Management is committed to ethical laboratory practices, and all employees are responsible for following the data integrity, ethics, and data documentation policies, which in turn, assure high quality data.

Continued quality improvement

Laboratory management is dedicated to continued quality improvement by means such as corrective and preventive action when warranted, root cause analysis, internal audits, and management system reviews. All staff are encouraged to bring suggestions for quality improvement to supervisors.

High customer satisfaction

The laboratory's standard of service to our clients and the citizens of the State of Wisconsin includes meeting all the objectives of our quality system as well as providing timely results, staying fiscally responsible, and addressing client concerns and complaints. In addition, our clients often call on us to provide outreach and outside training. Research and method development work are also requested, although this usually would fall outside of any accredited methods.

Compliance with applicable standards

The Environmental Health Division conducts testing that may be regulated under one or more of the following agencies: the United States Environmental Protection Agency, the Wisconsin Department of Natural Resources, The NELAC Institute, the Wisconsin Division of Health, Wisconsin Department of Agriculture, Trade and Consumer Protection, and local public health agencies. The management and laboratory professionals are committed to upholding the requirements of these standards.

Staff training

Training includes initial and continuing instruction on the quality system documented in this manual and referenced policies and procedures as required for specific job duties. The training ensures that the quality system is communicated to, understood by, available to, and implemented by the appropriate personnel.

1.4. Organization, Management Structure, and Responsibilities

Organizational charts can be found at O:\Organizational Charts.

The Wisconsin State Laboratory of Hygiene is a department of the University of

Wisconsin—Madison within the School of Medicine and Public Health. 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.

University of Wisconsin-Madison Dept.	**Director**	**Assoc. Director**
Wisconsin State Laboratory of Hygiene	Dr. James Schauer*	Dr. Peter Shult

*Address: 465 Henry Mall, Madison, WI 53706

1.4.1. Divisions

The Wisconsin State Laboratory of Hygiene (WSLH) is divided into several analytical divisions including the Environmental Health Division (EHD).

Division Directors report directly to the Laboratory Director, and are responsible for managing operations of the divisions. In addition, Division Directors are ultimately responsible for data quality and compliance with applicable standards. They ensure the integrity of the management system when changes are planned and implemented. In the absence of the Division Director, a temporary appointee will act in his/her place.

WSLH Division	**Director**
Environmental Health	David Webb

1.4.2. Offices

The laboratory's analytical divisions are supported by the following: Office of Information Systems, Office of Finance (includes Purchasing, Accounts Receivable, & Accounting departments), Office of Human Resources, Office of the Director.

1.4.3. Departments

The EHD is divided into several departments: Operations (including Shipping/Receiving, and Horizon Data Management/Sample Receiving/Customer Service), Environmental Chemistry (including Organic Chemistry, Air Chemistry, Inorganic Chemistry, Metals, and Chemical Emergency Response), Trace Element Clean

Lab, Environmental Microbiology (including Environmental Toxicology, Water Microbiology, Glassware/media, and Flow Cytometry), Radiochemistry, and Forensic Toxicology. Also within the EHD is the National Atmospheric Deposition Program.

All of the departments included in this QA Manual are part of the EHD. The Metals department does work for both EHD and Occupational Health & Safety Division.

Each Department has a Department Supervisor who is responsible for day-to-day operation of the laboratory. Department Supervisors report to the Division Director. As a group these supervisors oversee technical operations, sample analysis, quality assurance activities, data entry, report generation, provision of resources, and all other related areas. In addition, they are responsible for employee management and review. Supervisors will appoint a person or persons to cover their duties during an absence. If substitution appointments have not been made in advance, managerial decisions will be made by the Division Director.

Departments covered by this QA Manual:

Department	**Supervisor**
Envir. Chemistry (Inorganic Chemistry)	Noel Stanton (Graham Anderson)
Envir. Chemistry (Metals)	Noel Stanton (Graham Anderson)
Envir. Chemistry (Organic Chemistry)	Noel Stanton (Erin Mani)
Trace Element Clean Lab	Christa Dahman Zaborske
Radiochemistry	Vacant (David Webb, acting)
Envir. Micro. (Water Microbiology/ Glassware/Media)	Jocelyn Hemming (Martin Collins)
Envir. Micro. (Environmental Toxicology)	Jocelyn Hemming (Dawn Perkins)
Operations (Horizon Data Management/ Customer Service/Sample Receiving & Shipping/Receiving)	Tracy Hanke (Kathleen Dax-Klister & Barb Woehrl)

1.4.4. QA Officers (QA Coordinators)

The QA Officers are responsible for implementing and maintaining quality assurance procedures throughout the laboratory, and ensuring compliance with applicable standards. They work with the supervisors and division directors to insure that QA procedures are followed by all staff. Responsibilities of the QA Officers include oversight of the procedures that generate quality control data. QA Officers are also

responsible for conducting internal audits annually. The internal audit reports include a listing of deficiencies and a means of monitoring corrective action. The QA Officers oversee the laboratory's certification status and coordinate the various regulatory programs. They also maintain working relationships with regulatory agencies and closely monitor any program or statutory changes. Other duties of QA Officers include managing performance evaluation (proficiency testing) samples, coordinating the review and writing of SOPs (including the Quality Assurance Manual), and evaluating QC limits.

The QA Officers report to the Division Director. Their QA duties are independent of any laboratory work that they may perform, or oversight is such that objectivity is maintained.

All QA Officers have knowledge in the quality system as defined under TNI, DNR, & EPA and experience in the concomitant QA/QC procedures. Documentation of this knowledge and experience includes dated signatures on the QA Manual, SOP's, DOC statements, and other documents that are part of the quality system. QA Officers also have general knowledge of the analytical test methods performed in their departments.

If a quality assurance question arises when the QA Officer for a particular department is absent, the question may be directed to either the department supervisor or a QA Officer from another department.

1.4.5. 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.

1.4.6. Position Descriptions

Specific position descriptions for all personnel are located in the main WSLH Human Resources Office (465 Henry Mall), and on-line at https://www.ohr.wisc.edu/weblisting/classified/approvedpdwebgrid.aspx. In addition,

each Department Supervisor has copies of the position descriptions for their staff. Organizational charts for WSLH and all divisions are located at O:\Organizational Charts.

1.4.7. Education and Experience—Division-wide Employees

Name	**Title**	**Degree**
David Webb	Division Director (EHD)	BS Chemistry, MS Environmental Science
Susan Hill	Chemist Advanced / QA Coordinator	BS Chemistry, MS Water Chemistry
Donna Johnsen	Chemist Senior / QA Coordinator	BS Chemistry

1.5. Security and Access

Access to the Wisconsin State Laboratory of Hygiene (WSLH) Agriculture Drive site is 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 are open to our customers from 7:45 a.m. to 4:30 p.m., Monday through Friday. Saturday hours are 6:30 a.m. – 12:30 p.m. 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. LABWIDE GENOP 1101, "Visitor Access to WSLH Facilities and LABWIDE GENOP 1004, "Building Access "Authorization" detail the specifics of security and access.

1.6. Service to the Customer & Complaint Resolution

The WSLH will maintain communication with customers to ensure testing is completed as requested by the customer or according to agreement between the customer and the lab (also see section 1.15, Review of Requests, Tenders, and Contracts). Any questions, problems, or major delays with a sample will be communicated to the customer as appropriate. Documentation of customer communications will be maintained. Sampling kits or other supplies needed by customers will be sent out in a timely manner according to procedures (see internal website, Divisions & Offices/Environmental Health Division/EHD Customer Service). Questions will usually be answered by Customer Service representatives. Technical questions regarding specific tests can be passed on to

departmental representatives.

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. EHD GENOP 017 "Managing Customer Feedback" details how complaints and other feedback should be handled and documented.

The Environmental Health Division seeks feedback from clients in regular meetings with the Department of Natural Resources and the Department of Health Services.

1.7. Confidentiality Policy

We will handle oral or written requests for sample results according to the following policy:

We will release sample results to the person(s) or entity that submitted the sample and/or is paying for the testing. We will also release sample results to the person(s) or agency that was identified as the receiver of the report.

Note: Under Wis. Stat. 280.13(1)(d) and Admin. Rule NR 812 private drinking water test results are released electronically to the WDNR.

If information is requested under the Freedom of Information Act, the request is forwarded to the WSLH Director's Office.

Most test results within the EHD are not considered protected health information under the Health Insurance Portability and Accountability Act (HIPAA).

Laboratory records that support accreditations will be made available to the applicable accreditation bodies. These include, but are not limited to quality records, technical records, all information necessary for the historical reconstruction of data, administrative records, purchasing records, and personnel records.

Personal information about all staff of WSLH is considered confidential. Conversations regarding staffs' personal and professional information must be conducted in a site where confidentiality can be maintained.

1.8. Hiring Process

The Wisconsin State Laboratory of Hygiene (WSLH) is part of the University of Wisconsin and conforms to the University of Wisconsin's policies for hiring staff. These requirements are designed to ensure the laboratory's ability to hire highly qualified personnel. The WSLH Office of Human Resources (OHR) is responsible for developing and maintaining all policies, procedures, and documentation related to hiring all personnel at the WSLH. All records associated with hiring staff for the WSLH will be retained by the Office of Human Resources. OHR will be responsible for the maintenance and final disposal of these records. For details on the hiring process please see HR Website http://slhicmsprod/administrative-services/human-resources/.

1.9. Safety

The "Chemical Hygiene Plan and General Laboratory Safety Plan" (AG DR SAFETY GENOP 102), contains comprehensive information on general laboratory safety procedures and operations for the WSLH's Agriculture Drive facility. Information includes chemical storage, waste disposal, safety showers, fume hoods, controlling exposure, employee safety training, housekeeping, emergency procedures, eye protection, personal protective equipment, and more.

At the University level, see Environment, Health, and Safety Division of Facilities Planning and Management, University of Wisconsin—Madison, https://ehs.wisc.edu/, (608) 265-5000. Their "Laboratory Safety Guide" is a resource guide geared toward promoting safe use and disposal of hazardous materials as well as general safety and regulatory awareness. It is published by the Office of Chemical Safety and is a collection of valuable information. Lab Safety Guide: https://ehs.wisc.edu/laboratory-safety-guide/

The WSLH has a Health and Safety committee that meets regularly and conducts safety inspections. Minutes of this committee are available at O:\Teams\Safety\Minutes and Attendance. Membership of this committee consists of a cross section of laboratory personnel.

1.9.1. Note regarding Safety Data Sheets

A Safety Data Sheet (SDS) is a document containing chemical hazard and safe handling

information. There are 16 sections including: Section 2: Hazard identification, Section 4: First-aid measures, Section 7: Handling and storage. For more information about Safety Data Sheets see the Chemical Hygiene Plan and the Laboratory Safety Guide noted above.

1.10. Training

1.10.1. Initial Training of New Employees

Training of employees takes place in a logical progression that meets applicable requirements. The Office of Human Resources has a new employee checklist (http://slhicmsprod/administrative-services/human-resources/) that includes some lab-wide training items (click on the New Employee tab). HR also offers on-boarding materials.

WSLH has an Employee Handbook available through the intranet home page: http://slhicmsprod/ (click on button near center of the home page).

To ensure 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. A key tool in safety training is the Agriculture Drive Employee Safety Checklist (click on the New Employee tab) (http://slhicmsprod/administrative-services/human-resources/), which comprehensively lists safety issues such as the location of safety showers and fire extinguishers, evacuation procedures, policies on eating and drinking in the lab, use of potentially dangerous instruments and chemicals, safety apparel use, fume hood use, and much more. The AD Employee Safety Checklist also lists external references that contain more information on laboratory safety. Other safety training tools that new employees are required to review are the AG Drive Chemical Hygiene Plan (AD SAFETY GENOP 102) and the EHD Emergency Action Plan (AD SAFETY GENOP 101). Depending on their position, some employees may require more specialized instruction such as a 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 receive training in the "Handling and Shipping of Hazardous Materials," also offered by the University.

New employee training continues with:

- Data Integrity training (EHD GENOP 029)
- A review of the EHD Quality Assurance Manual
- Occurrence management training (also see section 1.21.5). The occurrence management training is available on the intranet (on the home page, hover over the Regulatory & Compliance drop-down list, choose the Quality Management button, then click on the Quality Tools tab (http://slhicmsprod/regulatory-compliance/quality-management/).
- HIPAA—Varying levels of HIPAA Privacy Rule training are required depending on an employee's position. For HIPAA training information, consult HR and see the University of Wisconsin—Madison, Office of Compliance, HIPAA training website: https://compliance.wisc.edu/hipaa/training/.

Each department has a new employee training form or checklist (see departmental chapters), which ensures that a new employee receives information important to working in that department. Usually an experienced employee guides the new employee through the checklist.

These items complete the initial training of new employees. Next they will move on to analytical method training if required for their position. Other non-testing training materials may be required by the departments.

1.10.2. Analytical Method Training

Analytical method training is for new employees who have completed the initial training and for any employee who is learning a new procedure. Departments may have method training forms (see departmental chapters), which help guide a person through the process of learning a new method. Generally, the trainee will review the department's SOP and/or the regulatory method as well as the instrument manual. He/she will observe an experienced analyst prepare samples and operate the instrument. He/she will next work under the direct supervision of the experienced analyst until becoming familiar with the analytical procedures. Training includes sample handling and preparation, safety specific to the method, documentation procedures, calibration procedures, QC requirements, data management, data reporting, and troubleshooting.

If applicable, the trainee will perform an initial Demonstration of Capability (DOC) and

document the results on a DOC Certification Statement (EHD QA 115, "Initial and Ongoing DOC Procedures"). In addition, the trainee must sign a Certification Statement for Current Technical Methods (Figure 1) or an attestation statement at the end of an SOP, which states that he/she has read, understood, and agreed to perform the most recent version of the test method. When initial DOC 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 his/her own with only routine supervision.

1.10.3. Continuing Training

All employees receive continuing training and must demonstrate continued proficiency. Whenever there is a change in instrument type, personnel, or test method a new DOC must be performed. Annually, each analyst must demonstrate continued proficiency on technical methods for which they are responsible (see departmental chapters for procedures). When a new revision of an SOP is written, analysts who are responsible for that method must sign a new Certification Statement for Current Technical Methods.

All employees must annually review the following documents:

a. Quality Assurance Manual
b. Chemical Hygiene Plan
c. Data Integrity, Ethics, and Data Documentation Procedure
d. Emergency Action Plan

1.10.4. Additional Education/Training

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.

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.

1.10.5. Training Documentation

All training forms, checklists, sign-off sheets, certification statements, and DOC forms related to the above requirements will be signed and dated by the employee and given to the QA coordinator responsible for that department. The QA coordinator will insure that

the documentation is complete and filed appropriately. Most training documentation will be filed in the personnel training files in each department.

1.10.6. Data Integrity Procedures

All employees have access to the Data Integrity, Ethics, Data Documentation Procedure for the WSLH, Environmental Health Division (EHD GENOP 029). The procedure includes our organizational mission relating to data integrity, our steps for data integrity training and training documentation, our methods for monitoring data integrity, and steps for the reporting of data integrity concerns. In addition, it documents our Ethics Policy and our Data Documentation procedure.

1.10.7. Employee Training Summary Tables

Initial Training of New Employees

Training Document	Link or Reference to Document	Documentation of Training
Lab-wide new employee checklist	http://slhicmsprod/administrative-services/human-resources/	Sign checklist
Dept. new employee checklist	See departmental chapters	Sign checklist
EHD Employee Safety Checklist	http://slhicmsprod/administrative-services/human-resources/	Sign checklist
EHD Chemical Hygiene Plan	O:\SOP\Safety\Final\AD_SAFETY_GENOP_102_Chemical Hygiene Plan.doc	Sign-off sheet
EHD Emergency Action Plan	O:\SOP\Safety\Final\AD_SAFETY_GENOP_101_Emergency Action Plan.doc	Sign-off sheet
EHD Data Integrity Procedure	O:\SOP\EHD\Division Wide\Final\EHD GENOP 029 SOP	Sign-off sheet
QA Manual	O:\SOP\EHD\Division Wide\Final	Sign-off sheet
Occurrence Management	http://slhicmsprod/regulatory-compliance/quality-management/	Training form/attestation statement

Analytical Method Training

Training Document	Link or Reference to Document	Documentation of Training
Method Training form	See departmental chapters	Sign form
DOC Certification Statement	EHD QA 115	Sign DOC statement
Cert. Statement for Current Technical Methods	QA Manual Figure 1 (Note: other forms such as SOP Attest Statement are also acceptable)	Sign Certification statement

Continuing Training

Training Document	Link or Reference to Document	Documentation of Training
DOC or Annual Continued Proficiency documentation (annually or when there is a change)	EHD QA 115	Sign DOC statement or other doc.
Cert. Statement for Current Technical Methods (when new SOP revision is written)	QA Manual Figure 1 (Note: other forms are also acceptable)	Sign Certification statement
QA Manual (annually)	O:\SOP\EHD\Division Wide\Final	Sign-off sheet
EHD Chemical Hygiene Plan (annually)	O:\SOP\Safety\Final\AD_SAFETY_GENOP_102_Chemical Hygiene Plan.doc	Sign-off sheet
EHD Data Integrity Procedure (annually)	O:\SOP\EHD\Division Wide\Final\EHD GENOP 029 SOP	Sign-off sheet
EHD Emergency Action Plan (annually)	O:\SOP\Safety\Final\AD_SAFETY_GENOP_101_Emergency Action Plan.doc	Sign-off sheet

Additional Education & Training

Training Document	Link or Reference to Document	Documentation of Training
Course or class work		Certificate or note about title and content of course

Figure 1: Example of a Certification Statement for Current Technical Methods

Analyst Cert. Statement
Rev. 2
April, 2015
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Wisconsin State Laboratory of Hygiene
Environmental Health Division

Analyst Certification Statement
For Current Technical Methods

Analyst Name: _____

SOP Number: _____

SOP Title: _____

SOP Revision Number: _____

SOP Effective Dates: _____

I, the undersigned, CERTIFY that I have read, understood, and agreed to perform the most recent version of the above test method.

_____ _____
Analyst Signature Date

1.11. Document Control System

1.11.1. Control & Maintenance of Standard Operating Procedures (SOPs)

SOPs that may apply to operations covered by this QA Manual exist at three different levels:

Lab-wide SOPs: O:\SOP\Labwide SOP Policies\Final

Lab-wide SOP table of contents: O:\SOP\Labwide SOP Policies\Final\001_TOC_FOR_LABWIDE_SOPs.xls

Division-wide SOPs: O:\SOP\EHD\Division Wide\Final

Division-wide SOP table of contents: O:\SOP\EHD\Division Wide\Final\001 TOC Internal Documents.xlsx

Department-level SOPs: see departmental chapters for details

There are three SOPs within the EHD that document information about writing and managing SOPs within the division. EHD GENOP 101, "EHD SOP Directory Structure" describes the electronic directory structure wherein EHD SOP documents (both division-level and department-level) are saved. EHD GENOP 102, "Guidance for Writing and Managing Division-Level SOPs" gives guidance for writing SOPs that apply to the entire EHD. EHD GENOP 103, "Guidance for Writing and Managing Department-Level SOPs" gives guidance for writing SOPs that apply to departments within the EHD. Most departments also have an SOP that describes in more detail how SOPs are managed within that department (see departmental chapters). Analytical method SOPs will contain all TNI-required sections and information.

All SOPs associated with drinking water testing (including administrative as well as technical) must be reviewed for content annually and updated if necessary.

Schedules for the reviews of division-level SOPs and this QA Manual can be found at O:\Teams\EHD QC Team\Schedules

1.11.2. Components of Document Control

All internally-generated documents (SOPs, policy statements, spreadsheets, forms, bench record cover sheets, certification statements, sign-off sheets, instruction sheets, etc.) must

be listed in a table of contents and have document control [TNI V1M2 4.3.2.3; WI NR 149.39(1)] including:

a. Issuing authority (e.g. WSLH, division, department)
b. Unique ID (number or title)
c. Effective date and/or revision number
d. Page x of y

1.12. Records Retention, Control, & Storage

1.12.1. Records Disposition Authorization (RDA)

RDAs are also called records schedules. They are key documents for establishing a records management program for organizations within Wisconsin State government. In essence, records schedules describe the organization's information resources, how long they are going to be retained, and what their ultimate disposition will be. They are the policy statements that govern the ultimate disposition of records.

Some EHD records fall under General Records Retention Schedules (GRS), which are approved for UW-Madison campus-wide use. General records schedules codify retention policies for record types that are common to all offices across the UW system. Refer to https://www.library.wisc.edu/archives/records-management/retention-disposition/general-records-schedules/

The EHD also has a unique records schedule, which covers records not included in any campus-wide GRS. It can be found at: O:\RDA's\Final RDA\EHD_2017_WSLH.pdf

If an RDA needs to be amended or resubmitted upon 10-year sunset, contact:

University Records Officer, University of Wisconsin-Madison
432 Steenbock Library
550 Babcock Drive
Madison, WI 53706-1201
Phone: 608-262-3284

recmgmt@library.wisc.edu

1.12.2. Laboratory Notebook/Logbook

A laboratory notebook or logbook is any physical book in which testing data is recorded; this includes experimental data, standards logs, instrument logs, etc.

All laboratory notebooks are assigned a unique number by QA staff. This number is then used for tracking notebooks in the laboratory. Each notebook will be labeled with the following information: organization name, unique laboratory notebook number, analyst or instrument assigned to, department, notebook's contents, start date and end date.
All notebook information on the label is entered into a database, which also includes information on archival date, storage location, archival box number and disposal date. The database is on drive: R:/EHD/QC/Archived Records.mdb
Notebooks will be retained according to the applicable RDA.

1.12.3. Storage of Records in Basement room 14

Refer to Labwide GENOP 1002, "Records Storage and Disposal," section 4.0 for the procedure for storing documents in record storage boxes. Boxes may be immediately sent to the State Records Center or may be placed in an approved storage area at the WSLH for up to three years. Approved storage areas are organized with specific locations where new inventory may be added. The approved storage area for Agriculture Drive is room 14 in the basement. Complete records of items placed in storage must be maintained by the divisional or departmental records coordinator. The records coordinator has the responsibility for knowing what is in each box, where each box is located, and the destruction date of each box.

If it is necessary to temporarily remove records (under NELAC-accreditation) from boxes stored in room 14, information including date removed and returned will be recorded in the Archived File Checkout logbook (ESS801) that is hung near the entrance of room 14 nearest the stairwell. Logbook pages can be printed from the file located at: R:\EHD\Forms\Drafts\Archived File Checkout Log for AD basement EHD rev. 2.doc

Labwide GENOP 1002 also details the procedure for immediate destruction of records and the procedure for electronic records.

1.13. Traceability of Measurements

All analytical results and measurements are fully traceable to standards, reagents, reference materials, and instrumentation used in deriving the results. Stock and working standards are given codes that are documented in the analytical runs in which they are used. Reagents (including pH paper and other chemical test strips) are also given

traceability codes. Standards along with effective and expiration dates are retained in the LIMS and linked to the batches and samples associated with them. Analytical instrumentation and equipment (including filters, pipets, and thermometers) are assigned identification numbers, which are also tracked at the batch level in analytical runs.
Date and time of analysis and analyst initials are documented at the batch and/or sample level with all functions in the Horizon LIMS being marked with date/time/users initials. Test results are entered into LIMS either automatically from the instrument or through manual entry by the analyst. All results are directly linked to each sample.

Individual departments may have more detailed information on how they achieve traceability.

1.14. Policy for Estimating Uncertainty of Measurement

Each department must write a policy or procedure for estimating uncertainty of measurement. The policies will cover all analytical tests performed by the department. The uncertainty needs to be reported only if required by the regulating agency, but it must be available upon request. The policies must attempt to identify all the components of uncertainty, make a reasonable estimation, and ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation will be based on knowledge of the performance of the method, on the measurement scope, and will make use of previous experience and validation data.
If the department analyzes samples following a well-recognized test method, which specifies limits to the values of the major sources of uncertainty and also specifies how calculated results will be reported, then the department meets the uncertainty requirements for that method.

1.15. Procedures for Accepting New Work/Review of Requests, Tenders, & Contracts

1.15.1. Requests for service

All requests for analysis are reviewed by department staff. WSLH Board approval is not needed for our environmental departments to do work for units of government (WDNR, DHHS, USGS, etc.). Established and new tests can be offered to units of government.

Our approved environmental tests that we can offer to the public on a fee for service basis include the tests on our "public" price list: http://www.slh.wisc.edu/environmental/water/public-environmental-and-water-testing-prices/. The prices for these tests are submitted for Board approval every year.

To offer a new test to the general public (which could include non-governmental entities such as a paper mill) on a fee for service basis, Board approval is needed. Three areas in which a new test may be brought to the board for approval are 1) An emergency or urgent need as indicated by a state agency or local public health agency; 2) State agency determines a testing service is necessary; 3) WSLH wishes to offer a test to the public for a fee. For more information, consult with Office of the Director.

We also offer fee for service tests to public water suppliers, mostly government entities but including some non-governmental public water supplies (e.g., a private campground). The testing of public water suppliers by the State Laboratory has a long history, and WDNR has reaffirmed several times the importance of the State Laboratory doing this testing. Microbiological testing makes up the majority of these tests, and is funded through the WDNR's GPR allocation to the State Laboratory (i.e., the basic agreement). Other testing (e.g., nitrate, volatile organics) is charged back to the public water supplier. Note that these drinking water tests that are not offered to the general public have not been part of the Board pricing exercise. Prices are jointly determined by WDNR and WSLH and updated annually.

1.15.2. Review of laboratory capability

If new work is to be accepted based on the authority granted in the above references, a review of laboratory capability and the scope of work is conducted. The following items may be considered:

- Availability of desired analytical methods and accreditations
- Adequate capacity for facilities and instrumentation
- Adequate staffing, training, and experience (both analytical and support staff)
- Capability to meet desired LODs and LOQs
- QA/QC that meets data quality objectives
- Desired turn-around-time

- Desired reporting capabilities and information technology resources

Contracts with various entities follow the customary Board notification and/or approval process, and are also subject to UW policies and procedures.

Contact the Office of Finance for any research, fee-for-service, academic (and other) agreements, grants, contracts, leases or purchase orders from an outside entity that require a signature from WSLH. Specifications for terms and conditions that can be agreed upon within these items are set by the Wisconsin State Legislature, WSLH and UW System by-laws, and Federal regulations.

1.15.3. Review of requests for service

Routinely, the lab receives samples or requests from the public and WDNR staff who are not sure what they want the samples to be tested for. In other cases the client has asked for tests that may not be ideal for their samples. In these cases, WSLH staff work with the client to determine the best tests that will meet their objectives. When samples and test request/chain of custody forms have already been received at the lab, these communications are documented either on the paperwork (which will then be scanned and saved in Horizon) or in Horizon. E-mail communications with clients may also be attached to the sample in Horizon. Each contract (written or oral agreement to provide a customer with testing services) will be acceptable both to the lab and the customer; any differences will be resolved prior to work commencing on the samples.

Records will be maintained of reviews of requests, tenders, and contracts and pertinent discussions with a customer relating to the customer's requirements, contracts for work, the results of work, any deviation from the accepted contract, and any significant changes to the work being done. If a contract needs to be amended after work begins, the review process noted above will be repeated and all changes communicated to affected personnel. These records may be in the form of comments appended to test results in Horizon, e-mails, notes from phone conversations, or written correspondence.

This process also applies to sample testing that may need to be sub-contracted (see section 1.23). The complete history of client and staff agreement regarding sample testing

will be traceable through Horizon and associated documentation. See also section 1.6, Service to the Customer and Complaint Resolution.

Additional details regarding accepting new work may be found in the departmental chapters.

1.16. Sample Handling and Submission Procedures

1.16.1. Sample Acceptance Policy

Samples arrive at the laboratory in several ways: they may be mailed, shipped by a commercial carrier, brought to the laboratory by client field representatives, or brought to the front desk of the laboratory by the general public. Environmental Toxicology samples may be collected and brought to the lab by WSLH personnel. Each sample received is accompanied by a test request form (TRF). The TRFs, chain of custody forms, and any other documentation received with the sample are retained according to section 1.12. Please see EHD GENOP 033, "Sample Acceptance Policy," which describes the policy used by the Environmental Health Division Receiving Department for accepting samples, into the lab for environmental testing. This SOP also includes links to departmental SOPs that describe specific acceptance and rejection policies for each department.

1.16.2. Horizon LIMS

Samples logged into the LIMS receive a unique sample number as described below. Other attributes of the sample documented in the LIMS at the time of sample log-in are preservatives, container type, client/project name, date/time of receipt at the lab, field ID, collection date/time, tests requested, and sample condition at receipt. Durable labels, with unique workorder/letter identifiers and barcodes, are printed for each sample container and test request form. Two labels are generated for each sample container—one is placed on the body of the container, and another one is placed on the lid of the container if desired for convenience.

Samples are received and noted in the LIMS as being located in the sample receiving department. As samples move through the lab, the location of the sample is changed in the LIMS. Disposal of samples is noted in the LIMS.

1.16.3. Sample ID Generation in Horizon LIMS

Summary

The workorder ID is generated from a database sequencer and is always unique and chronological.

The sample ID is derived from the workorder ID if the sample is part of a workorder, or it is generated from a database sequencer if the sample is an internal QC sample. In both situations the sample ID is unique. The container ID is derived from the sample ID with a letter suffix for each container in the sample, -A, -B, -C, etc. and is always unique. Sub-sample (extracts, aliquots, etc.) IDs are derived from the container ID followed by a number, -1, -2, -3, etc.

Workorder ID Generation

A workorder is a collection of samples that all share various things in common, like customer, due date, collection time, etc. Each workorder is assigned a unique number in the database field PROJECT_SEQ. The system installation setting PSEQASSIGN controls how the PROJECT_SEQ is assigned to the workorder. The laboratory uses the NEXTVAL setting, which assigns each workorder a unique number generated by an Oracle database sequencer and is always unique. Each workorder also has a laboratory identifier in the database field LAB_WO_ID. This ID can be the same as the PROJECT_SEQ or formatted differently, and is controlled by the system installation setting named WO_ASSIGN. The laboratory uses the SYSTEM setting, which sets the LAB_WO_ID equal to the PROJECT_SEQ.

Sample ID Generation

Each sample is assigned a unique number in the database field HSN. The system installation setting HSN_ASSIGN controls how the HSN is assigned to a sample. The laboratory uses the NEXTVAL setting, which assigns each sample a unique number generated by an Oracle database sequencer and is always unique. If a sample is part of a workorder (i.e. not an internal QC sample) the HSN number is derived from the PROJECT_SEQ, using three digits to indicate the number of samples within the workorder (i.e. 001, 002, 003, etc.). This creates a unique number for each sample in a workorder. Each sample also has a laboratory identifier in the database field LAB_SAMPLE_ID. This ID can be the same as the PROJECT_SEQ or formatted differently, and is controlled by the system installation setting named LAB_ASSIGN. The laboratory uses the SYSTEM setting, which sets the LAB_SAMPLE_ID equal to the

HSN.

Container ID Generation

Each container in the LIMS is assigned a unique number in the database field CONTAINER_SEQ. This number is assigned using an Oracle database sequencer, and is always unique. Each container also has a container ID in the database field CONTAINER_ID. The container ID is derived from the LAB_SAMPLE_ID, and is controlled using the system installation setting CONTNAME. The laboratory uses the setting ALPHABETIC, which results in a –A, -B, -C, etc. suffix to the LAB_SAMPLE_ID.

Documentation access

Requisition forms that are submitted with samples are scanned into Horizon, associated with the samples via the workorder and are available for viewing. Instrument data that is transferred into the LIMS via NuGenesis is captured, associated with each sample in the run and available for viewing.

1.16.4. Labeling of sub-samples

After initial sample log-in, all sample aliquots or sub-samples must be labeled or otherwise identified to ensure that there can be no confusion regarding the identity of such samples at any time.

All digestion or other intermediate processing step tubes, containers, or vessels must have an ID number and associated documentation that will give a one-to-one correspondence between this ID number and the unique sample ID number generated by Horizon. Examples include etching an ID number onto a digestion tube, creating a Horizon aliquot label to place on a container, and using a permanent marker to label a drying pan. There would then be a bench sheet, run log, logbook, or work-list that documents the aliquot ID number and the corresponding Horizon number.

All autosampler vessels will be labeled as above or alternately, the autosampler tray will have ID numbers at each position. These ID numbers will be recorded, and a one-to-one correspondence will be established between this number and the unique sample ID number generated by Horizon.

1.17. Instrumentation, Equipment, & Facilities

1.17.1. General

The Environmental Health Division relies heavily on instrumentation. It is imperative that all equipment and instruments are calibrated, verified, operated, and maintained in a proper manner in order to obtain reliable data.

The departmental chapters of this manual contain lists or references to information about support equipment and analytical instruments used in that department. These lists or references include type of instrument, make and model, type of analysis conducted, where the instrument/equipment is located, and assigned instrument numbers or serial numbers. Calibration, maintenance, and verification procedures are found in the appropriate instrument operation manual, instrument operating procedure (IOP), or standard operating procedure (SOP).

If an instrument fails to operate within defined limits or specifications, the problem is identified, corrective action performed, and samples re-run or qualified as required by the method. 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 are covered by maintenance contracts directly with the instrument manufacturer. Preventive maintenance is always recorded in the instrument log (physical logbook or electronic).

If equipment is sent out of the lab, its calibration will be verified when it is returned to the lab. The exception to this is when equipment (e.g. motorized pipets) is sent out for calibration. If the equipment is returned to the lab with a certificate of calibration, this certificate will be reviewed, and if acceptable, the instrument will not need independent verification in the lab.

Maintenance (routine or non-routine) performed by other than SLH staff needs to be

summarized in a document provided to SLH. This document must detail the work performed, which should include as found and as left information. If software updates are required, OIS should be notified to determine if a copy of the computer should be taken prior to the new software being installed.

1.17.2. 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 pre-filter 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 recirculating tank vent filter (replaced annually), and cation, anion and mixed bed tanks that will be replaced as needed. The water is ASTM Type II and is used for the glass washing activities, filling water baths, and as a precursor for other "polished" water throughout the building.

ASTM Type I Polisher

These polishers are located throughout the laboratory and provide ASTM 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 lamp will be changed on a regular schedule. Please see EHD GENOP 032 "Monitoring and Maintaining Water Purification Systems", for specific water purification system monitoring and maintenance procedures.

1.17.3. Ovens, Incubators, Cold Rooms, Refrigerators, Freezers, and Muffle Furnaces

Please see EHD GENOP 013 "Responding to Freezer/Refrigerator/Incubator Failures," which describes the procedures that shall be used at the Agriculture Drive facility when constant-temperature storage equipment (e.g. freezers, refrigerators, incubators) fails. The procedure describes the processes that shall be followed to keep samples within the required temperature range after a failure and the steps necessary to obtain service for the repair of constant-temperature storage equipment.

Each Department maintains a logbook for its temperature sensitive equipment. The

temperatures of the walk-in refrigerators/incubators are monitored continuously, and an alarm will be activated if limits are exceeded. Each department has procedures for monitoring and documenting temperatures according to requirements.

1.17.4. Computers

The WSLH Office of Information Systems (OIS) maintains a number of **quality objectives** to insure the integrity, security and reliability of our systems:

- High-end data center that meets "best practice" standards including climate control, UPS and partial generator backup power, multiphase fire suppression system, physical security safeguards and other safety features (e.g. emergency lighting)
- Separate test and production systems whenever possible, so changes can be tested prior to being implemented in production
- Nightly backups to tape using a grandfather-father-son rotation scheme with weekly offsite rotation (both onsite and offsite tapes are stored in "media proof safes" which are UL-125 rated to not exceed 125 degrees F). All shared drives are flash copied at set intervals during the day to preserve changes made since the last backup (allow more recovery options).
- Multiple, redundant network paths for our servers and workstations including 2 firewalls that establish a secure IPSEC tunnel between our primary buildings. All network traffic between our sites is connected through a fiber-optic ring owned by the "Metropolitan Unified Fiber Network (MUFN) Consortium" <https://mufn.org/> (of which we are a voting member) back to the UW-Madison campus and protected behind firewalls. The UW-Madison is our Internet service provider. Secured VPN service with Remote Desktop Connections (RDC) is used for remote access to enterprise resources.
- Numerous security controls to prevent unauthorized access. Use of HIPAA's "minimum necessary standard" for our clinical systems whereby users are only granted the minimum necessary access they need to do their jobs.
- Inventory and asset management including anti-malware/antivirus, periodic searches for sensitive or restricted data and routine patching for operating system and 3rd party patches (e.g. all servers are patched within 30 days of update release)

Special protections for our Instrument Workstations:

- Instrument workstations typically cannot run the latest operating system patches for fear of breaking the acquisition/analysis software. These are protected by 2 different, segregated VLAN's (virtual networks) with firewall rules:
  - **Instrument VLAN** - contains instrument workstations where we can run antivirus/anti-malware software. These PCs are allowed open access to internal servers but Internet traffic is restricted to only a small number of whitelisted sites (e.g. www.PerkinElmer.com).
  - **Protected VLAN** – contains instrument workstations that cannot run antivirus/anti-malware software or those that meet other high risk criteria. These PCs have very limited access to internal resources and no Internet access.
- When possible, instrument interfaces are established to transfer data into our LIMS systems (e.g. Epic, WindoPath, ChemWare).
- Instrument data (80%) is stored locally and then backed-up to shared drives on the network (see Network/SLHDATAEHD/) using a centralized job scheduler (i.e. the instrument workstation is configured to store its data on the local C:\ drive and then at scheduled times is picked up and copied by one of our servers to a shared drive location on another of servers). OIS staff work with laboratory staff to ensure the correct data is being backed-up to the shared location. Being on a server, the shared drive is again backed-up to tape using our routine process. It is the laboratory's responsibility to ensure any new data storage locations on the local machine are being backed up to our servers.
- We also maintain "bare metal" backups of our instrument workstations using disk imaging software on an annual basis or whenever laboratory staff notify us of a change to the workstation. It is preferred to take a backup image prior to any vendor changes in case these changes need to be rolled-back and then another backup image after verifying the changes work as expected. It is the responsibility of laboratory staff to notify OIS in advance of these changes so the work can be scheduled. These backups are maintained to allow recovery of the PC configuration in case of a catastrophic loss of hardware at the PC level.

- All vendor technicians are required to have their removable media scanned before it can be inserted into the instrument PC to prevent the spread of viruses and malware.

1.17.5. Bulk Argon

The Agriculture Drive facility has a bulk liquid argon tank near the loading dock that supplies argon gas throughout the building for instruments that require it. Our supplier of the bulk argon (purity = 99.999%) is Airgas Merchant Gases, Madison, WI.

1.17.6. Thermometers

Thermometers used for NELAC, WDNR, or EPA testing must be calibrated annually. If thermometers are calibrated in-house, the thermometer will be labeled with the date calibrated and the expiration date (same month of next year-e.g. calibrated 01/12/2017, exp. 01/2018).
If thermometers are purchased new annually with a certificate of calibration, the thermometer will be labeled with the date it was received and the expiration date (same month of next year-e.g. rec'd 05/10/2017, exp. 05/2018).

1.17.7. Facilities

The Wisconsin State Laboratory of Hygiene (WSLH) is currently housed in three separate facilities: 465 Henry Mall on the UW Madison Campus, 2810 Walton Commons, and 2601 Agriculture Drive on the southeast side of the city of Madison. The Agriculture Drive facility includes the original building built in 1999 and a newer co-located building completed in 2013. The analytical departments covered in this manual are housed in the Agriculture Drive facility.

The WSLH is a part of the University of Wisconsin System under the School of Medicine and Public Health. As part of the University, the WSLH benefits from 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 departments are supported by a fully automated glassware washing room, a media preparation room, shipping & receiving department, and the

offices mentioned in section 1.4. Some of these support departments/offices are located at the main Henry Mall facility or at Walton Commons.

The Agriculture Drive facility is equipped with an air handling system that is maintained by the Department of Administration. The system consists of three intake fans and three exhaust fans. The air handling system ensures that there is always negative pressure in all fume hoods and snorkels. Except for the trace metal clean lab (rm. 256), 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. Departmental chapters describe facilities that are used within each department.

1.18. Purchasing

1.18.1. Supplies and Services

The WSLH Purchasing Department purchases goods and services necessary for operations performed by the WSLH except as noted below. Procedures for purchasing services are the same as for purchasing supplies with the addition of a certificate of insurance requirement. To order through the Purchasing Department, submit an E-48 form through the WSLH Intranet Home Page (click on the WSLH Service Desk icon, log into Footprints, and select the Purchasing workspace). For instructions on submitting an E-48, see the link below. The Purchasing Department operates under authority of the UW Madison Purchasing Department and must adhere to University and State of Wisconsin Policies. The internal website purchasing page http://slhicmsprod/administrative-services/wslh-purchasing-department/ contains policies, procedures, and guidelines for purchasing.

In addition to submitting an E-48 to Purchasing, departments have designated purchasing staff that are responsible for ordering supplies for their departments from specified vendors. These orders are placed through the University of Wisconsin's Materials Distribution Services (MDS).

A third purchasing option is the use of a Procurement Card (ProCard), which is a State of Wisconsin VISA credit card. A limited number of employees are authorized to make

purchases using a ProCard, and strict guidelines exist. Please see the internal website under purchasing for policies and procedures.

1.18.2. Capital Equipment

Capital equipment purchases (i.e., > $5000) are generally sent out for bid. Laboratory staff works closely with the Purchasing Department to assure that all necessary specifications are included in the bid. All items are purchased through the Purchasing Department.

1.18.3. Receipt of Supplies

When standards, reagents, reference materials, and media are received, assign them unique traceability codes (see section 1.13), document their receipt in the appropriate logbook, and file the certificates of analysis in the appropriate departmental file (either paper or electronic). Certificates of analyses must be maintained at the lab if the manufacturer has them available (in either hard copy or electronic format). It is the responsibility of the person receiving the supplies to seek out the certificates of analysis if they do not arrive with the items. The certificates must be labeled with the unique traceability code. For original containers of standards, reagents, reference materials, chemicals, media, etc., if an expiration date is provided by the manufacturer or vendor it must be recorded on the container.

1.18.4. Verification and Evaluation of Supplies

It is the responsibility of the individual user of supplies, reagents, and consumable materials to verify that the supplies comply with requirements specified in technical SOPs. An example would be checking blanks on autosampler tubes that come from a vendor. The records of these verification checks would be maintained along with the normal data output from the instrument that is used. Individual technical SOPs also state specific grades of reagents, standards, or chemicals required for the procedure and the reception and storage requirements for reagents, standards, and chemicals.

1.18.5. Approved Vendor Criteria

The criteria for approval of vendors may include:

- Acceptable historical performance as determined by the verification and evaluation procedure noted above or in specific technical SOPs.
- Acceptable reliability and robustness as required by the application are met by

instrumentation and equipment.

- Specific applications may require the vendor to hold certification by third parties.
- Acceptable use by other labs with applications similar to ours.
- Acceptability by UW Purchasing.
- All details of vendor acceptability beyond what is noted here will be in departmental chapters of this QA Manual or in SOPs.

1.18.6. Approved Vendor List

The following list contains vendors commonly used by the departments covered in this QA Manual. Other vendors are used occasionally, and information about them can be found either in the departmental chapters or in individual SOPs.

Vendor	Envir. Tox.	Inorg. Chem.	TECL	Metals	Org. Chem.	Water Micro.	Radio Chem.	Shipping
Accustandard					X			
Acros	X	X			X		X	
Agilent	X			X	X			
Airgas	X	X			X		X	
Amazon	X							
Applied Biosystems						X		
Badger Graphics						X		
Barnstead Thermolyne		X			X	X		
Beckman/Beckman Coulter	X	X				X		
Biorad	X					X		
BioScience		X						
Brinkman	X					X		
Calibrate, Inc.	X						X	
Chem Service					X			
ChemWare	X	X		X	X	X		
Cole Parmer	X	X				X		
Control Company	X	X		X	X	X		
Corning						X		
CPI International						X		
CRM	X					X		
CS Logistics	X							X
DAI Scientific						X		
Dairyland Packaging USA								X

Vendor	Tox	Inorg	TECL	Met	Org	Micro	Rad	Ship
Decon Labs							X	
Dell Computer		X						
Eckert & Ziegler Isotope Products							X	
ELGA/Purelab Ultra	X	X		X	X	X		
EMD Millipore	X	X				X	X	
Entech Instruments					X			
Environmental Express	X	X		X				
Environmental Resource Assoc. (ERA)	X	X			X	X	X	
Eppendorf	X	X		X	X	X		
ESI			X					
EVOQUA (water polisher)	X	X						
Fisher Scientific	X	X		X	X	X	X	
Glass Expansion				X				
Grainger	X	X			X	X	X	X
HACH	X	X			X	X		
Hardy Diagnostics						X		
High Purity				X				
Idexx						X		
Industrial Glassware					X			
Inmark Life Science						X		
InnoCal (Cole-Parmer)		X						
Integrated DNA Technologies						X		
JT Baker	X	X			X		X	
Kimberly Clark		X						
Kimble Kentes		X						
Lachat		X						
Life Technologies	X					X		
MDS Warehouse	X	X			X	X	X	
MERI-Madison Environmental Resourcing Inc.						X		
Mettler-Toledo	X	X		X	X	X		
Microtech	X					X		
Millipore (Millipore Sigma)		X						
MP Biomedicals						X		
National Research Council Canada				X				
NIST	X			X		X		
North Central Labs		X						
Perkin Elmer	X			X	X		X	

Vendor	Tox	Inorg	TECL	Met	Org	Micro	Rad	Ship
Phenova	X					X		
Qiagen	X					X		
Rainin	X	X		X	X	X	X	
Restek					X			
Ricca	X	X				X		
Rice Lake Weighing Systems	X	X			X	X	X	
Sarstedt						X		
SCP				X				
Shimadzu		X						
Sigma-Aldrich	X	X			X	X	X	
Spex Certiprep		X			X			
Staples	X	X		X	X	X		
Steris						X		
Sunset Labs					X			
Supelco	X				X			
Thermo Fisher	X	X		X	X	X	X	
U.S. Postal Service	X	X				X		
Uline		X	X				X	X
Ultra Scientific					X			
Univ. of Arizona-Sterling Parasitology						X		
VHG Labs				X				
VWR	X	X			X	X	X	
Waterborne						X		
Waters	X				X			
Whatman/GE (filters)	X	X				X		
Wild Rose Glass	X				X			

1.19. Management Review of the Quality System

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 and a report will be written. The report will include suitability of our policies and procedures, reports from supervisors, a review of important findings from internal audits, major corrective actions that took place in the past year, a review of any external audit reports, any problems with PT samples that need to be addressed, changes in the volume or type of work performed by the departments, a review of client feedback and complaints, and other relevant factors. For details please see EHD GENOP 023 “Procedure for the Management Review of the

Quality System." A schedule for the Management Reviews can be found at O:\Teams\EHD QC Team\Schedules

1.20. Internal Audits & Data Review

1.20.1. Internal Audits:

Internal audits address all elements of the quality system along with environmental testing activities as related to each specific department. Internal audit findings are summarized in the annual management review document.

Internal audits are conducted annually according to EHD QA 120, "Internal Audit Procedures." The purpose of internal audits is to help meet quality goals such as:

- Ensure that procedures specified in SOPs and regulatory methods are being followed.
- Promote consistent practices across all areas of the Department.
- Ensure that the department is meeting requirements of regulating agencies (e.g. NELAC, USEPA, and Wis. DNR).

Internal Audits will be conducted by the Quality Assurance (QA) Coordinators or designees. They will be done annually or when corrective or preventative actions reveal a need for one. Internal audits consist of two parts: a system internal audit and a group/series of method internal audits. All accredited methods will be audited. Non-accredited methods may also be audited.

- System internal audits are a general review of each department's quality system.
- Method internal audits look at one method or a group of methods in greater detail.

Internal audit reports, including findings/deficiencies, will be generated. Corrective actions in response to the internal audit report will be coordinated by the supervisor and reported back to the QA Coordinator. In some cases an occurrence management report will be filed. If any of the findings of the internal audit casts doubt on the validity of client results, the client will be notified, in writing, within one month of the findings.

A schedule for internal audits can be found at O:\Teams\EHD QC Team\Schedules

1.20.2. Data Review:

Some data calculations are housed within the LIMS and are performed when results are entered. All data is reviewed by a second person before it is released. Review of data is documented in the LIMS with date/time and reviewer's initials. Note that for results calculated by spreadsheets or other software, the software calculations must be verified as giving accurate results before initial use of the software. This verification must be documented and kept on file. Once verified, the calculations must be secured to prevent unauthorized amendment (see EHD GENOP 040, "Protecting Excel and Access Data.") If a calculation cannot be secured (for example, by locking a cell), then some other means of preventing unauthorized amendment must be documented. Software used to record data must have some means of maintaining data integrity and preventing unauthorized amendment of those records. This could include printing out spreadsheets or using a track changes feature to document that data was not changed at a later date. Details on review of data for specific analytical tests can be found at the departmental level.

Quality control samples that are analyzed and entered in the LIMS are given a unique sample number. Those QC samples are related to all samples in the batch in LIMS. The LIMS does an evaluation of the QC sample results based on limits entered into the LIMS and displays pass or fail and the samples related to those QC samples.

1.21. Corrective and Preventive Action

1.21.1. Corrective Action for Non-Conforming Work

Generally, when any aspect of sample testing does not conform to our standard operating procedures (nonconforming work), including quality control procedures, corrective action will be promptly performed, starting with a root cause analysis, and documented. Department-specific details of corrective action documentation within analytical runs can be found in the chapter of this manual dedicated to that department and in individual technical SOPs. Corrective actions will be performed by the analyst in consultation with the peer review auditor and their supervisor. Responsibility for the management and evaluation of non-conforming work rests with the supervisor. The supervisor is responsible for halting work or withholding test reports if deemed necessary. The supervisor will also have the responsibility for authorizing resumption of work if it was halted for corrective action procedures. The supervisor may consult with the client (data user) to determine the usefulness of any qualified data and to determine a subsequent

course of action. In some cases more samples may need to be collected. If a data report has already been released when an error is discovered, the client will be notified as soon as possible and an amended report will be issued. All communications with clients will be documented and archived using e-mail, telephone logbooks, written records, or Occurrence Management Reports (through Footprints software). If the non-conformance of work casts doubt on the laboratory's compliance with its own policies and procedures or compliance with TNI standards, an internal audit will be performed.

1.21.2. Corrective action for departures from documented policies, procedures & quality control

Generally, when departures from documented policies, procedures and quality assurance occur corrective action will be promptly performed and documented. Department-specific details of corrective action documentation can be found in the chapter of this manual dedicated to that department and in individual technical SOPs. Department-specific corrective action procedures identify the person responsible for assessing each QC data type and initiating and recommending corrective action. The treatment of a data set, the reportability of test sample results, and the use of appropriate laboratory-defined data qualifiers is outlined in department-specific methods. Departures from policies and procedures and out-of-control situations and their subsequent corrective actions are documented through the use of occurrence management reports (through Footprints software). If the departures from documented policies, procedures and quality control cast doubt on the laboratory's compliance with its own policies and procedures or compliance with NELAC standards, an internal audit will be performed.

1.21.3. Permitting Departures from Documented Policies and Procedures

Invariably there will be exceptions to the policies and procedures documented and referenced in this QA Manual where corrective action as noted above does not resolve the problem. If an analyst is unsure about how to proceed, he or she should ask his or her supervisor or quality assurance coordinator. Any decision to proceed with work that does not conform to standard procedures must be approved by a supervisor or quality assurance coordinator. The supervisor may consult with the client (data user) or scientific experts to determine the usefulness of any qualified data and to determine a subsequent course of action. Decisions must be documented.

1.21.4. Preventive Action

Preventive action is essential in providing accurate, reproducible, reliable data. Preventive action is necessary to ensure that equipment and all quality systems are functioning properly. Preventive action is done routinely by the laboratory quality assurance staff and the analysts. Preventive action includes such items as:

- Reviewing operational procedures
- Reviewing occurrence management reports for trends
- Reviewing QC data for trends
- Conducting periodic instrument maintenance
- Reviewing instrument logs for problems
- Reviewing customer (internal & external) comments and complaints
- Reviewing PT results
- Reviewing staffing and training needs
- Performing DOCs

If needed improvements or nonconformities arise, actions are taken to ensure that future occurrences are prevented. The use of the Occurrence Management Report (through Footprints software) will allow for the identification and implementation of further preventive actions.

1.21.5. Occurrence Management (OM) Reports

The Wisconsin State Laboratory of Hygiene utilizes Footprints software for tracking Occurrence Management Reports. Guidelines for use of Occurrence Management Reports for documentation of corrective action include when there is a non-routine QC failure that cannot be resolved within the analytical batch or a systemic problem that needs to be tracked over time. Examples of occurrences entered are PT failures, customer complaints, reporting errors, HIPAA violations, etc. Our internal web site includes a link to the Footprints software for Occurrence Management Reports.

There are two lab-wide SOPs that document the use of Occurrence Management forms:
LABWIDE GENOP 706, Occurrence Reporting Procedure
LABWIDE GENOP 707, Occurrence Management System Policy

The procedure for review, monitoring, and close-out of Occurrence Management forms is detailed in LABWIDE GENOP 707. For testing under the quality system as documented in this QA Manual (1.1), open Occurrence Management Reports are discussed at

departmental staff meetings or at the EHD QA team meeting. This allows dissemination of information and encourages progression of corrective actions and follow-up activities.

For occurrences associated with TNI-accredited testing, a root cause analysis must be conducted and documented in the root cause tab on the OM form. Where applicable, follow-up monitoring must be conducted and documented in the OM form. Whenever possible, link all supporting documentation to the OM form in the attachments tab.

Documentation of occurrences must be made by the person discovering the problem, their supervisor, or their QA representative. The documentation must be made as soon as possible after discovery, and at most within a week. Follow-up action taken, monitoring results, and root cause determinations must be documented within the OM form. Documentation of these processes must be completed in a timely manner, and at most within a week of the action taken.

OM training: A training program consisting of three parts has been established. There is a link on the internal website and in appendix 1 of LABWIDE GENOP 706. All employees of EHD must take this on-line training and document the training by filling out and signing the attestation statement for WSLH General Occurrence Management Training (Appendix 1, LABWIDE GENOP 706). The completed attestation statement must be given to the supervisor, who will make a copy for the employee's personnel training file, and send the original to HR. Also see section 1.10.

Any questions regarding the use of the Occurrence Management Reports in Footprints can be directed to any quality assurance coordinator.

1.22. Reporting Analytical Results

Analytical documentation

Reporting of data from the LIMS can be accomplished through either electronic transfer, printed, faxed or emailed version of the result report.

WSLH Laboratory Reports generated by Horizon LIMS for the Environmental Health Division contain:

- Address and contact information for the Agriculture Drive laboratory site and the responsible managers
- A title, unique report ID, and pagination
- Customer name and address

- Sample identification information
- Analytical method used and date of analysis
- Test results along with appropriate units, LODs, LOQs, comments, qualifiers, and additional information as necessary
- Statements indicating compliance with applicable standards, applicability of test results, and instructions for reproducibility of reports.
- Pending tests, if results are reported prior to the completion of all requested analysis

Drinking Water Requirement

If an MCL (Maximum Contaminant Level) for an analyte regulated under ch. NR 809 has been exceeded for a PWS (Public Water Supply) sample, the water supply facility must be notified within 48 hours of completing the analyses (Wis. Admin. Code NR 149.19(7)). The analysis is considered complete when a batch is finalized in Horizon after the peer review audit. Applicable MCLs will be listed in SOPs. Horizon LIMS runs an MCL report daily, the report is sent to HDM staff, and the HDM staff or other designated staff contact the water suppliers who have MCL exceedances.

1.23. Subcontracting of Environmental Tests

If there is an emergency such as an instrument break-down where accredited work would need to be subcontracted, there are several considerations and requirements:

- DNR work certified under NR 149 must only be subcontracted to another lab certified under the same code for the specific field of certification.
- Drinking water compliance samples must only be analyzed by laboratories certified by the EPA or via reciprocity by an authorized state.
- TNI-accredited work must only be subcontracted to another lab that complies with TNI standards for the specific field of accreditation.
- The WSLH must advise the data users of the subcontract arrangement. This must be documented.
- The WSLH must request proof from the subcontractor of compliance with applicable standards (e.g. a certificate and scope of accreditation). These documents must be kept on file.
- The final report must include the name of the laboratory (and Wisconsin Facility

Identification Number—FID, if applicable) performing any subcontracted work. The subcontractor's full report must be made available to the data user if requested.

1.24. Proficiency Testing Sample Procedures

All proficiency testing samples are analyzed in the same manner as used for routine environmental samples, including:

- same staff
- Same methods (ensure method codes match NELAP scope of accreditation, where applicable)
- Same procedures
- Same equipment and facilities

The lab independently analyzes proficiency testing samples and reports the results according to the following rules:

- Not sub-contract the analysis nor analyze another lab's PT sample
- Not communicate with another lab about a PT sample prior to the close of the study
- Not attempt to obtain the PT results from the proficiency testing provider prior to close of the study

Each department is responsible for ordering, analyzing, and reporting PT samples and results that they need to maintain accreditation. See departmental chapters and associated SOPs for details.

Each department is responsible for performing appropriate corrective action for PT failures and documenting the corrective action in Occurrence Management reports.

Each department is responsible for organizing and maintaining PT records including bench sheets, instrument printouts, data calculations, data summary reports, and PT study report forms according to the applicable RDA.

1.25. Method References

1.25.1. Policy

Approved editions of reference methods will be used when required by State of Wisconsin administrative codes or federal rules.

1.26. General References

1.26.1. WSLH Lab-wide Procedures and Policies

Lab-wide policies and references to University of Wisconsin and State of Wisconsin policies and procedures that apply to the WSLH can be found in the WSLH Employee Handbook (see intranet home page). In addition, lab-wide SOPs can be found in the following directory: O:\SOP\Labwide SOP Policies\Final. This directory also contains a table of contents

1.26.2. EHD Division-wide Procedures and Policies

Division-wide procedures and policies can be found at O:\SOP\EHD\Division Wide\Final

1.27. Link to Chapter 2: Inorganic Chemistry, Trace Element Clean Lab, & Metals

O:\SOP\EHD\Division Wide\Final\NELAC QA Manual rev 18 2020\NELAC QA Manual rev 18 2020 2 Inorg Chem & Metals.docx

1.28. Link to Chapter 3: Radiochemistry

O:\SOP\EHD\Division Wide\Final\NELAC QA Manual rev 18 2020\NELAC QA Manual rev 18 2020 3 Radiochem.doc

1.29. Link to Chapter 4: Environmental Toxicology

O:\SOP\EHD\Division Wide\Final\NELAC QA Manual rev 18 2020\NELAC QA Manual rev 18 2020 4 Environ Tox.doc

1.30. Link to Chapter 5: Water Microbiology

O:\SOP\EHD\Division Wide\Final\NELAC QA Manual rev 18 2020\NELAC QA Manual rev 18 2020 5 Water Micro.doc

1.31. Link to Chapter 6: Organic Chemistry

O:\SOP\EHD\Division Wide\Final\NELAC QA Manual rev 18 2020\NELAC QA Manual rev 18 2020 6 Organic Chem.docx

1.32. Link to Chapter 7: Operations

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Operations.doc

1.33. Link to Chapter 8: Shipping

O:\SOP\EHD\Division Wide\Final\NELAC QA Manual rev 18 2020\NELAC QA Manual rev 18 2020 8 Shipping.doc

INORGANIC CHEMISTRY, TRACE ELEMENT CLEAN LAB & METALS

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2.1. Personnel

Table 1: Education and Experience—Inorganic Chemistry

Name	**Title**	**Degree**	**Yrs Exp.**
Graham Anderson	Senior Chemist (Inorg. & Metals Supervisor)	BS Biology / Med. Tech	27
Lindsey Klicko	Assoc. Chemist	BS Water Resources	3
Anthony Plourde	Chemist Senior	BS Chemistry	22
Royce Riessen	Chemist	BA Chemistry	10
Mason Shields	Assoc. Chemist	BS Chemistry	6
Jennifer Thorngate	Chemist Senior	BA Biological Sciences	14

Table 2: Education and Experience—Trace Element Clean Lab

Name	**Title**	**Degree**	**Yrs Exp.**
Christa Dahman Zaborske	Senior Chemist (TECL Supervisor)	BS Chemistry	8
Joel Overdier	Senior Research Specialist	HS + 4 yrs University (Chemistry)	30
Sean Scott	Post-Doc Research Assoc.	Ph.D. Geology	13
Martin Shafer	Associate Scientist	Ph.D. Water Chemistry	32
Pam Skaar	Chemist Senior	BS Biochemistry, MS Fisheries & Wildlife Ecology	36
Nic Slater	Senior Chemist	MS Geology	7
Kirsten Widmayer	Chemist	BS Environmental Science	3
Na Zhang	Chemist Advanced	Ph.D. Chemistry	8

Table 3: Education and Experience—Metals

Name	**Title**	**Degree**	**Yrs Exp.**
Brian Clary	Chemist Senior	BS Mech Engineering	14
Kevin Kaufman	Chemist Advanced	HS + 4 yrs University (Chemistry)	30
R.J. Messling	Chemist Senior	BS Biology	17
Roger Schulz	Chemist Advanced	BS Biology	33

Table 4: Student Employees

Name	**Dept.**
Haley Westerfield	TECL

2.2. Training

Please see section 1.10 of this QA manual for training requirements common to all EHD employees. Training specific to Inorganic Chemistry, Trace Element Clean Lab (TECL), and Metals includes:

- Inorganic Chemistry/Metals New Employee Training Checklist: O:\SOP\EHD\ESS\Inorganic\Final\New Employee Train Check Rev5.doc
- Inorganic Chemistry/Metals Analyst Method Training form: O:\SOP\EHD\ESS\Inorganic\Final\Analyst SOP Training Form ver 5.doc
- Certification Statement for Current Methods: O:\SOP\EHD\ESS\Inorganic\Final\Analyst Certification Statement, rev. 2.doc
- Initial Demonstration of Capability (DOC) form with calculating cells: O:\SOP\EHD\Division Wide\Final\EHD QA 115_DOC CALC FORM_rev 5.xls
- Continued proficiency for technical methods: see requirements in O:\SOP\EHD\Division Wide\Final\EHD QA 115 rev 0_DOCs.docx

Training requirements for both new and continuing employees of the Inorganic Chemistry and Metals departments can be found in ESS INO GENOP 112, "Personnel Training Files." This SOP also contains links to documents that are required reading and links to checklists and sign-off sheets.

2.3. Document Control System

Please see section 1.11 of this QA Manual for information about location and control of lab-wide and division level SOPs.

For the Inorganic Chemistry, Trace Element Clean Lab (TECL), and Metals departments, all "official" and current SOPs, related forms, and documents are kept in electronic format on O:\SOP\EHD\ESS\Inorganic\Final or on O:\SOP\EHD\EHD Metals\Final and will be available to all staff members as "read-only" documents. Printed hard-copy versions of department SOPs exist in binders at the QA Coordinator's desk and may exist at laboratory work stations. Copies will be made available to customers or to regulatory agencies if requested. Please see section 17 of this chapter for a link to a table of contents for department SOPs.

All SOPs associated with drinking water testing (including administrative as well as technical) must be reviewed for content annually and updated if necessary. SOPs under other accreditations will be reviewed at least every three years. SOPs for non-accredited tests will be reviewed at least every four years. SOPs for methods that are available upon special request do not have a periodic review requirement.

SOPs are revised when necessary due to instrument/technology changes, regulatory changes, etc. Each new revision will have a new revision number, an effective date, and will list the SOP it is replacing. Any archived documents (retained according to applicable records disposition authorizations, RDAs) 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. For detailed information regarding writing and managing SOPs, please see ESS INO GENOP 001, "How to Write an SOP" And EHD METALS GENOP 1001, "Writing and Managing SOPs."

External reference documents such as sources of reference methods, instrument instruction manuals, and software versions are controlled via the Related Documents/References sections of SOPs. If new versions of external references become available and are implemented, a new SOP revision would be started and the new external reference would be listed in the Related Documents/References section. An additional means of controlling and tracking external documents is a table of contents for external documents, which is located in O:\SOP\EHD\Division Wide\Draft\In Progress\TOC External Documents.xlsx.

2.4. Records Retention, Control, and Storage

Please see section 1.12 of this QA Manual for RDA information, laboratory notebook information, and information about storage of records in the basement and at the State Records Center.

Records information specific to the Inorganic Chemistry, Trace Element Clean Lab, and Metals departments can be found in ESS INO GENOP 650, "How to Store Inorganic Record Storage Boxes in the Basement."

A listing of the record boxes stored is available on the shared EHD server: R:\EHD\ESS(4900)\ESS Inorg(4910)\Forms\Record Storage Boxes.xlsx

2.5. Sample Handling Procedures

Please see section 1.16 of this QA Manual for sample acceptance information on the divisional level. Also included is sample login and tracking information and sample ID generation in Horizon LIMS.

A spreadsheet was developed that lists required preservatives, minimum volumes, hold times, and acceptable bottle types for common Inorganic and Metals water tests: O:\SOP\EHD\ESS\Inorganic\Final\Bottle Types_Tests_Preserv_Vol_Inorg4910 one page.xlsx. More specific information can be found in the "Sample Handling and Preservation" sections of analytical SOPs.

Inorganic Chemistry/Metals sample disposal procedures, including documentation in Horizon as well as physical disposal techniques, are detailed in ESS INO GENOP 110, "Sample Disposal Protocol."

Special handling for enforcement samples:

Inorganic and Metals chemists verify the pH of enforcement samples upon receipt in the laboratory testing areas.

Chemists use Horizon to "track" enforcement samples in and out of the locked 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 coldroom. The analyst has

responsibility for law enforcement samples during times that the samples are out of the coldroom for analysis.

Dilution Verifications:

If a sample result is above the calibration range, the sample must be diluted until the concentration is within the calibration range (between the low standard/LOQ and the high standard). When a dilution is made, the dilution must be verified by comparing the diluted result with the original result. The comparison is done by dividing the diluted result by the original result and expressing the calculation as a percent. The acceptable range is 90% to 110%. If the dilution verification is not within the acceptable range, a different dilution must be made. This second dilution would then be verified against the first dilution. If a group of samples is diluted identically, at least 10% of the dilutions must be verified.

2.6. Data Review and Reporting

Please see section 1.22 of this QA Manual for division wide information on reporting analytical results.

When the analyses are completed, the results are audited by a chemist familiar with the analysis. The data packet is reviewed along with the batch data in Horizon. In addition, the QC parameters and any required comments or qualifiers for the batch are reviewed in Horizon. Dilution calculations and documentation also must be reviewed.

If all analytical requirements are met, the auditor completes the batch review schedule, which changes the batch status to final. For details on the data review procedure see ESS INO GENOP 113, "Horizon Procedures for EHD Inorganic Chemistry" and EHD METALS GENOP 105, "Horizon Procedures for EHD Metals."

Reporting is done through Horizon LIMS. Results are sent to appropriate parties either electronically or by mail as requested. More details are available through the Horizon Data Management department.

Drinking Water Requirement
If an MCL (Maximum Contaminant Level) for an analyte regulated under ch. NR 809 has been exceeded for a PWS (Public Water Supply) sample, the water supply facility must be notified within 48 hours of completing the analyses (NR 149.19(7)). The analysis is considered complete when a batch is finalized in Horizon after the peer review audit. Applicable MCLs will be listed in SOPs. Horizon LIMS runs an MCL report daily, the report is sent to HDM staff, and the HDM staff contact the water suppliers who have MCL exceedances. Documentation of the contact is made in Horizon. The following MCLs apply to Inorganic Chemistry and Metals testing:

Contaminant	**MCL (units)**	**SOP**
Sb	6 µg/L	EHD METALS METHOD 400.4
As	10 µg/L	EHD METALS METHOD 400.4
Ba	2000 µg/L	EHD METALS METHOD 400.2
Be	4 µg/L	EHD METALS METHOD 400.2
Cd	5 µg/L	EHD METALS METHOD 400.4

Cr	100 µg/L	EHD METALS METHOD 400.4
Cyanide (as free)	0.2 mg/L	ESS INO METHOD 180.1
Fluoride	4.0 mg/L	ESS INO METHOD 220.9
Hg	2 µg/L	EHD METALS METHOD 540.3
Ni	100 µg/L	EHD METALS METHOD 400.2
Nitrate (as N)	10 mg/L	ESS INO METHOD 220.9
Nitrite (as N)	1 mg/L	ESS INO METHOD 220.8
Tot. Nitrate + Nitrite (as N)	10 mg/L	ESS INO METHOD 220.9
Se	50 µg/L	EHD METALS METHOD 400.4
Tl	2 µg/L	EHD METALS METHOD 400.6
Cu	1300 µg/L	EHD METALS METHOD 400.2
Pb	15 µg/L	EHD METALS METHOD 400.4

2.7. Procedures for Accepting New Work/Review of Requests, Tenders and Contracts

Please see section 1.15 of this QA Manual for division wide information and policies for accepting new work based on authority granted by the WSLH Board. This section also includes considerations for reviewing laboratory capability and capacity to cover the requested scope of work. Also, be aware that the WSLH has procedures about who can sign contracts and what terms and conditions can be agreed upon (contact the Office of Finance for details). Discussions with clients regarding handling, testing, or disposition of samples must be documented. Also see section 1.6 of this QA Manual for division wide information regarding customer communications and services. Inorganic Chemistry and Metals departments have no department-specific information to add.

2.8. Laboratory Facilities

The Inorganic Chemistry, Metals, and Trace Element Clean Lab departments are composed of three main areas: Metals (room 117/118), Wet Chemistry (room 119), and the Trace Element Clean Lab (TECL) (256 suite, co-lab building). All laboratory areas are separated from office cubicles by doors and hallways.

2.8.1. Metals

The Metals department is housed in three rooms. Facilities and instrumentation are shared with the Wisconsin Occupational Health Lab (WOHL) and other EHD departments that hold accreditations not covered by this QA Manual.

- Room 117 (1280 sq. ft.) is the Metals' instrument room. It contains four ICPs, two FIMS for mercury analysis, two graphite furnaces (GFAAS), a cyanide distillation system, a mercury vapor analyzer (NADP), a fume hood, a HEPA-filtered vertical laminar air flow fume hood, a small vertical laminar air flow fume hood, and two refrigerator/freezers.
- Room 117E (200 sq. ft.) is a balance room and a chemical storage room. It also contains a water polisher.

- Room 118 (600 sq. ft.) is the prep room for metals tests. It contains six hoods used for metals digestions and TCLP extractions.

2.8.2. Wet Chemistry

The Wet Chemistry area is a large general laboratory space with several lab benches. It is used for general chemistry tests like BOD, solids, spectrophotometer tests, and others. It also contains several Lachat flow injection colorimetric instruments for the various nutrients tests.

- Room 119 (2920 sq. ft.) contains two water polishers, four ovens, one muffle furnace, three incubators, one refrigerator, one freezer, and four fume hoods. This area also contains the Inorganic sample check-in area and the wash room pick-up and delivery area. This room is shared with the Communicable Disease Division and with the National Atmospheric Deposition Program.
- Room 119C (170 sq. ft.) is a locked sample storage cold room maintained at 4°C ± 2°C.

2.8.3. Trace Element Clean Lab (TECL)

The TECL suite (3,000 sq. ft.) in the co-located lab building is used for ultra-low level elemental analysis. It was designed to minimize metal components and it is supplied with HEPA filtered air. Routine monitoring of air-borne particle counts is done.

- The TECL suite is entered through an ante room (class 100,000). The ante room is a place to put on Tyvek lab coats and shoe covers, which are required for the other rooms of the TECL suite. The ante room also contains a refrigerator/freezer and general storage.
- Room 256A (class 10,000) is a balance room. It has tight temperature and humidity control.
- Room 256B (class 10,000) is a bottle cleaning room. It has two fume hoods with hot acid baths, one HEPA-filtered drying bench, and one HEPA-filtered vertical laminar air flow fume hood, and two water polishers.
- Room 256C (class 10,000) is the instrument room. It contains four ICP-MS instruments, two microwave digestion ovens, one conventional oven, and one water polisher. It also houses two HEPA-filtered vertical laminar air flow fume hoods.
- Room 256D (class 10,000) is the mercury analysis room. It contains two atomic fluorescence spectrophotometers for ultra-low level Hg analysis (both total and methyl) and one atomic absorbance spectrometer fed by thermal decomposition for direct total mercury analysis. There is also a water polisher, two hot blocks, and a fume hood.
- Room 256E (class 1,000) is an ultra-clean prep room. It contains a water polisher and a HEPA-filtered vertical laminar air flow fume hood.

2.9. Instrumentation and Equipment

Major analytical instrumentation operated by the Inorganic Chemistry, TECL, and Metals departments is listed in a spreadsheet: O:\SOP\EHD\ESS\Inorganic\Draft\Instrument number listing.xls

The instrument spreadsheet lists instrument make/model, serial number, type of instrument, analyses done with the instrument, location of instrument, assigned department instrument number, and dates in use. The assigned department instrument number in the spreadsheet is also placed on the instrument via a label. In Horizon, these same instrument numbers are used along with an IC prefix for instruments in rooms 119 and 256 (the TECL suite), and a MET prefix for instruments in rooms 117 and 118. These measures maintain traceability of instrumentation through analysis.

Details of instrument calibration, maintenance, and verification are found in individual technical SOPs (please refer to section 17 of this chapter for SOPs).

General practice is to have a logbook (physical or electronic) for each instrument where details of daily use, periodic maintenance, and repairs are recorded.

Air displacement pipettes used for the measurement of liquids in the laboratory must be tested on a regular basis to insure volumes delivered are accurate and precise. ESS INO GENOP 200, "Pipette Performance Checks" describes the means of monitoring the performance of air displacement pipettes. Pipettes must be labeled with the date calibrated and the date the calibration expires (first month of next quarter). This also applies to repipettes.

A list of pipettes for ESS Inorganics, the TECL, and EHD Metals is found at:
O:\SOP\EHD\ESS\Inorganic\Draft\Inorg pipettes.xls

Class A glassware may be used for quantitative measurement without further verification. Other glassware used for quantitative measurement must be verified or otherwise meet accuracy requirements of the analytical method (e.g. wide bore pipets are specified in the BOD and Solids reference methods). All verifications must be documented and saved on file.

New thermometers along with NIST-traceable certificates of calibration are ordered annually. When a new thermometer is received, it must be entered in M:\EHD\ESS(4900)\ESS Inorg(4910)\METALS\Controlled documents\thermometers and given a code. Thermometers and certificates of calibration are labeled with the code, date received, and expiration date (one year after month received).

The thermometer certificates are checked to ensure that the calibration was done over the range of use of the thermometer (usually, 0°C-100°C). For the thermometer that is used in our 180° oven, we will do an additional verification at 180°C-200°C against our NIST-traceable high temperature thermocouple.

In addition, we will ensure that the expiration dates on the thermometer certificates are at least a year from the date the thermometer was received. The certificates are kept in a binder on the bookshelf inside the southern door to room 119.

Our high temperature thermocouple (Oakton Thermocouple SN 4005714) is sent out for NIST-traceable calibration annually. It is labeled the same as the other thermometers, and the calibration report is filed in the same binder.

2.10. Laboratory Supplies and Chemicals

Please see section 1.18 of this QA Manual for purchasing procedures, verification and evaluation of supplies and chemicals, approved vendor criteria, and a general list of approved vendors.

2.10.1.Chemical Inventory

A chemical inventory for Inorganic Chemistry and Metals is located in a spreadsheet at: O:\SOP\EHD\ESS\Inorganic\Final\Chemical Inventory & SDS 2019.xlsx

A chemical inventory for the Trace Element Clean Lab is located: M:\EHD\ESS(4900)\ESS Inorg(4910)\METALS\Clean Room\New Clean Lab (Rm 256)\256 Chemicals SRMs on hand.xlsx

The chemical inventories linked above list the chemical name, CAS number, supplier, catalog number, approximate quantity, where the chemical is stored, extreme hazard notes, and links to the Safety Data Sheets (SDS) for each chemical. Any special storage requirements for chemicals are noted in the individual technical SOPs for which the chemicals are used. All chemicals are kept in containers and at temperatures that will not alter their integrity. The spreadsheets have a separate tab with lists of approved suppliers and the basis for approval (historically acceptable analytical results including QC parameters like LRB, QCS, IPC, LFB, and LOD).

2.10.2.Safety Data Sheets

A Safety Data Sheet (SDS) is a document containing chemical hazard and safe handling information. There are 16 sections including: Section 2: Hazard identification, Section 4: First-aid measures, Section 7: Handling and storage. For more information see O:\SOP\EHD\ESS\Inorganic\Final\Safety Data Sheets 2019\001 SDS Info for Chemists.doc. This information sheet is posted in the weigh room (117E).

The SDS pdf files linked through the chemical inventory spreadsheets are located in: O:\SOP\EHD\ESS\Inorganic\Final\Safety Data Sheets 2019

M:\EHD\ESS(4900)\ESS Inorg(4910)\METALS\Clean Room\MSDS_jo

2.10.3.Collection Bottles & Preservation Vials

Sample collection bottles are ordered from various manufacturers. Bottles are randomly selected from cases of a specific lot to check for contamination. Bottles are not used for the collection of samples until the bottle check tests show that contamination level criteria are met for the parameters of interest. For details on this procedure please see ESS INO QA 101, “Bottle Check Procedure.”

Acid (both sulfuric and nitric) vials are ordered from Fisher-EP Scientific Products. Vials are randomly selected from shipments to check for contamination. Vials from a shipment are not used to preserve samples until the vial check tests show that contamination level criteria are met for the parameters of interest. For details on this procedure please see ESS INO QA 102, “Acid Vial Check Procedure.”

2.11. General Quality Control Procedures

2.11.1. Quality Control Data Gathering and Documentation

Required QC is listed in each technical SOP. Generally, required QC samples include lab reagent blanks (LRB), calibration blanks (CB), quality control samples (QCS), instrument performance checks (IPC), lab fortified blanks (LFB), lab duplicates (LD), and matrix spikes (MS). See the following table for definitions and other names and abbreviations that may be used for the same QC sample. Horizon sometimes uses different terms than our SOPs do. Also, reference method sources (e.g. Standard Methods, EPA, ASTM) use various terms to mean the same thing.

Quality control samples that are analyzed and entered in the LIMS are given a unique sample number. Those QC samples are related to all samples in the batch in LIMS. The LIMS does an evaluation of the QC sample results based on limits entered into the LIMS, and it displays pass or fail.

2.11.2.QC Sample Designations Table

Abbrev.	Full Name	Definition	other designations
LRB	Lab Reagent Blank	blank matrix taken through all sample prep steps	reagent blank (RB), digested blank (DB), method blank (MB)
CB	Calibration Blank	Reagent water fortified with the same matrix as the calibration standards.	Initial calibration blank (ICB), continuing calibration Blank (CCB)
QCS	Quality Control Sample	A solution of method analytes of known concentrations (different concentration than the IPC) that is obtained from a second source.	Initial calibration Verification (ICV)
DQCS	Digested Quality Control Sample	A digested QCS	QCS
IPC	Instrument Performance Check	A solution of method analytes used to evaluate instrument performance. Prepared from same stock as calib. Standards.	Continuing calibration Verification (CCV)
I-IPC	Initial Instrument Performance Check	same as IPC, but run immediately after calibration (sometimes has different limits)	initial check (ICHECK)
LFB	Lab Fortified Blank	known quantities of method analytes spiked into blank matrix	digested spiked blank (DS), ongoing precision and recovery (OPR)
LD	Laboratory Duplicate	second aliquot of a sample analyzed same as first aliquot	duplicate (D, DUP)
MS	Matrix Spike	known quantities of method analytes spiked into a sample aliquot	spike (S, SP); lab fortified sample matrix (LFM)
MSD	Matrix Spike Duplicate	a second aliquot of a matrix spike	spike duplicate (MS)

- 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.

2.11.3.Corrective and Preventive Action Procedures

Please see section 1.21 of this QA manual for additional information on corrective and preventive action.

Corrective action must be taken after any QC failure and any time an analyst notices an instrument problem or analytical problem. Procedures for taking corrective action in the laboratory are often instrument specific. Please see individual technical SOPs for corrective action information related to common problems for specific procedures. Corrective action may include repairing, adjusting, or servicing instrument hardware or software; changing sample handling or treatment; or correcting analyst errors. Corrective action may be performed by the analyst or may require an outside service call. If outside service is required the Department Supervisor will be consulted.

When a specific problem has been identified and corrective action completed, any samples that may have been affected by the problem need to be addressed by a subsequent action. Possible subsequent actions include: qualifying results, reanalyzing (and/or re-digesting) a single sample, reanalyzing an entire QC group, reanalyzing all samples from the last acceptable QC sample, and re-standardizing and reanalyzing all samples within the analytical run.

Results are qualified by adding a comment in Horizon. The comment is added by going to the Edit Results screen from either the Batch Edit screen or the Sample Edit screen. In the Task Details section in the ANALYTES grid, locate the Comments column and click on the blue link next to the corresponding parameter that requires a comment that will say “X comments” where “X” is the number of comments that have been added thus far. The Edit Comments box will open. Click Add Comment. At this point a pre-defined comment can be entered by selecting the Predefined options and then searching in the dropdown menu next to it or a free text comment may be entered in the box. Click Save when finished.

The corrective action must be documented either within the analytical run, in the appropriate instrument logbook, or in an Occurrence Management Report, which is accessed through the internal website. Corrective action documentation will include: a) identifying the QC failure and its root cause (if known); b) noting specific corrective actions that were taken or attempted; c) verifying that the corrective action worked, and stating any further action that will be taken. Attached to each analytical run will be a cover sheet (see section 2.15.4 of this chapter), which includes lists of specific analytical items that will be checked routinely in the event of a QC failure. These lists, which are tailored to the specific method and instrumentation, will aid in the corrective action documentation. If the analyst cannot pinpoint a specific problem, they will note, “Analytical Checks OK—Unknown cause” or “✓OK” on the bench sheet.

Major preventive actions taken by the Inorganic Chemistry and Metals departments include monitoring routine equipment performance and maintenance, monitoring QC sample performance, reviewing analyst DOCs, and verifying LODs. Improvement opportunities are identified through

staff meetings, client requests or questions, internal audits, and management system reviews.

2.11.4.Departures from Documented Policies and Procedures

Please see section 1.21.3 of the general chapter of this QA Manual for information about permitting departures from documented policies and procedures, who can approve departures, and how to document them.

2.11.5.LOD & LOQ Procedures

LODs (Limits of Detection) must be determined for most tests that are performed by the Inorganic Chemistry, TECL, and Metals departments. LODs are sometimes referred to as MDLs (Method Detection Limits). LODs are method, matrix, and analyte specific values that show at what level a measurement can be distinguished from a blank. LODs are mathematically related to LOQs (Limits of Quantification). For more information on procedures for determining LODs and LOQs please see the following SOP: EHD QA 116, "LOD/LOQ Procedures."

Please see the Inorganic Chemistry LOD schedule at: M:\EHD\ESS(4900)\ESS Inorg(4910)\LODs\Inorg Chem LOD Schedule.xls

The Metals LOD schedule is located: M:\EHD\ESS(4900)\ESS Inorg(4910)\METALS\LOD and RL data\001 METALS LOD Schedule.xls

2.12. Quality Control Limits Procedures

Quality control (QC) limits define the precision and accuracy of all reported data. Limits are established and created in Horizon for each parameter, matrix, and QC type (e.g. blanks, performance samples, duplicates, and spikes). Limits are contained in Horizon. If a QC result exceeds control limits, corrective action must be taken and documented. Affected data must either be re-analyzed or qualified.

2.12.1.Precision Control Limits

Precision is defined as the measure of mutual agreement among individual measurements (US EPA, 2005). Precision limits are based upon relative percent difference calculations.

A normalized method for specifying precision is the relative percent difference (RPD) expressed as follows:

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

Where:

A, B = the two analysis results (duplicates)

The upper control limit defines the acceptable relative percent difference for duplicated samples. The control limit is calculated using the following equation:

$$CL = \overline{RPD} + 3S$$

Where: $\overline{RPD}$= the mean relative percent difference for a series of duplicated samples analyzed over a given period of time.

S = the standard deviation of the relative percent difference for a series of duplicated samples analyzed over a given period.

2.12.2.Accuracy Control Limits

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy control limits are based upon the mean and standard deviation of the percent recoveries of 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., reagent water), or a laboratory matrix (i.e., Ottawa sand) which may be used for calibration check verifications, laboratory control samples, outside source check standards, etc. The percent recovery (% Rec) is calculated using the formula:

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

Where:

A = the observed concentration of the spiked sample
B = the background concentration of the sample
C = the known concentration of the spike

The control limits (upper and lower) define the acceptable percent recovery for spiked samples. The control limits are calculated using the following equation:

$$CL = \overline{\%Rec} \pm 3S$$

Where:

$\overline{\%Rec}$ = the mean percent recovery for a series of spiked samples analyzed over a given period of time.

S = the standard deviation of the percent recovery for a series of spiked samples analyzed over a given period.

2.12.3.Limit Creation, Evaluation & Modification

Limits are reviewed and evaluated annually. Modifications are made as required. For details on quality control limit creation, evaluation, and modification please refer to: EHD QA 113, which describes procedures for Horizon LIMS.

2.13. Internal and External PT Studies

2.13.1.PT Programs Schedule

Opening Month	PT Program	PT Samples to Order	Source
Jan.	Internal Blinds	Entire suite	ERA
Feb.	WP	Minerals Hardness pH Volatile Solids Simple Nutrients Complex Nutrients Nitrite Demand Trace Metals Hg Tin & Titanium Cyanide Oil & Grease	ERA
	WS	Hardness Inorganics pH Metals Turbidity Hg Nitrite o-Phosphate Nutrients Cyanide	ERA
	Standard Reference Sample round robin	Majors, Nuts, LL Nuts	USGS
April	Internal Blinds	Entire suite	ERA
	SOIL	Trace Metals (offered quarterly)	ERA
	WS	Si (offered quarterly)	ERA
	WP	Si & LL Hg (offered quarterly)	ERA
	Sediment Lab Quality Assurance	Suspended Sediment	USGS

July	Internal Blinds	Entire suite	ERA
Aug.	WP	Minerals Hardness pH Volatile Solids Simple Nutrients Complex Nutrients Nitrite Demand Trace Metals Hg Tin & Titanium	ERA
	WS	Hardness PT Inorganics PT pH PT Metals PT Turbidity PT	ERA
	Standard Reference Sample round robin	Majors, Nuts, LL Nuts	USGS
Oct.	Internal Blinds	Entire suite	ERA
	SOIL suite	Metals (offered quarterly)	ERA
	WP	LL Hg (offered quarterly)	ERA
	Sediment Lab Quality Assurance	Suspended Sediment	USGS

2.13.2.PT Program Notes

Please see ESS INO QA 109, "PT Sample Procedures" for details about ordering, checking in, tracking, reporting, and following up with PT samples.

Each "suite" mentioned in the table above includes several PT samples. Each PT sample includes several PT analytes. Each analyte may be analyzed by one or more methods. Our New Hampshire TNI scope of accreditation lists method codes. PT results for TNI accreditation must be reported out by these method codes.

To make ordering the PT samples more efficient, for each suite there is a master order spreadsheet listing all the PT samples that must be ordered. The spreadsheets may be edited and attached to electronic Footprints orders. The spreadsheets are located at: R:\EHD\ESS(4900)\ESS Inorg(4910)\Admin\QC issues\PTs and Blinds\Order Spreadsheets\Master Order Spreadsheets

There are also master test request forms for each PT sample located at R:\EHD\ESS(4900)\ESS Inorg(4910)\Forms\PT Sample Forms

PT study result reports are saved in separate folders for each PT program at: R:\EHD\ESS(4900)\ESS Inorg(4910)\Admin\QC issues\PTs and Blinds. If analysis results of PT samples fall outside the acceptable range, the QA Coordinator will complete an Occurrence Management Report (with a copy sent to the QA Committee co-leader). A make-up sample may be analyzed in order to maintain accreditation status.

There is a quick-glance PT result history with separate spreadsheet tabs for each PT program at: R:\EHD\ESS(4900)\ESS Inorg(4910)\Admin\QC issues\PTs and Blinds\PT SAMPLE HISTORY.xls

2.14. Internal Audits

Internal audits for the Inorganic Chemistry, Trace Element Clean Lab, and Metals departments are conducted according to EHD QA 120 (see section 1.20 of this QA Manual).

Inorganic Chemistry, Trace Element Clean Lab, and Metals internal audit reports are located in: R:\EHD\ESS(4900)\ESS Inorg(4910)\Admin\Internal Audit Reports\Final

A schedule for internal audits is located in: O:\Teams\EHD QC Team\Schedules

2.15. Traceability of Measurement

Please see section 1.13 of this QA Manual for basic information about traceability.

Standards and reagents of required purity are obtained from approved suppliers (see section 10 of this chapter). When available these chemicals 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 code number (see below) that will be marked on the bottle. This certificate is then kept on file. All standard and reagent bottles are dated when received to monitor the shelf life. All chemicals will be replaced before exceeding their expected shelf life.

2.15.1. Standards

Stock standard solutions are recorded in the proper stock standard logbook, which may be paper, electronic, and/or Horizon LIMS, and are assigned a unique standard code number (e.g. a combination of the logbook page number and the line number). When a working standard is prepared, the compound(s), standard code number, date prepared, analyst, expiration date, and solvent are noted in the working standard logbook, which may be paper, electronic, and/or Horizon LIMS. All containers are clearly labeled with compound names, concentrations, unique standard code number, and expiration date. The stability of all solutions is carefully monitored. They are re-standardized and/or prepared at a frequency determined by the appropriate analytical method.

2.15.2. Reagents

The Inorganic Chemistry Department has a stock reagent logbook (Book # 1 and continuing in R2) located in the dry chemical storage area/weigh room. Each chemical (including pH paper and other chemical test strips) received by the lab is assigned a unique reagent code number (a combination of

the logbook page number and the line 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. If no expiration date is provided by the manufacturer, it is not required. The Inorganic Chemistry Department also has working reagent logbooks, which may be paper, electronic, and/or Horizon LIMS. 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.

2.15.3. Horizon Standards Log

The Horizon Standards log may be accessed by opening Horizon, clicking on Data and then Standards. When a working standard is prepared, standard code number, date prepared, analyst, expiration date, analyte, and concentration are noted in the Standards Log within Horizon. The code number and all relevant information provided in the Horizon Standards Log will be the same information provided in the Standards Logbook in the lab. All standards must be up to date within Horizon in order to finalize test results. If not up to date, an error message will occur preventing the results from being finalized.

2.15.4. Bench Record Cover Sheets

A bench record cover sheet is attached to most instrumental data packets. The cover sheets aid in traceability and corrective action documentation and are specific to each method. The cover sheets generally contain items such as method identifying numbers (SOP and reference method), analysis identifying information (batch number), analysis date, analyst name, standard and reagent traceability codes, instrument numbers, corrective action documentation information, and analysis notes. Critical environmental conditions (temperature, barometric pressure, etc.) may be recorded on the bench record cover sheet or elsewhere in the data packet.

2.16. Method References

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), Clean Water Act (CWA) and other programs regulated by the Wisconsin Department of Natural Resources Laboratory Certification Program, 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.

Analytical methods used by the Wisconsin State Laboratory of Hygiene Inorganic Chemistry, TECL, and Metals departments are documented in the individual technical SOPs. These SOPs state specifically how the lab carries out and implements approved methods. The majority of approved methods used by Inorganic Chemistry are from either the EPA or Standard Methods for the Examination of Water and Wastewater. The specific references to the approved methods can be found in the reference section of the individual SOPs. Following is a list of sources for approved reference methods:

- American Public Health Association, American Water Works Association, Water Environment Federation, Standard Methods for the Examination of Water and Wastewater, 21st edition, 2005 (and earlier editions).
- United States Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, 1979, 1982, 1997.
- Welschmeyer, Fluorometric analysis of chlorophyll a in the presence of chlorophyll b and pheopigments, Lmnol. Oceanogr. 39(8), pp. 1985-1992, 1994.
- United States Environmental Protection Agency, Office of Solid Waste and Emergency Response, Test Methods for Evaluating Solid Waste, 3rd edition, SW-846, 1996.
- United States Environmental Protection Agency, Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, 1993.
- QuikChem methods for flow injection analysis, Lachat Instruments, Loveland, CO.
- American Society of Testing and Materials, Annual book of ASTM Standards, Section 11, Water and Environmental Technology, vol. 11.02. 1993.

2.17. Standard Operating Procedures

A complete list of standard operating procedures can be found in the tables of contents located at:

- Inorganic Chemistry (including TECL) (click on tab for Inorganic Chem): O:\SOP\EHD\Division Wide\Final\001 TOC Internal Documents.xlsx
- Metals (click on tab for Metals): O:\SOP\EHD\Division Wide\Final\001 TOC Internal Documents.xlsx

The tables of contents include unique SOP identifiers, titles, revision numbers, effective dates, review dates, and hyperlinks to the actual documents. The tables of contents are in spreadsheet format and can be sorted and filtered via the Excel data tab along the top of the page.

2.18. Accredited Methods (NELAC, EPA, DNR)

2.18.1.Drinking Water

Regulatory Method	**Edition**	**TNI Code**	**Description**	**WSLH Method #**	**Accreditation**		
					NELAC	EPA	WDNR
SM 4500-H+ B	-2000	20105219	pH, Electrometric	INO 300.0	X		
EPA 200.7	4.4 (1994)	10013806	Inductively Coupled Plasma-Emission Spectrometry	MET 400.2	X	X	X
EPA 200.9	2.2 (1994)	10015404	Atomic Absorption Spectroscopy--Tl	MET 400.6		X	X
SM 3113 B	-2004	20058837	Atomic Absorption Spectroscopy (GFAA)	MET 400.4		X	X
EPA 245.1	3 (1994)	10036609	Mercury - Atomic Absorption	MET 540.3		X	X
SM 2340 B	-1997	20046600	Hardness as $CaCO_3$ (Calc.)	INO 200.2	X		
SM 4500-Cl^- E	-1997	20086800	Chloride	INO 141.0	X		
SM 4500CN-E	21st (1999)	20096202	Cyanide, Total	INO 180.1		X	X
LACH 10-109-12-2-A	2009	60006362	Fluoride	INO 220.9		X	X
EPA 353.2	2 (1993)	10067604	Nitrate and Nitrate + Nitrite Nitrogen	INO 220.9		X	X
EPA 375.2	2 (1993)	10073004	Sulfate	INO 370.3		X	X
SM 2130 B	21st (2001)	20042608	Turbidity, Nephelometric	INO 380.3	X		
SM 2510B	21st (1997)	20048402	Auto. Alkalinity, pH, and **Conductivity**	INO 115.1	X		
SM 2320B	-1997	20045607	Automated **Alkalinity**, pH, and Conductivity	INO 115.1	X	X	
SM 2540C	21st (1997)	20050208	Total Dissolved Solids	INO 320.1		X	
SM 4500-NO_2-B	21st (2000)	20112805	Nitrite Nitrogen, Manual Colorimetric	INO 220.8		X	X
EPA 365.1	2 (1993)	10070005	Phosphorus, Reactive Diss. (Orthophosphate) —auto	INO 310.6		X	
LACH 10-114-27-1-A	1999	No TNI code	Silica, Dissolved, Low Level	INO 360.2		X	

2.18.2.Non-Potable Water

Regulatory Method	**Edition**	**TNI Code**	**Description**	**WSLH Method #**	**Accreditation**	
					NELAC	WDNR
EPA 200.7	4.4 (1994)	10013806	Inductively Coupled Plasma-Emission Spectrometry	MET 400.2	X	X
SW-846 6010 B	2 (1996)	10155609	Inductively Coupled Plasma-Emission Spectrometry (Sludge rule)	MET 400.2	X	X
EPA 1638	1996	10124002	Inductively Coupled Plasma-Mass Spec	INO 400.4		X
EPA 245.1	3 (1994)	10036609	Mercury	MET 540.3	X	X
EPA 1631E	2002	10237204	Mercury - Atomic Fluorescence	INO 541.2	X	X
SW-846 7470 A	1 (1994)	10165807	Mercury (Sludge rule)	MET 540.2	X	X
SM 4500-Cl- E	-1997	20086800	Chloride	INO 141.0	X	X
EPA 300.0	2.1 (1993)	10053200	Anions by Ion Chromatography	INO 200.5		X
SM 4500CN^-E	-1999	20096417	Cyanides, Total	INO 180.1		X
SW-846 9014	0 (1996)	10193803	Cyanides, Total	INO 180.1		X
SM 4500CN-G	-1999	20097216	Cyanides, Amenable to Chlorination	INO 180.1		X
EPA 351.2	2 (1993)	10065404	Total Kjeldahl Nitrogen with Copper Sulfate	INO 230.3	X	X
EPA 350.1	2 (1993)	10063602	**Ammonia** & Nitrate + Nitrite	INO 220.3	X	X
EPA 353.2	2 (1993)	10067604	Ammonia & **Nitrate + Nitrite**	INO 220.3	X	X
EPA 353.2 (calc)	2 (1993)	10238809	Nitrate (calc.)	INO 220.3	X	X
EPA 375.2	2 (1993)	10073004	Sulfate	INO 370.3	X	X
ASTM D1252-06B	2006	30006189	Chemical Oxygen Demand	INO 280.3	X	X
EPA 445.0	1.2 (1997)	10081400	Chlorophyll	INO 151.1	X	
SM 2540 D	-1997	20051201	Total Suspended Solids	INO 340.1	X	X
SM 2540C	-1997	20050402	Total Dissolved Solids	INO 320.1	X	X
SM 2540 E	-1997	20051585	Volatile Solids	INO 340.1 INO 330.1	X	X
SM 4500-NO_2-B	-2000	20113104	Nitrite Nitrogen, Manual Colorimetric	INO 220.8	X	X
EPA 365.1	2 (1993)	10070005	Total Phosphorus	INO 310.2	X	X
EPA 365.1	2 (1993)	20124805	Phosphorus, Reactive Dissolved	INO 310.6	X	X

			(Orthophosphate)—auto			
SM 5210B	-2001	20135255	Biochemical Oxygen Demand (includes carbonaceous BOD)	INO 260.1	X	X
SM 2340B	-1997	20046600	Hardness (Calculation Method)	INO 200.2	X	X
SM 2320B	-1997	20045607	Alkalinity	INO 115.1	X	X
SM 2510B	-1997	20048606	Conductivity	INO 115.1	X	
SM 2540 B	-1997	20049405	Total Solids	INO 330.1	X	X
SM 4500-H+ B	-2000	20105219	pH	INO 300.0	X	X
EPA 1664 A	1999	10127807	Oil & Grease	INO 250.3		X
LACH 10-114-27-1-A	1999	No TNI code	Silica	INO 360.2		X
SM 4500 S^{2-} G	-2000	20127053	Sulfide	INO 375.3		X

2.18.3.Solid and Chemical Materials

Regulatory Method	**Edition**	**TNI Code**	**Description**	**WSLH Method #**	**Accreditation**	
					NELAC	WDNR
SW-846 1311	0 (1992)	10118806	Toxicity Characteristic Leaching Procedure	MET 715.1	X	X
SW-846 6010 B	2 (1996)	10155609	Inductively Coupled Plasma-Emission Spectrometry	MET 400.2	X	X
SW-846 7471A	1 (1994)	10166208	Mercury, Solids	MET 540.2	X	X
EPA 1631E	2002	10237204	Mercury, Atomic Fluorescence	INO 541.1		X
SW-846 9045C	3 (1995)	10198400	pH, Solid	INO 295.0		X
SW-846 9040C	---	10244403	pH, Hazardous Waste Characterization (Corrosivity)	INO 301.0		X
SW-846 9014	0 (1996)	10193803	Cyanide, Tot. in Solids	INO 180.2		X
EPA 325.2	1978	10057202	Chloride in Solids	INO 141.3		X
LACH 13-107-06-2-D			TKN in Soils	INO 230.4		X
LACH 12-107-06-1A			Ammonia in Soil Extracts	INO 220.2		X
LACH 12-107-04-1B			Nitrate + Nitrite in Soil Extracts	INO 220.2		X
USGS I-6600-85			Total Phosphorus in Soil	INO 310.4		X
SM 2540G	-1997	20005269	Percent Solid, Solid & Semi-Solid Samples	INO 330.2		X

Radiochemistry Department

Table of Contents

3.1. Personnel

Table #: Education and Experience

Name	Title	Degree	Yrs Exp
Gary Krinke	Chemist Advanced	H.S. + 3.5 yrs University	30
Genevieve Martinez	Associate Chemist	B.S. Chemistry	2
Michael Populin	Associate Chemist	B.S. Biochemistry	3
Vacant	Chemist Supervisor		

3.2. Training

Please see section 1.10 of the general chapter of this QA manual for training requirements common to all EHD employees.

Radiochemistry Specific Training

Initial demonstration of capability is completed according the Section 1.10 of this QA Manual. Ongoing Demonstration of Capability (DOC) is completed annually by four consecutive laboratory control samples with acceptable levels of precision and accuracy or by an alternative method (refer to Section 1.10 of this QA Manual). Calculations are carried out using the same forms as the initial DOC's. The only exceptions are gamma scan and radon in air, which use the successful completion of proficiency test samples as the DOC's**.** The Analyst Training record will keep track of training and the trainer.

Documentation and Forms

- R:\EHD\ESS(4900)\ESS Radiochem(4970)\RAD QC\Analyst Certification Statements
- R:\EHD\ESS(4900)\ESS Radiochem(4970)\Forms\The Forms
    - 4970.2F Instrument Certification
    - 4970.133F Analyst Training Record

Radiation safety training is required of all Radiochemistry department staff and is obtained through UW-Madison Radiation Safety Office (http://www.ehs.wisc.edu/radiationsafetytraining.htm)

3.3. Document Control System

Please see section 1.11 of this QA Manual for information about location and control of lab-wide and division level SOPs.

For the Radiochemistry department, all "official" and current SOPs, related forms, and documents are kept in electronic format on O:\SOP\EHD\ESS\Radiochemistry\Final and will be available to all staff members as "read-only" documents. A hard copy of the SOP with signature page is maintained with the department. Copies will be made available to customers or to regulatory agencies if requested. Please see section 1.25.1 of this QA manual for a link to a table of contents for department SOPs.

SOPs will be reviewed at least every three years and SOPs (IOP, GENOP and Method) related to drinking water samples will be reviewed annually. SOPs are revised when necessary due to instrument/technology changes, regulatory changes, etc. Each new revision will have a new revision number, an effective date, and will list the SOP it is replacing. Any archived documents 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. For detailed information regarding the control of documents, please see EHD GENOP 103 SOP Guidance for Writing and Managing Department-Level SOPs.

External reference documents such as sources of reference methods, instrument instruction manuals, and software versions are controlled via the Related Documents/References sections of SOPs. If new versions of external references become available and are implemented, a new SOP revision would be started and the new external reference would be listed in the Related Documents/References section.

3.4.Records Retention, Control, and Storage

Paper Records

Please see section 1.12 of the general chapter of this QA Manual for RDA information, laboratory notebook information, and information about storage of records in the basement and at the State Records Center.
Records information specific to the Radiochemistry department can be found in ESS RAD GENOP 014 Rad Records and ESS RAD GENOP 30 How to Store Record Storage Boxes.

A listing of the record boxes stored is available on the shared EHD server: R:\EHD\ESS(4900)\ESS Radiochem(4970)\Record Storage and Management\WSLH Records Disposal Request Forms-Radiochemistry.

Electronic Data Back-up

Please see section 1.17.4 of the general chapter of this QA Manual for electronic and computer back-up information.

3.5.Sample Handling Procedures

Please see section 1.16 of the general chapter of this QA Manual for sample acceptance information on the divisional level.

The Sample Acceptance Policy for the Radiochemistry department is detailed in ESS RAD GENOP 020. This Sample Acceptance Policy includes what to do if sample acceptance criteria are not met.

Procedures related to sample receipt, distribution, handling, and storage for Radiochemistry samples are documented in:

- ESS RAD GENOP 011 Radiochemistry Sample Disposal
- ESS RAD GENOP 020 Radiochemistry Sample Receiving and Check-In

Sample Collection

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 Website: http://www.slh.wisc.edu/environmental/radiochemistry/collection-kits/

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. Samples are received in the sample receiving area and assigned a unique laboratory number and add the tests and client information is entered into Horizon. The samples are passed on to the department's staff to be assessed for acceptability. 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). HDM staff will complete the data entry of demographic information.

Sample are logged into Horizon LIMS and labeled according to section 1.16 of this QA Manual.

Sample Handling

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. For liquid samples, homogenization is achieved through thorough shaking. 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.

Sample Storage and Disposal

Samples are stored in the sample storage room. Samples that exceed 0.5 mrem/hr will be stored in a separate location. Perishable samples, such as milk, are stored in the cooler. Radon in air and water samples are kept in the lab since they are analyzed the same day they arrive. 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 Laboratory Safety Guide.

Chain of Custody

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.

3.6.Data Review and Reporting

Data Review

Analysis results are reviewed by the analyst and approved by another analyst or supervisor. The approval process includes matching the work list data with the data transferred to the instrument software or Horizon either 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, emailed or faxed to the client.

A complete hand calculation of programs is performed to verify results if the program is modified during the year. If the program has not been modified, this is not necessary. Documentation of program verification is kept either in the QC lab notebook or in the R:\EHD\ESS(4900)\ESS Radiochem(4970)\Programs\QC folder.

Data Reporting

The analyses required for a particular sample are indicated by the test codes selected within Horizon. Default codes are generally presented for a specific analysis. When the results of a chemical analysis are calculated, the results are automatically downloaded to Horizonwhere they can be viewed and printed.

After the completion of the gamma analysis, the results must be transferred from the Canberra Gamma Counting System to Horizon.

Radon data is reported applying a Moisture correction factor. Calculation is performed using a spreadsheet located R:\EHD\ESS(4900)\ESS Radiochem(4970)\Programs\Radon in Air

Hard copies of testing results are provided to the submitter of the sample.

Drinking Water Requirement

If an MCL (Maximum Contaminant Level) for an analyte regulated under ch. NR 809 has been exceeded for a PWS (Public Water Supply) sample, additional analysis will be scheduled for the sample (i.e. Radium 226/228, uranium, etc.) and the water supply facility may be notified within 48 hours of completing the analyses (NR 149.19(7)). The analysis is considered complete when a batch is finalized in Horizon after the peer review audit. Applicable MCLs will be listed in SOPs so that the analyst has easy access to them. The analyst may notify the water supply facility if necessary.

Estimating Uncertainty of Measurement

Uncertainties are calculated for each sample within Horizon and are based on counting statistics. Formulas are given in the SOPs for each method under the calculation section.

3.7. Procedures for Accepting New Work/Review of Requests, Tenders and Contracts

The department follows the described procedure in Section 1.15 in the general chapter for accepting new work/review of requests, tenders and contracts.

3.8. Laboratory Facilities

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, 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 both for cold storage of samples and for room temperature storage of samples adjacent to the sample preparation area. Additionally, the lab has a six-foot hood and workspace near the loading dock for soil grinding and the receipt of emergency response samples. The entire building is security locked.

The counting room is equipped with a sixteen detector low background alpha-beta counter, a single detector low background alpha-beta counter with an automatic sample changer, six intrinsic germanium detectors using Canberra gamma analysis software, an EG &G Ortec alpha spectrometer (16 detectors), and six Ludlum alpha scintillation cell counting systems. The weighing room includes a liquid scintillation analyzer.

Computer Facilities

All of the PC's are connected via a LAN network administrated by the Office of Information Systems.

3.9. Instrumentation and Equipment

Laboratory Instruments Summary for Radiochemistry

ITEM	MANF.	MODEL	ACQ. DATE	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	Dec 1999	Calibration Weight Sets
Geiger Counter	Ludlum	Model 3	2005	Survey Meter System

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	Mar 1999	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	Oct 1999	16 port drying chamber
Centrifuge	IEC	GP8	2000	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 16 detectors; P-10 gas
Liquid Scintillation Analyzer	PerkinElmer	Tri-CARB 3170TR/SL	Nov 2007	adj. discriminator channels
Radon Gas Counters	Ludlum	2000	1983 & 2001	2 cells per system
Gamma Spectroscopy	Apex Analysis Software		2004, 1996, 1997, two in 2008, 2012	6 intrinsic germanium 3-Canberra crystals, 1 PGT
Alpha Spectrometer	EG & G Ortec	Octete-Plus	Aug 1999	8 PIPS, Ultra AS 600
Alpha Spectrometer	EG & G Ortec	Octete-Plus	June 2010	8 PIPS, Ultra AS 600
Geiger Counter	Ludlum	Model 3	June 2010	Survey Meter System
Muffle Furnace	Fisher	750-126	June 2010	Programmable forced draft furnace up to 1125˚C

EG & G Ortec Eight Channel Alpha Spectroscopy System. (Instrument System ID #3)

The analyses performed on this piece of equipment are for research purposes, polonium, and SDWA uranium analysis. Energy calibrations and efficiencies are done prior to initial use, after significant maintenance, and when the instrument’s response continually exceeds predetermined acceptance criteria for quality control. The energy calibration is checked weekly and the efficiency is checked monthly using a purchased standard disk containing U-238, U-234, Pu-239, and Cm-244. Both energy calibrations and efficiency checks are plotted in the Alpha Vision software. The energy calibrations should have a gain between 4.1 and 5.2 and an offset range between 2500 and 3300. If the quadratic is greater than ± 0.001, the calibration should be reevaluated. The Alpha Vision software calculates the limits for the efficiency calibration checks based on counting statistics. If an efficiency check is outside the calculated limits, the efficiency will be reevaluated. Background measurements are performed on a monthly basis. Important

information concerning the instrument performance is stored in the electronic equipment database or the instrument's maintenance logbook.

Protean - Low Background 12 detector α-β counter. (Instrument System ID #1)

A Sr-90 sealed un-calibrated source and a Th-230 sealed 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 be recounted up to three times. If the count rate for that detector remains outside of the upper control limits that detector is not used that day and the reason is investigated. See ESS RAD IOP 009 Protean Alpha Beta for information on how daily control check trends are dealt with. Important information concerning the instrument performance is stored in the electronic equipment database or the instrument's maintenance logbook.

A contamination check of at least 30 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 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 control limit, the detector is not used until the problem is resolved. Also, all samples counted since the last good check will be re-evaluated. See ESS RAD IOP 009 Protean Alpha Beta for detailed instructions on decontamination procedures and other trouble shooting techniques.

A background count of 1000 minutes is also done for use as background subtraction in calculations. The upper control limit set at 2.0 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. Also, all samples counted since the last good check will be re-evaluated. 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 equipment database or the instrument's maintenance logbook. See the instrument IOP for information about instrument efficiency.

If a significant repair was required, such as replacement of electronic equipment, detector, or other hardware, then it will be necessary to perform energy and efficiency calibrations. Any adjustments or maintenance of the system is recorded in the instrument's maintenance log.

Count and store the results of 10-20 daily checks to re-establish the control limits.

PerkinElmer 3170TR/SL Liquid Scintillation Analyzer. (Instrument System ID # 33)

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 printed for 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, whenever the system does not perform according to manufacturer's specifications, call for service. Do not use the instrument until the service has been completed.

If a significant repair was required, such as replacement of electronic equipment, detector, or other hardware, then it will be necessary to perform energy and efficiency calibrations. Any adjustments or maintenance of the system is recorded in the instrument's maintenance log.

Count and store the results of 10-20 daily checks to re-establish the control limits.

Canberra/PGT Gamma Spectroscopy System (Instrument System ID #5, 6, 7 34 & 35)

The QC software was professionally set up by the Canberra technician when the system was installed. The Daily Check routine simultaneously checks the linearity, the FWHM of the Co-60 1332 peak, and checks the decay corrected activity of the 1332 peak. The sources used to perform this check are a 0.5L simulated water sample for the p-type co-axial detectors and a 2 oz simulated soil sample for the n-type planar detector. The sources are purchased from Isotope Products Labs each year. The sources are different from the sources used for instrument calibration.

The data is stored in the Canberra QC database and the control plots can be viewed at any time. The limits are set dynamically.

If the daily check is out of the upper warning limits, recount the daily check. If the results continue to be out of the limits, check to be sure that all of the demographic information has been entered correctly. Make sure the correct analysis sequence is being selected. If the daily check is still out of control, attempt to determine if there is a system malfunction. Contact Canberra for assistance if necessary. The most usual problem is that the linearity has drifted slightly, and the peaks are no longer being found. The message displayed in the report window is "data is not stored with results". Adjustment of the super gain will usually correct the problem. According to the Canberra technical support, it is not necessary to perform a new efficiency calibration after adjusting the fine gain.

If a significant repair was required, such as replacement of electronic equipment, detector, or other hardware, then it will be necessary to perform energy and efficiency calibrations. Any adjustments or maintenance of the system is recorded in the instrument's maintenance log.

Count and store the results of 10-20 daily checks to re-establish the control limits.

On each day of use, a 900-second empty shield background check is performed. The Canberra software displays the results of the performance check and flags any results outside of the upper control limits.

The data is stored in the Canberra QC database and the control plots can be viewed at any time. The limits are set dynamically.

If the background check is out of the upper warning limits, recount the background. If the results continue to be out of the limits, carefully clean the surface of the detector and inside the shield with a solvent. If the background is still elevated, attempt to determine if there is a system malfunction. Contact Canberra for assistance if necessary. If it is determined that the system is operating properly, but the contamination persists, then count a long background to be used for background subtraction on samples. Count and store the results of 10-20 short background checks to re-establish the control limits.

If the daily background check was performed incorrectly, be sure to remove this data from the QC database.

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 cannot be removed. Detector #2 has measurable uranium progeny inherent in the shield or pre-amp materials which cannot 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.

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 day of use 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. The instrument software generates limits dynamically, though a maximum 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 logbook in the equipment database.

If a significant repair was required, such as replacement of electronic equipment, detector, or other hardware, then it will be necessary to perform energy and efficiency calibrations. Any adjustments or maintenance of the system is recorded in the instrument's maintenance log.

Count and store the results of 10-20 daily checks to re-establish the control limits.

Ludlum Scintillation Cell Counters (Instrument System ID #8 & 11-20)

A thorium-230 standard is counted on each day of use 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 each day of use 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.

If a significant repair was required, such as replacement of electronic equipment, detector, or other hardware, then it will be necessary to perform energy and efficiency calibrations. Any adjustments or maintenance of the system is recorded in the instrument's maintenance log.

Count and store the results of 10-20 daily checks to re-establish the control limits.

Efficiency Determination

Each instrument's efficiency is determined prior to initial use and yearly for each nuclide in each procedure or counting geometry. Efficiencies are also performed in the event of major repair or instrument modification and when the instrument's response exceeds predetermined acceptance criteria for quality control. The specific instructions for preparing standards for efficiency determinations are found in the standard operating procedures for the various analytical procedures.

The efficiency is confirmed by using verification standards according to the schedule outlined in the method SOP's and IOP's. All instruments follow a regular maintenance schedule as needed.

3.10. Laboratory Supplies and Chemicals

LABORATORY SUPPLIES

Pipettes

A logbook is maintained for all micropipets. Each pipette is identified by either a serial number or a unique ID number. The pipettes are calibrated professionally on a quarterly basis. All verification information and required maintenance are recorded in the logbook.

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 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 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).

CHEMICALS

See R:\EHD\ESS(4900)\ESS Radiochem(4970)\RAD Health and Safety\Chemical Inventory

3.11. General Quality Control Procedures

3.11.1.Quality Control Data Gathering and Documentation

Required QC is listed in each technical SOP. Generally, required QC samples include blanks, accuracy, and precision samples. Quality control samples that are analyzed and entered in Horizon LIMS are given a unique sample number and are linked to all samples in the batch.

3.11.2.Corrective and Preventive Action Procedures

Please see section 1.21 of the general chapter of this QA manual for additional information on corrective and preventive action.

Corrective action must be taken after any QC failure and any time an analyst notices an instrument problem or analytical problem. Procedures for taking corrective action in the laboratory are often instrument specific. Please see individual technical SOPs for corrective action information related to common problems for specific procedures. Corrective action may include repairing, adjusting, or servicing instrument hardware or software; changing sample handling or treatment; or correcting analyst errors. Corrective action may be performed by the analyst or may require an outside service call. If outside service is required the Department Supervisor will be consulted.

The corrective action must be documented either within the analytical run, in the appropriate instrument logbook, or in an Occurrence Management Report, which is accessed through the internal website. Corrective action documentation will include: a) identifying the QC failure and its root cause (if known); b) noting specific corrective actions that were taken or attempted; c) verifying that the corrective action worked, and stating any further action that will be taken.

3.11.3. Departures from Documented Policies and Procedures

Please see section 1.21.3 of the general chapter of this QA Manual for information about permitting departures from documented policies and procedures, who can approve departures, and how to document them.

3.12. Quality Control Limits Procedures

Quality control (QC) limits define the precision and accuracy of all reported data. Limits are established and created in Horizon for each parameter, matrix, and QC type (e.g. blanks, performance samples, duplicates, and spikes). Limits are contained in Horizon. If a QC result exceeds control limits, corrective action must be taken and documented. Affected data must either be re-analyzed or qualified.

3.12.1. Precision Control Limits

Precision is defined as the measure of mutual agreement among individual measurements (US EPA, 2005). Precision limits are based upon relative percent difference calculations for either the sample and its duplicate or for the Matrix Spike/Matrix Spike Duplicate pair.

A normalized method for specifying precision is the relative percent difference (RPD) or the replicate error ratio (RER) expressed as follows:

$$\text{RER} = \frac{|A-B|}{\sqrt{U_A^2 + U_B^2}} \leq 2$$

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

Where:

A, B = the two analysis results (duplicates)

U = the uncertainty of the analytical results (Duplicates, A & B)

According to the SDWA Manual, 5th edition, the replicate error ratio of two duplicate results (i.e. A and B) must be less than or equal to two (standard deviations).

The upper and lower control limits define the acceptable relative percent difference for duplicated samples. The control limit is calculated using the following equations:

Upper Control Limit = $\bar{x}$ + 3S (upper warning limit + 2S)

Lower Control Limit = $\bar{x}$ – 3S (lower warning limit – 2S)

Where:

$\overline{x}$ = mean percent recovery

S = the standard deviation of the percent recovery, analyzed over a given period (refer to SDWA Manual 5th edition and the applicable analytical method SOP).

3.12.2. Accuracy Control Limits

Accuracy is the degree of agreement between an observed value and an accepted reference value. Accuracy control limits are based upon the mean and standard deviation of the percent recoveries of 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 include standard matrices (i.e., reagent water) and a laboratory matrix (i.e., Ottawa sand) which may be used for calibration check verifications, laboratory control samples, outside source check standards, etc. The percent recovery (% Rec) is calculated using the formula:

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

Where:

A = the observed concentration of the spiked sample
B = the background concentration of the sample
C = the known concentration of the spike

The control limits (upper and lower) defined in section 3.12.1 are calculated as described in the previous section.

3.13. Internal and External PT Studies

All internal and external PT samples will be handled the same as routine samples. See ESS RAD QAOP 002 PT Sample Procedures.

3.13.1.External PT Programs Schedule

Program Name	**Method/Technology**	**Schedule**
Bowser Morner	Radon	monthly
MAPEP (Mixed Analyte Performance Evaluation Program)	Air Filter Gamma Air Filter Alpha Beta Water Vegetation Soil Water Alpha Beta Alkaline Water	2/year
ERA RAD	Gamma Emitters Gross Alpha/Beta Naturals Strontium-89/90 Tritium Iodine-131	2/year

3.14. Internal Audits

Please see section 1.20 of the general chapter of this QA Manual for internal audit procedures. The Radiochemistry Department will participate in the annual systems audit conducted by divisional quality assurance personnel. Radiochemistry department staff, with oversight from QA personnel will conduct method internal audits of accredited methods.

3.15. Traceability of Measurement

Please see section 1.13 of the general chapter of this QA Manual for basic information about traceability. 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.

A detailed procedure is in place that describes ordering, preparing, and disposing of radioactive materials: ESS RAD GENOP 008, "Radioactive Standards Procurement and Preparation."

For non-radioactive reagent traceability information see ESS RAD GENOP 018, "Radiochemistry Reagent Guide."

3.16. Method References

- USEPA - Bishop, C.T., et.al., "Radiometric Method for the Determination of Uranium in Water", EPA 600/7- 79-093, EMSL-LV, April (1979)
- Edwards, K.W. "Isotopic Analysis of Uranium in Natural Waters by Alpha Spectrometry, Radiochemical Analysis of Water", Geological Survey Paper 1696-F, U.S. Government Printing Office, Washington D.C., (1968)
- USEPA, "Tentative Reference Method for Measurement of Tritium in Environmental Waters". EPA-600/4-75-013. December (1975)
- USEPA, Prescribed Procedures for Measurement of Radioactivity in Drinking Water. EPA 600/4-80-032, pp 38-49, 49-57, 58-74, 75-81 and 96-102. August (1980)
- APHA et. al., "Standard Methods for the Examination of Water and Wastewater", 17th ed. (1975).
- APHA et. al., "Standard Methods for the Examination of Water and Wastewater", 14th ed. (1990).
- USEPA - Earl L. Whittaker and Herman L. Krieger, "Section 9 Radioactive Strontium in Drinking Water Method 905.0", EPA -600/4-80-032; August (1980)
- USEPA, "Radioactive Strontium in Drinking Water"; by Herman L. Krieger. EPA -600/4-75-008 Revised March (1976)
- US EPA, “Manual for the Certification of Laboratories Analyzing Drinking Water”, 5th Edition, EPA 815-R-05-004 Revised January (2005)

3.17. Standard Operating Procedures

A complete list of standard operating procedures for the Radiochemistry department can be found in the tables of contents located at:
O:\SOP\EHD\Division Wide\Final\001 TOC Internal Documents.xlsx

The table of contents is in spreadsheet format (click on the tab for Radiochemistry). The spreadsheet can be sorted and filtered via the Excel data tab along the top of the page. The table of contents includes unique SOP identifiers, titles, revision numbers, effective dates, review dates, and hyperlinks to the actual documents.

3.18. Accredited Methods

Note 1—The Radiochemistry department has some specialized accreditations, which are included below for ease of referral:
AARST-NRPP = American Association of Radon Scientists and Technologists-National Radon Proficiency Program

3.18.1.Drinking Water

Regulatory Method	**Description**	**WSLH Method #**	**Accreditation[1]**
EPA 900.0	Gross Alpha	RAD 001	NELAC, EPA
SM 7110C	Gross Alpha	RAD 035	NELAC, EPA
EPA 900.0	Gross Beta	RAD 001	NELAC, EPA
EPA 903.1	Radium 226	RAD 009	NELAC, EPA
EPA 904.0	Radium 228	RAD 009	NELAC, EPA
EPA 906.0	Tritium	RAD 015	NELAC, EPA
EPA 901.1	Gamma Emitters	RAD 018	NELAC, EPA
SM 7500 U-C	Uranium	RAD 032	NELAC, EPA
EPA 901.1	Iodine-131	RAD 007	NELAC, EPA
EPA 905.0	Strontium 89/90	RAD 013	NELAC, EPA

3.18.2.Non-Potable Water

Regulatory Method	**Description**	**WSLH Method #**	**Accreditation**
EPA 900.0	Gross Alpha	RAD 002	NELAC
EPA 900.0	Gross Beta	RAD 002	NELAC
EPA 903.1	Radium 226	RAD 009, RAD 014	NELAC

Environmental Toxicology Department

Table of Contents

4.1.Personnel

4.1.1. Table 1. Education and Experience

The Environmental Toxicology Department team is staffed with full-time toxicologists, a manager, a supervisor, and University of Wisconsin student workers that assist during summers and weekends. The Environmental Toxicology Department works as a self-directed workgroup managing test design, quality assurance and other lab issues as a team.

Name	Title	Degree	Yrs Exp.
Dawn Perkins	Environmental Toxicology Supervisor	BS Zoology, MS Limnology and Marine Science	24
Jocelyn Hemming	Environmental Microbiology and Toxicology Manager	BA Biology, PhD Environmental Toxicology	21
Dagmara Antkiewicz	Assistant Scientist	BS Biology, PhD Environmental & Molecular Toxicology	12
Rebecca Fahney	Associate Environmental Health Specialist	BS Conservation Biology	2
Abigail Merrick	Associate Environmental Health Specialist	BS Natural Resources & Environmental Science, MS Ecology	1
Nina Desianti	Environmental Health Specialist	BS Biology, MS Botany, PhD candidate Biodiversity, Earth and Environmental Science	1

4.2.Training

See section 1.10 of this QA manual for training requirements common to all EHD employees.
Training specific to the Environmental Toxicology Department includes:

- Environmental Toxicology New Employee Training Checklist: M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\Forms\FINAL FORMS\Training\EHD ET New Employee Training Plan & Form.docx Note: prior to conducting lab work involving fish cultures new employees must complete New Animal User Training via the University of Wisconsin-Madison's Research Animal Resource Center (RARC) and read associated RARC Protocols as outlined in the training checklist.
- Certification Statement for Current Methods: All Method SOPs for accredited testing must be reviewed initially. Method SOPs must be reviewed and signed on the last page by each analyst whenever a new revision is completed.
- Initial Demonstration of Capability (DOC): All new employees must perform a variety of toxicity tests as they are trained. The tests and required criteria are recorded on the following spreadsheet: M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\LABWORK\Q A\Demonstration of Capability\DOC Initial - all Staff\initial demonstration of capabilities.xlsx and on the training checklist.

- Continuing Demonstration of Capability (DOC): All employees must perform an annual Demonstration of Capability. Due to the unique nature of whole effluent toxicity testing it is not practical to have one analyst complete all parts of a single test. Instead, each analyst is required to complete at least one set up, renewal and shut down for each routine WET test method. Renewal of the algae test is an exception since no renewal occurs. DOC's for each calendar year and each analyst are recorded on a spreadsheet and found in the following directory: M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\LABWORK\Q A\Demonstration of Capability

4.3. Document Control System

See section 1.11 of this QA Manual for information about location and control of lab-wide and division level SOPs.

All "official" and current Environmental Toxicology department SOPs and related documents are kept in electronic format in the following directory: O:\SOP\EHD\ESS\Enviro Organic\Enviro Tox\Final. SOPs are available to all staff members as "read-only" documents. Printed hard-copy versions of departmental SOPs exist in binders located in the laboratory. Copies will be made available to customers and regulatory agencies upon request.

SOPs are revised when necessary due to instrument/technology changes, regulatory changes, etc. Each new revision will have a new revision number, effective date, and the revision number of the SOP it is replacing. Archived SOPs will remain available. Each item will be clearly labeled and dated so it is apparent when the document was in force. A new revision number will not be assigned if updates do not significantly affect content, such as typos, grammar, adding a footer, etc. Starting in 2017, new and revised SOPs also contain a Revision Tracking Table. In 2007, the department adopted a new SOP format and numbering system resulting in revision numbers starting at 1. Old SOP numbers are listed under the new SOP number only for the first revision from old to new format. New SOPs created after 2007 are numbered Revision 0 for the first version. For detailed information regarding document control, see ESS ENV TOX GENOP 1000 "How to Write and Manage SOPs".

External reference documents such as sources of reference methods, instrument instruction manuals, and software versions are controlled via the Related Documents/References sections within SOPs. Additionally, a list of external documents referenced in SOPs is in an Excel spreadsheet with the location listed and for electronic documents, a link to the document: O:\SOP\EHD\Division Wide\Draft\In Progress\TOC External Documents.xlsx. If new versions of external references become available and are implemented, the SOP will be revised and the new external reference will be listed in the Related Documents/References section.

4.4. Records Retention, Control, and Storage

See section 1.12 of this QA Manual for RDA information, laboratory notebook information, and information about storage of records in the basement and at the State Records Center.

The laboratory utilizes bench sheets, logbooks and databases for various purposes including: instrument

maintenance, cataloging of standards and their preparation, cataloging of chemical storage and expiration date, recording sample numbers, and temperature record-keeping for incubators, ovens, refrigerators, freezers and walk-in coolers. Logbooks are labeled with title, effective dates and logbook number. A master spreadsheet tracks logbook locations and effective dates and is located at: M:\EHD\QC\Archived Records.mdb. Hardcopy records including logbooks, bench sheets, training records and other documents necessary to confirm electronic records are stored in file cabinets in the Environmental Toxicology office area for two years. After two years they are stored in the basement and after three years they are stored at the State Records Center.

4.5.Sample Handling Procedures

See section 1.16 of this QA Manual for sample acceptance information on the divisional level. Sample login and handling for the department is described in ESS ENV TOX GENOP 1101 "Sample Receiving".

Environmental Toxicology department samples are collected either by laboratory sampling staff or external field personnel. Sample collection is performed according to ESS ENV TOX GENOP 1100 "Sample Collection for Whole Effluent Toxicity Tests".

There are no special handling procedures for enforcement samples, except for the following:

a. Horizon Data Management staff maintains possession of all original sample submission forms (e.g. test request forms, chain of custody forms). Photocopies of each submitted form are provided to the Environmental Toxicology Department when Receiving Department staff delivers enforcement samples to the department.
b. Upon completion of all tests, enforcement samples are disposed of as described in ESS ENV TOX GENOP 1101 "Sample Receiving" unless a different sample retention time period is requested by the sample requestor.

4.6.Data Review and Reporting

See section 1.22 of this QA Manual for division wide information on reporting analytical results.

During testing and at the conclusion of each test, results are recorded by the analyst on the bench sheets along with any data qualifiers. Test results, QC data, and any data qualifiers are entered into an official report form as described in ESS ENV TOX GENOP 1103 "Report Writing". Raw data worksheets and supplemental records are reviewed during result entry. The entire data package and report is reviewed by a peer or supervisor prior to reporting. Reports are saved electronically on the server and sent via email to the Biomonitoring Coordinator at the Wisconsin Department of Natural Resources. A paper copy of the final report is printed, signed, dated and stored in the Environmental Toxicology office files. An occurrence report is completed for amended reports. Final sample results are also entered into Horizon LIMS. The results are peer reviewed by another analyst before they are released to the client. The full report is scanned and attached in PDF format.

4.7. Procedures for Accepting New Work/Review of Requests, Tenders and Contracts

See section 1.15 of this QA Manual for division wide information and policies for accepting new work based on authority granted by the WSLH Board. Section 1.15 also includes considerations for reviewing laboratory capability and capacity to cover the requested scope of work. The WSLH has procedures regarding who can sign contracts and what terms and conditions can be agreed upon. New whole effluent testing, sediment testing and other potential research projects are discussed at the monthly meeting of the Environmental Toxicology/Wisconsin Department of Natural Resources (WDNR) team. Laboratory capabilities, workload and personnel are discussed and the proposed new work is either accepted or rejected. New vertebrate animal testing protocols must be submitted to University of Wisconsin-Madison RARC (Research Animal Resources Center) for approval.

4.8. Laboratory Facilities

The Environmental Toxicology department 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, an incubator and a walk-in 4ºC cooler. Room 204 is separated from Room 205 by a door with an automatic closing mechanism. Room 205 (785 sq. ft.) is comprised of the fathead minnow aquaculture and bench space and incubators used for culturing algae and daphnia. Room 205 is separated from the hallway by a door. Room 205A (105 sq. ft.) is used for special projects. This room is separated from Room 205 by a door. 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. The office area for the Environmental Toxicology department is located across the hallway from the laboratory space.

4.9. Instrumentation and Equipment

See section 1.17 of this QA Manual for division wide information on equipment.

Major analytical instrumentation operated by the Environmental Toxicology Department is listed in a spreadsheet: M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\Equipment\Environ Tox Equipment List.xls

A SpectraMax M5e Microplate Reader is used for determining fluorescence intensity in the 96-hour *Selenastrum capricornutum* chronic bioassay (refer to ESS ENV TOX METHOD 4204 "*Selenastrum capricornutum* 96-h Growth Test") as well as other assays where absorbance, fluorescence and/or luminescence are measured. The M5e is used following instrument operating procedure ESS ENV TOX IOP 3000 "SpectraMax M5e Microplate Reader for Use with the 96-hour *Selenastrum capricornutum* Chronic Bioassay".

Preventative maintenance, cleaning and calibration of all balances is performed yearly by an outside contractor. Balances are checked with certified weights each day of use and are maintained as detailed in ESS ENV TOX GENOP 1302 “Balance Calibration”.

Temperatures are monitored daily as covered in ESS ENV TOX GENOP 1300 “Temperature Monitoring”. The light intensity is measured annually in all chambers and the photoperiod is checked quarterly following ESS ENV TOX GENOP 1303 “Photoperiod Verification”. Light intensity is checked daily when algae testing is being conducted.

An Elga Purelab Option R-7 BP Reverse Osmosis (RO) System provides reverse osmosis (RO) water to the Elga water polisher located in the laboratory. An Elga PureLab Ultra Genetic water polisher provides Type I water used in the preparation of reagents and synthetic laboratory water. See ESS ENV TOX GENOP 1301 “Monitoring and Maintenance of Water Purification Systems” for water system maintenance procedures.

Conductivity, pH and dissolved oxygen are measured using meters following the applicable SOPs. For pH see ESS ENV TOX METHOD 4102 “pH (Electrometric)”. For conductivity see ESS ENV TOX METHOD 4103 “Conductivity”. For dissolved oxygen see ESS ENV TOX METHOD 4104 “Dissolved Oxygen”. Chlorine is measured using ESS ENV TOX METHOD 4107 “Hach Total Chlorine”. Digital thermometers and thermometer probes are NIST traceable and checked for accuracy quarterly against a valid NIST-certified traceable thermometer at a single temperature. All thermometers are also verified annually at two temperatures bracketing the normal range of use. See ESS ENV TOX GENOP 1300 “Temperature Monitoring”.

A calibration check is done quarterly for all pipettes. A list of pipettes in use and calibration data can be found at: M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\LABWORK\Q A\Pipette Calibrations. Pipette calibration is either conducted by an external pipette calibration vendor, by gravimetric method or by using the Artel PCS pipette calibration system following ESS ENV TOX GENOP 1304 “Pipette Performance Checks”.

4.10. Laboratory Supplies and Chemicals

See General Chapter, section 1.18.5 of this QA Manual for division wide information on purchasing. In addition to the approved vendors listed in the general chapter, the Environmental Toxicology Department also uses: ATCC, Modern Water Inc., Bioassay Systems, Aquatic Biosystems, Aquatic Research Organisms, Pentair/Aquatic Ecosystems, That Pet/Fish Place, Pet Warehouse, Dr. Foster & Smith, Naja, Argent Aquaculture LLC, Teledyne Isco, Bulb Connection, CLEPCO/Intelligent Heater, Leisure Concepts, Syndel, and Gem Sensors. The ongoing spreadsheet of vendors used in the Environmental Toxicology Department is located at M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\Forms\Supply & Pro Card Info\Supply Info\SUPPLY - EHD Env Tox Dept.XLS.

Suppliers are used based on past historical ability to meet laboratory needs and those supplies that result in applicable quality control checks meeting criteria. UW MDS/Shop@UW system is used for the

majority of purchases. Toxicity tests all have quality control samples included to evaluate if any of the components impact the testing. If quality control failures are encountered lab staff would investigate the supplies for any issues. If it is decided there is a problem with a purchased component which is impacting the tests or RTTs, an occurrence report would be completed. Chemicals and reagents purchased from outside vendors are ordered and received according to ESS ENV TOX GENOP 1001 "Purchasing and Receiving of Laboratory Supplies". For chemicals and some culture-related food supplies, all pertinent information including manufacturer, storage conditions, date received, date opened and expiration date are recorded in the chemical, reagents and standards Microsoft Access database located at: M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\LABWORK\Chemicals, Reagents & Standards Database.mdb. The lot number, expiration date, and concentration are indicated on the bottle or cylinder. All standards are dated when received to monitor the shelf life. All chemicals will be replaced before exceeding their expected shelf life. Reagents prepared in the lab and outside sourced culture organisms and culture related supplies are also tracked in the Access database in separate tables. When conducting analyses under certified programs (i.e. NELAC and WDNR) the lot number and prepared or opened date are recorded and traceable back to our Access database. This is required to be done for calibration standards and known standards.

4.11. General Quality Control Procedures

The methods used in this department are fully documented in the 2nd edition of the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual" (M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\Methods and Manuals\WI Methods Manual\WET Methods Manual Edition 2 Nov 2004.pdf). In addition, each Environmental Toxicology method SOP includes a section of references used in the generation of the SOP. The references include documents such as the Federal Register, USEPA Methods Manuals, the AOAC Methods Manual or Standard Methods. Detailed, specific quality assurance procedures are also included in each method since quality control is such an integral part of all procedures.

4.11.1. Test Organisms

Selenastrum capricornutum (Algae)
Algae cultures are renewed weekly using fresh media and examined microscopically for density and morphology. Cell counting may also be done by flow cytometry. If flow cytometry is done algae morphology is examined microscopically at least monthly. 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 the wrong cellular morphology are discarded. Species identification by a trained expert is performed annually. Algae cultures are maintained following ESS ENV TOX GENOP 1503 "Culturing of *Selenastrum capricornutum*".

Pimephales promelas (Fathead Minnow)
The following culture conditions are monitored and changes are made when limits are exceeded.

4.11.2. Table 2. *Pimephales promelas* Culture Requirements

Parameter	Acceptable range	Frequency
Temperature	25-27 ºC	2x daily, 1x weekends/holidays
Dissolved oxygen	5.5 mg/L to saturation*	Weekly
Ammonia (Unionized ammonia)	≤ 0.2 mg/L total ammonia	Weekly
Nitrite	≤ 0.5 mg/L as NO_2-N	Weekly
Nitrate	≤ 40 mg/L as NO_3-N	Weekly
Light (16 hours/day)	540-1075 lux	Visually daily, quarterly Hobo
Egg production	3,000 - 12,000/week	Daily
Circulation	300-800 mL/min	Visually daily

*If DO is over 7.5 mg/L then the saturation value needs to be determined at the time by checking temperature and barometric pressure and referencing the saturation DO table found in ESS ENV TOX GENOP 1500 "Culturing of *Pimephales promelas*".

Egg production is graphed weekly and displayed in the lab and recorded on a Microsoft Excel spreadsheet. Any change in food, equipment or parameters is noted on the egg production graph. Results and responses to problems are recorded in logbooks or electronically. Species identification by a trained expert is performed annually.

Ceriodaphnia dubia
Ceriodaphnia are cultured according to ESS ENV TOX GENOP 1501 "Culturing of *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. Species identification by a trained expert is performed annually.

4.11.3. Laboratory water used for culturing and test dilution water

Laboratory control waters (Moderately Hard Water, Hard Water and dechlorinated Madison tap water), used in testing, are analyzed for hardness, alkalinity, conductivity, pH and DO with every new batch prepared. This data is recorded in culture log books or electronically along with any necessary corrective action. During testing, water chemistry data is recorded daily on the corresponding test bench sheets.

Water used for culturing, food preparation and test dilution should be analyzed for toxic metals and organics whenever significant difficulty is encountered in meeting minimum acceptability criteria for control survival, reproduction or growth.

4.11.4. 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. Trout or salmon chow is ordered semi-annually, at a minimum, to ensure freshness. Yeast, fish food and cerophyll (YFC) mixture used to feed daphnids is assigned an expiration date of ten days from the thaw date. Algal slants are ordered and used to restart the culture approximately every three months. Algal culture performance is recorded on the algae batch tracking sheets and on the algae harvest run chart located at: M:\EHD\ESS(4900)\ESS Biomonitoring(4922)\LABWORK\Q_A\Algae_Culture\Algae_Harvest_Run_Chart.xlsx. If the culture performance does not meet criteria, this may reflect the food quality. 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. If food is suspect and cultures are not performing as expected, organics and metals can be measured. Concentrations of total organochlorine pesticides shall not exceed 0.15µg/g wet weight. Concentrations of total organochlorine pesticides plus PCBs shall not to exceed 0.30µg/g wet weight. Concentrations of toxic metals (Al, As, Cr, Cd, Cu, Pb, Ni, Zn, expressed as total metals) shall not exceed 20 µg/g wet weight. If any of these parameters are exceeded the food shall not be used.

4.11.5. Quality Control Data Gathering and Documentation

Quality control documentation, including, but not limited to equipment temperatures, equipment calibrations, equipment checks, test QC, culture temperatures and culture performance, is recorded in bound laboratory logbooks, on log sheets, on test bench sheets, on control charts, or electronically. QA data is gathered and recorded in a centralized electronic location on a weekly, monthly or quarterly basis. This documentation is monitored on a continuous basis to ensure the laboratory is producing high quality data and quality control limits are not being exceeded. QC results of controls are reported with each toxicity test. Chemistry analyses have blanks, spike and replicates performed on each analysis day if required. Each METHOD SOP outlines the required QC samples and parameters.

4.11.6. Corrective and Preventative Action Procedures

Procedures for taking corrective action are outlined in each specific SOP. All corrective actions are noted either on run charts, test bench sheets, or in the permanent laboratory logbook. Corrective action is generally performed by the analyst at the time the problem occurs. Long term corrective actions are decided upon and monitored by the Environmental Toxicology team through the use of the occurrence management form. Occurrence management forms will be used for problems such as two consecutively failed RTTs, failed PT, long-term poor culture performance, failed equipment or supplies that affect testing performance, repeated failures in test control organisms, amended reports, etc.

4.11.7. Departures from Documented Policies and Procedures

Departures from documented policies and procedures will be discussed by the Environmental Toxicology team. The departure, the reason for the departure and the result of the departure will be noted in the

permanent lab log. If the departure has an impact on test results, the client will be contacted. Departures from normal testing methods will be noted on the final report to the client.

4.12. Quality Control Limits Procedures

4.12.1. Reference Toxicity Testing (RTT)

To ensure the health and sensitivity of the test organisms, reference toxicity testing (RTT) is performed monthly. Sodium chloride at concentrations appropriate to the organism is used as a reference toxicant for the following test systems: *P. promelas* - acute and chronic WET, *C. dubia* - acute and chronic WET, and *S. capricornutum* - chronic WET. RTT's are conducted like regular tests in regards to organism age, pretest requirements, test conditions, etc. Acute and chronic RTT endpoints are entered on control charts for each species in a Microsoft 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 RTT graph plots mean, and plus/minus two standard deviation lines. These graphs are maintained using floating standard deviation control limits (the control limit for each data point reflects the control limits calculated from the 20 preceding data points). Data points outside of the two standard deviation lines are considered "out of control" and a quality assurance violation. Data points outside of the control limits are also tested to determine if they are statistical outliers using Grubbs test. If they are confirmed to be statistical outliers then they are not included in the control limit calculations for future tests. After an RTT fails to meet control limits, corrective measures for culture conditions are discussed and implemented, if necessary. Those reference tests outside of control limits are repeated immediately. If two consecutive reference tests are outside of control limits, effluent testing is suspended until reference test data are within control limits.

Monthly reference toxicant tests with sodium chloride are used to determine organism performance and acceptability for use in toxicity testing. RTT data is entered monthly into a test report to ensure all quality control measurements are acceptable. 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 (ICp) to calculate an inhibition concentration for 25% of a population (IC25). *Selenastrum capricornutum* chronic test data is calculated using the same software with an IC50 being calculated.

4.12.2. Table 3. Acceptance Criteria for *Ceriodaphnia dubia* Pretest Culture Conditions

Ceriodaphnia dubia	
Average number of neonates in 3 broods	≥ 15 acute; ≥ 20 chronic
Mean survival	≥ 80%
Number of neonates in each brood used	≥ 8
Age of organisms	≤ 24 hours

4.12.3. Table 4. Requirements for Acute and Chronic WET Test Acceptability

Assess the quality control parameters for the primary (dilution water) control first and if it fails to meet criteria then assess the secondary (lab water) control. A test is considered unacceptable and must be repeated (within 30 days of the original test's end) when both controls experience a failure of one or more of these test acceptability criteria.

Acute and Chronic QC Requirements – WET and RTT Tests	
Acute Test – FHM and C.dubia	
Control survival	≥ 90%
Chronic - Ceriodaphnia dubia	
Control survival	≥ 80%
Control variability	≤ 40% CV (SD/mean) between replicates, excluding young from dead adults
Male production	≤ 20% in controls and ≤ 20% over all concentrations
Mean control production	≥ 15 neonates per surviving female in 1st 3 broods and ≥ 80% surviving females produce 3 broods
Chronic - Pimephales promelas	
Control survival	≥ 80%
Control variability	≤ 40% CV between replicates, excluding dead adults
Mean control biomass	≥ 0.25 mg / surviving fish – dry weight only, excluding dead adults
Selenastrum capricornutum	
Control performance	> 1x 10^6 cells/ml cell density at end of test
Control variability	≤ 20% CV between control replicates

4.12.4. Table 5. Chemistry Quality Control Samples

Parameter	QC Sample	Frequency	Criteria
Dissolved Oxygen	Calibration	Daily calibration	Calibration DO ± 0.5 mg/L of theoretical DO
	Duplicate	Every 10 samples	≤ 10% RPD
pH	Calibration	Daily calibration	Calibration slope 92-102% Check standard 7.0 ± 0.05 s.u.
	Duplicate	Every 10 samples	± 0.20 s.u.
Conductivity	Calibration	Daily calibration	Conductivity ± 5% of 1,413 µmhos cm^{-1} (1,342 – 1,484µS cm^{-1}) Cell constant must be 1.0 ± 15% (0.85 – 1.15)
	Duplicate	Every 10 samples	≤ 10% RPD
Total Residual Chlorine	Duplicate	Every 20 samples or once per run if < 20	≤ 10% RPD
	Known Standard	Every 20 samples or once per run if < 20	± 10% of true value
	Method blank	Each analysis day	< LOD
Alkalinity	Duplicate	Every 20 samples or once per run if < 20	≤ 10% RPD
	Known Standards (Two Known standards of different sources)	Two different standards initially, 2nd Known after 20 samples	± 10% of true value
	Method Blank	Each analysis day	< LOD of 17 mg/L
Hardness	Duplicate	Every 20 samples or once per run if < 20	≤ 10% RPD
	Known Standards (Two Known of different sources)	Two different standards initially, 2nd Known after 20 samples	± 10% of true value (1,000 mg/L CaCO3)
	Method Blank	Each analysis day	< LOD of 6.7 mg/L
	Matrix Spike	Every 20 samples	85 - 115% recovery

4.13. Internal and External PT Studies

4.13.1. Proficiency testing

Proficiency testing (Discharge Monitoring Report - DMR) samples are performed once yearly for *P. promelas* and *C. dubia* acute and chronic tests to examine inter-laboratory performance. DMR samples are processed and tested in the same way effluent samples are handled. The main exception is that DMR test dilutions and conditions must follow the PT provider's instructions to ensure comparable inter-lab

results and acceptance limits. DMR testing is carried out independently and reported according to the directions from the PT provider. The DMR-QA normally occurs in May - July of each year. DMR tests are currently not available for *Selenastrum capricornutum*. DMR tests in which the laboratory controls do not pass criteria are repeated and occurrence reports are filed. An internal PT sample called a reference toxicant test (RTT) is analyzed monthly (except on months when the DMR-QA proficiency test sample is analyzed) for each test type and repeated if they do not meet criteria. See Section 4.12 for more information about RTT assessment.

4.13.2. Continued Demonstration of Capabilities

Demonstrations of Capabilities are completed annually by each analyst. Due to the nature of toxicity testing, a test method (analysis) is the result of work completed by multiple analysts. It is very rare for a single test to be completed entirely by one analyst. Therefore, individual demonstrations of capabilities are determined by gathering work done by an analyst on a number of tests. Only analyses with passing laboratory controls will be used to satisfy demonstration of capability requirements. The components of each test method are separated and each component must be completed by an analyst on separate days of analysis. There are three components (set up, renewal and shut down) for the acute and chronic fathead minnow toxicity tests and also for the acute and chronic *Ceriodaphnia dubia* toxicity tests. There are two components (set up and shut down) for the algae test. An annual demonstration of capability for toxicity testing requires a total of five toxicity test methods, one replicate each, to be completed satisfactorily. Multiple categories (set up, renewal, shut down) can be satisfied from the same test.

4.14. Internal Audits

See section 1.20 of this QA Manual for division wide information on internal audits.

An annual internal method audit is performed during the first quarter of the following calendar year. The audit covers the entire preceding year and is conducted following the standards outlined by NELAC and the WDNR accreditation requirements.

The internal method audit reviews one report from each method accredited under NELAC and the WDNR. Each test method is reviewed for accuracy and suitability and current analyst certification statement. The completion of test request forms and test bench sheets is reviewed. The report is reviewed for completion, accuracy of the data reported and verification the correct statistical data package was used. QC logbooks are reviewed along with chemistry spreadsheets. Analysts training files are reviewed to ensure they are up to date with annual sign offs and DOCs are complete. Deficiencies will be noted in the audit report.

The Environmental Toxicology team will determine the appropriate corrective action to be taken. A completion date for the corrective action will be determined. The corrective action, the completion date and the responsible person will be recorded on the internal audit. Audit findings will be reviewed at lab meetings until completed. Follow up of corrective action plans will be done as part of the following annual audit. A system audit, using the checklists provided by the accrediting bodies may also be performed as an annual internal audit.

Prior to an on-site inspection by an accrediting agency, the corresponding checklist for the laboratory will be reviewed in preparation.

All internal audits and checklist reviews will be done by the QC Coordinator appointed to that department.

4.15. Traceability of Measurement

See section 1.13 of this QA Manual for division wide information on traceability.

Also see section 4.10 for details on records related to traceability. Standards and reagents of required purity are obtained from various suppliers including Sigma Aldrich, Acros, Chem Service, Fisher Scientific, Ricca Chemical, Hach, LabChem, Airgas, Scott Specialty Gases, UltraScientific, VWR, UW MDS, and others. When available, these chemicals are certified and traceable to the National Institute of Standards and Technology (NIST). When supplied, the manufacturer's certificates of analysis are kept on file. Commercially obtained standards and reagents may be pure materials or ready to use solutions. See Section 4.10 for more information on purchasing of standards and reagents.

4.16. Method References

See section 1.25 of this QA Manual for division wide information on method references.

- AOAC "Official Methods of Analysis of the Association of Official Analytical Chemists", 15th edition, Association of Official Analytical Chemists, P.O. Box 540, Washington, D.C. 20044, (1990)
- APHA et al., "Standard Methods for the Examination of Water and Wastewater", 20th ed., American Public Health Association, 1015 Fifteenth Street NW, Washington D.C. 20005, (1998).
- APHA et al, “Standard Methods for the Examination of Water and Wastewater”, 16th edition.
- American Public Health Association, Washington, DC, Mount, D.R. and D.I. Mount. "A Simple Method of pH Control for Static and Static-Renewal Aquatic Toxicity Tests". Environ. Toxicol. Chem. 11:609-614 (1992).
- WDNR, "Guidance Manual for the Certification and Registration of Laboratories Conducting Effluent Toxicity Tests". PUBL-TS-006 91 (1991).
- WDNR, "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual", 2nd Edition. PUB-WT-797 (2004).
- USEPA, "Handbook for Sampling and Sample Preservation of Water and Wastewater". EPA 600/4-82/029 (1982).
- USEPA, "Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms" (Fifth Edition). U.S. Environmental Protection Agency, Office of Water (4303T) Washington, DC. EPA-821-R-02-012. October (2002).
- USEPA, "Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms" (Fourth Edition). U.S. Environmental Protection Agency, Office of Water (4303T). Washington, DC. EPA-821-R-02-013. October (2002).

4.17. Standard Operating Procedures

See section 1.11 of this QA Manual for division wide information on standard operating procedures. Also see Section 4.3 for more specifics on standard operating procedures.

A complete list of departmental standard operating procedures can be found in the table of contents (TOC) found in the following directory: O:\SOP\EHD\ESS\Enviro Organic\Enviro Tox\Final.

The table of contents includes unique SOP identifiers, title, revision number, and effective date. There are hyperlinks in the TOC to the actual documents.

4.18. Accredited Methods (EPA, WDNR)

4.18.1. Non-Potable Water

Regulatory Method	**Description**	**WSLH Method #**	**Accreditation**
EPA 2000	Acute WET test for *Pimephales promelas*	ESS ENV TOX METHOD 4200	NELAC WDNR
EPA 2002	Acute WET for *Ceriodaphnia dubia*	ESS ENV TOX METHOD 4201	NELAC WDNR
EPA 1000	Chronic WET test for *Pimephales promelas*	ESS ENV TOX METHOD 4202	NELAC WDNR
EPA 1002	Chronic WET test for *Ceriodaphnia dubia*	ESS ENV TOX METHOD 4203	NELAC WDNR
EPA 1003	Chronic WET test for *Selenastrum capricornutum*	ESS ENV TOX METHOD 4204	WDNR

Water Microbiology

Table of Contents

5.1. Personnel

Table #: Education and Experience

Name	**Title**	**Degree**	**Yrs Exp.**
Jocelyn Hemming	Environmental Microbiology and Toxicology Manager	BA Biology, PhD Environmental Toxicology	21
Martin Collins	Senior Microbiologist	MS, Biology	21
Shannon Johnson-Windsor	Microbiologist	MS, Microbiology and Biology	8
Olivia Feider	Associate Microbiologist	BS, Env Public Health	2
Jason Janzen	Associate Microbiologist	BS, Conservation Biology and Geosciences	6
Paige Mullen	Associate Microbiologist	BS, Applied Science – Biochemistry and Molecular Biology	2
Zachariah Zopp	Microbiologist	MS, Water Resources Management	9
Kayley Janssen	Microbiologist	Ph. D Microbiology	12

5.2. Training

Initial Training for New Employee

The departments use all the checklists and all the safety procedures described in the general chapter (Section 1.10.1) except for the Radiation Safety Training to train a new employee. The new employee is also required to read Section 1090 in *Standard Method for the Examination of Water and Wastewater,* 22nd Ed., "Laboratory Occupational Health and Safety" and shown the location of Lab-wide SOPs and Divisional SOPs. The employee dates and initials each item in the annual review checklist as part of the initial training.

O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO FORM 171 Annual Review Sign Off.docx
O:\SOP\EHD\EHD Glassware Media\Final\Bench Sheets\EHD GLASSWARE MEDIA FORM 107 Annual Review Sign Off.docx

The Water Microbiology department also uses the procedure described in the general chapter (Section 1.10.2) for the analytical training of a new employee. An experienced analyst will date and initial the water microbiology training checklist after observing the new employee performing the procedure.

O:\SOP\EHD\ESS\Water Micro\Final\Bench sheets\ESS MICRO FORM 105 Analyst Training Checklist.xls

The employee will perform an initial demonstration of capability (DOC) according the Divisional procedure: O:\SOP\EHD\Division Wide\Final\EHD QA 115 rev 0_DOCs.docx A demonstration of capability (DOC) form is prepared for each method with acceptable results. ESS MICRO FORM 700 Demonstration of Capability Certification Statement is used and located here: O:\SOP\EHD\ESS\Water Micro\Final\DOC\Demonstration of Capability Certification Statement. DOC records are recorded on O:\SOP\EHD\ESS\Water Micro\Final\DOC\EHD QA 115_DOC CALC FORM_rev 5.xls and stored here: M:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\DOC documentation.

All checklists are kept in the employee's training/annual review file and records are recorded here: M:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\Annual Training & SOP Review.

Continued Training

The Water Microbiology department uses the procedure described in the general chapter (Section 1.10.3). Each employee is also required to read Section 1090 in *Standard Method for the Examination of Water and Wastewater,* 22nd Ed., "Laboratory Occupational Health and Safety". The employee dates and initials each item in the annual review checklist as part of the training. O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO FORM 105 Analyst Training Checklist.xlsx

Each analyst will perform a demonstration of capability (DOC) according to the Divisional GENOP: A demonstration of capability (DOC) form is prepared for each method with acceptable results.

Each employee is also required to read Section 1090 in *Standard Method for the Examination of Water and Wastewater,* 22nd Ed., "Laboratory Occupational Health and Safety". The employee dates and initials each item in the annual review checklist as part of the training. O:\SOP\EHD\EHD Glassware Media\Final\Bench Sheets\EHD GLASSWARE MEDIA FORM 107 Annual Review Sign Off.docx. The checklists are kept in the employee's training/annual review file.

5.3. Document Control System

The information described in the general chapter (Section 1.11) is used by the department for document control. The specific GENOP describing the writing and document control for the Water Microbiology Department is located: O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO GENOP 002 rev 1_SOP writing & doc control.doc

The official revision of a SOP and forms used are kept in the final folder. SOPs being revised or written are located in the draft folder. Revisions of SOPs no longer being used are placed in the archive folder. The signature page for retired SOPs is scanned in and placed in the archive folder. If the SOP contained handwritten revisions, the entire SOP or the specific pages are scanned and archived as well. All SOP folders are located: O:\SOP\EHD\ESS\Water Micro . A hardcopy of the current revision of a SOP with the "Analysts Certification Statement" signed by each analyst is kept in a 3-ring binder in the lab.

A list of external documents referenced in SOPs is in an Excel™ Spreadsheet with the location listed and for electronic documents a link to the document. O:\SOP\EHD\Division Wide\Draft\In Progress\TOC External Documents.xlsx When there are changes to the spreadsheet, a new revision of the spreadsheet is given. The old spreadsheet is archived for 6 years. External documents are typically stored here: O:\SOP\EHD\ESS\Water Micro\Final\External documents

5.4. Records Retention, Control, and Storage

The department follows the described procedure in the general chapter (Section 1.12), except the department does not send records to the State Record Storage. After 6 years and 10 years for research, the records are shredded onsite. The records are kept with like records in file cabinets and shelves in the walk-in incubator. Some of the QC is kept securely on the M Drive or associated with the samples in Horizon. The information is deleted after 6 years or after 10 years for research. After 6 years or 10 years the department uses the lab wide GENOP 1002 for immediate destruction of records.

5.5. Sample Handling Procedures

The department uses the procedures described in the general chapter (Section 1.16) for sample handling. Since microbiology samples have short hold times, the samples are setup the same day they are received. There are not any special handling procedures for Enforcement Samples. Since the collection container is also the testing container, the entire sample volume is used in the analytical process for most methods and the containers are disposed of at the end of testing. Along with the analytical test method SOPs, the sample acceptance GENOP 112 determines if a sample may be tested. O:\SOP\EHD\ESS\WaterMicro\Final\ESS MICRO GENOP 112 rev 2_Sample Acceptance.doc

The GENOPs 411 and 420 describe the procedures for capturing the date/time when samples are put into the incubator and taken out of the incubator, the analyst, QC data associated with the sample and the disposal of the sample. O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO GENOP 411 Chemware Process for Anaytical Testing.docx O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO GENOP 420 rev 1_Processing Samples when Chemware is Non-Operational.doc

5.6. Data Review and Reporting

The department uses both manual and electronic ways of data review. There are specific queries written in Horizon to electronically verify the correct holding times for public water supply total coliform samples. At the end of the day the "WSLH End of the Day 30 hr report " is run to assure all the samples are put into the incubator within 30 hours. Horizon automatically flags any sample result not within the correct holding time or incubation time. Another analyst reviews all unsafe drinking water samples, MPN by Quanty-tray samples, and agar medium/broth samples every business day. On weekends the analyst reviews their own data. Once the data have been reviewed a report is automatically generated from Horizon and e-mail, faxed and/or electronically sent to the DNR with the exception of *Cryptosporidium/Giardia* samples. *Cryptosporidium/Giardia* sample bench sheets and the manually generated report are reviewed by the supervisor or another analyst for accuracy. Once the data has been reviewed, the manually generated report is mailed to customer. The SOPS detailing data review and reporting are in SOPS: O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO GENOP 411 Chemware

Process for Anaytical Testing.docx and O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO GENOP 412 rev 3_Crypto & Giardia Data Analysis.docx

5.7. Procedures for Accepting New Work/Review of Requests, Tenders and Contracts

The department follows the described procedure in Section 1.15 in the general chapter for accepting new work/review of requests, tenders and contracts.

5.8. Laboratory Facilities

The department is approximately 1100 square ft. for total coliform, *E. coli,* and other non-pathogenic testing, 430 square ft. for pathogen testing, and separate molecular, glassware washing and media preparation rooms. The analytical work is performed in Room 203. This room has the following support equipment: -80°C freezer; variable temperature incubators; refrigerators for media, samples and *Cryptosporidium/Giardia* reagents and stains; centrifuge, water baths, a light microscope, and Quanti-Tray™ sealers. The total coliform and *E. coli* samples are incubated in Room 203C. The pathogen testing, where *Cryptosporidium/Giardia* and other pathogens are performed, is located in Room 202. This room has the following support equipment: biological safety cabinet, fume hood, peristaltic pumps, and wrist shaker. The Dark Room is Room 202B for the brightfield and fluorescent microscope, Biolog 96 well plate reader (Biolog Micro Station), Biolog turbidimeter, Nanodrop, Qubit. The molecular testing is performed in Rooms 108, 100A and 100C. Room 108 is used for mastermix preparation with the following support equipment: PCR dead air box and a -20°C freezer, for reagents; Room 100A is for adding samples to the master mix with the following support equipment: freezers, refrigerator, biological safety cabinet and table top centrifuges: Room 100C is for performing molecular analysis with amplicons using the following support equipment: ABI 7500 Fast, ABI StepOnePlus, Bio-Rad QX200 droplet digital PCR, biological safety cabinet, refrigerator, -20°C freezer. Room 117 is for Photodyne transilluminator and camera system

General cleaning and preventative equipment measures are done weekly by department analysts, generally on Mondays. These activities are documented on ESS MICRO FORM 107 and are as follows:

Clean and disinfect water bathes as needed (203-8/203-9)

Clean Walk-in Incubator (203-1)

Check and clean UV Lamps (203UV1/UV2) and Quanti-tray sealers (203 QT1/QT2)

Clean Rm 203 benches, sinks, and carts

Check volumes of Jug Buffer, ethanol bottles, iodine disinfection solution

Cleanout sample fridge (203-6)

Empty Jug Buffer waste

Stock paper towels and sample bottles; check Aux data lot numbers

Flush eyewashes, remove contaminated waste bags

Restart Computers, check Pipetman charge

Change conductivity probe solution (RO water), change pH meter storage solution

Restreak stock cultures (every other week)

Review Periodic QC list, download COAs (as needed)

Check Media Inventory (O:\SOP\EHD\ESS\Water Micro\Final\Bench sheets\ ESS MICRO FORM 136 Media Inventory Tracking)

Quality Control is performed by every analyst and analyst check to make sure all QC is performed in a timely fashion. These duties are listed in:

O:\SOP\EHD\ESS\Water Micro\Final\Bench sheets\ESS MICRO FORM 107 QC check off sheet.xlsx

All the rooms have computers for analytical testing (Horizon and PCR software) and media preparation (media SOPs/spreadsheets). 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 the 65°F to 80°F range. Work benches are cleaned with disinfectant.

Each analyst has an assigned cubicle area with a computer for any work that can be performed in a clean area.

5.9. Instrumentation and Equipment

A complete electronic list of all the equipment, pipettes, and their descriptions used by the department is updated annually and is available in the following folder: M:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\Equipment

Thermometers

All thermometers for refrigerators, incubators and freezers are digital NIST-certified. Thermometers in circulation are replaced with NIST calibrated thermometers or calibrated on an annual basis. If thermometers are calibrated in-house, the monitoring temperature must calibrated with two bracketed temperatures per EPA guidelines. Temperatures from these units are recorded all working weekdays twice with at least 4 hours in between readings and once on weekends. These digital thermometers have minimum/maximum temperature readings for weekends and holidays when temperatures can't be manually recorded twice per day with at least 4 hours in between. .

The following SOPS describe taking temperatures and calibration of thermometers:
O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 200 Temperature Monitoring.doc

Refrigerators & Freezers

In the event of a need for service or repair, refrigerators and freezers are maintained by CRM.

Centrifuges

Centrifuges are kept under a service contract and are serviced and calibrated annually. The centrifuge rotors are calibrated annually and the filter is changed quarterly by analyst as needed. Maintenance records are maintained by the supervisor in hard copy or electronically here: M:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\Equipment\Centrifuges.

Microscopes

Microscopes are cleaned once a year under a microscope maintenance agreement. Maintenance records are maintained by the supervisor in hard copy or electronically here: M:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\Equipment\Microscopes.

Gelman Membrane Filtration System, (located in Room 203)

The components of these units are made of polysulfone. The funnels and bases 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 sanitized for a minimum of 2 minutes.

UV Sterilization — Millipore UV sterilizers, (located in Room 203)

The performance of the sterilizer is checked quarterly using O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 230 rev 6_UV Sterilizer.docx

Biological Safety Cabinets

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 units are 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. The pressure gauge is checked before each use for air flow.

Fume Hoods

UW Environmental Health Services annually certifies the units.

Quantitray Sealers

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.

O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 214 rev 4_Quanti-Tray Sterility Check.doc

O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 218 rev 2_Quanti-Tray Sealer Check.doc

Thermal Cyclers

7500 Fast Real-Time PCR System - There is a maintenance contract with Life Technologies for the instrument.

Monthly, the lamp status is check, the background calibration is checked, and disk cleanup and disk defragmentation is performed.

Semiannually, the lamp status is checked, the regions of interest calibration are performed, the background calibration is performed, the optical calibration is performed, and all pure dye calibrations are performed.

As needed, the instrument is decontaminated, the halogen lamp is replaced, the instrument fuses are replaced, the windows operating system is updated and the 7500 software is updated.

Laboratory Reagent Grade Water

Reverse Osmosis (RO) Water – Type II (See General Section 1.17.2) and Elga water polisher (Type I). All lab water QA/QC are stored electronically in this location in the M Drive: M:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\Lab Water\Lab Water Chemistry QC Forms. Conductivity, pH, ammonia, nitrate, total organic carbon and total residual chlorine determination are performed at least monthly; conductivity meter is calibrated once a day as needed for the use of sterile type I water and is used as dilution water for sample analysis. The meter is calibrated with a 10 microseimen standard and the calibration is verified with a 5 microseimen standard.

Conductivity calibration is recorded on Form 114. Calibration charts can also be found: O:\SOP\EHD\ESS\Water Micro\Final\ ESS MICRO FORM 114 Conductivity Log

Metals analysis for Cd, Cr, Cu, Ni, Pb, Zn and Silicate is performed on an annual basis and the results recorded on ESS Form 135 located here: O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 222 rev 4_Monitoring Lab Water for Metals, Nutrients and TOC.docx.

O:\SOP\EHD\ESS\Water Micro\Final\Bench sheets\ESS MICRO FORM 135 Laboratory Water Chemistry QC

Heterotrophic Plate Counts are performed by the pour plate method monthly. The results recorded on ESS Form 135 and stored electronically here: O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO METHOD 304 rev 6_Heterotrophic Plate Count (HPC).docx.

Chlorine Residual Test is conducted monthly using colorimetric method (See Method ESS Micro QA/QC SOP 260) and results are recorded on ESS Form 134 and stored electronically by year M:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\Sodium thiosulfate\Chlorine.

O:\SOP\EHD\ESS\Water Micro\Final\Bench sheets\ESS MICRO FORM 134 Chlorine worksheet template

O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 260 rev 5_Lab Water Chlorine Standards & Testing and Sodium Thiosulfate Neutralization in Sample Bottle.docx

Computers

The department follows the procedures in the general chapter (Section1.17.4).

Minor equipment and miscellaneous equipment are listed in the method SOPs.

5.10. Laboratory Supplies and Chemicals

The departments keep an inventory of supplies and chemicals including the name of vendor in spreadsheets.

The Water Microbiology spreadsheets are located here: M:\EHD\ESS(4900)\ESS Micro(4920)\Purchasing and Supplies\WM Order list.xlsx

The quality of the reagents used by the Water Microbiology are either in the technical SOP or in the following SOPs:

O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 202 rev 5_Enzymatic Substrate Quality.docx

Three bottles from each new lot of sample bottles/Quanti-Trays received from a vendor or prepared internally are randomly selected and checked for sterility and fluorescence bottles. The volume is checked on each lot of bottles received. The results are recorded in the QC logbook in the Water Microbiology Department. O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 212 rev 6_Sample Bottle Sterility Calibration Fluorescence.docx .

Bottles received with sodium thiosulfate are checked with diluted bleach (15 ppm free chlorine) to verify the sodium thiosulfate will neutralize at least 15 ppm of chlorine. O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 260 rev 5_Lab Water Chlorine Standards & Testing and Sodium Thiosulfate Neutralization in Sample Bottle.docx. Results are recorded on ESS Form 134 (see Section 5.9, Laboratory Water). The bottle checks are performed before bottles are used or sent to clients.

5.11. General Quality Control Procedures

Analytical SOPs are written in enough detail so that an analyst can clearly understand and follow the procedures. In addition, each SOP includes the specific quality control criteria and references to the

applicable authoritative source. In the case of a non-regulated "in-house" method, references to scientific papers are used, or modification to authoritative methods are included.

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, DATCP, 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.

Quality Control Data Gathering and Documentation

All QC data that affects the testing is recorded into QC logbooks, QC log sheets, or directly into Horizon as a comment or as an assigned QC for the test. Positive and negative performance checks of media and reagents are recorded before use. If the correct reactions are not observed, the media and reagents will not be used. If any matrix spikes, matrix spike duplicates, method blank, IPR (initial precision and recovery) or OPR (on going precision and recovery) testing fails established QC criteria for *Cryptosporidium/Giardia*, new samples will be requested and a new method blank, matrix spike, matrix spike duplicates, IPR and OPR testing will be performed after making any corrections. Graphs of the last 20 samples from OPRs and matrix spikes are maintained. If any results are out of bounds, corrective actions are investigated. Equipment operation is checked at various intervals.

All definitions or QC checks are either part of the analytical SOP or QA SOP located in the methods final folder. O:\SOP\EHD\ESS\Water Micro\Final

Corrective and Preventive Action Procedures

The departments follow the procedures described in the general chapter (Section 1.21).

Corrective actions are taken if there is a QC failure which is described in the analytical SOPs. The corrective actions are taken when an analyst notices a problem with a procedure, (e.g., incorrect incubation time, incubation temperature out of range) or equipment (e.g., Quanti-Tray sealer not sealing). If QC failure involves samples, the client is notified by telephone, e-mail, fax, or US mail. The sample may be invalidated or if the client wants the results, the results are flagged. The result is flagged by adding a comment in Horizon either in the analytical batch or as a sample comment. The SOP describing the procedures is: O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO GENOP 411 Chemware Process for Analytical Testing.docx The corrective action is documented in the appropriate logbook, Horizon or in an Occurrence Management Report, which is located on the internal website. The corrective action will include the QC failure, cause of the failure, specific corrective actions and if the corrective actions worked and were implemented. The Occurrence Management SOP describes the procedure: O:\SOP\Labwide SOP Policies\Final\LABWIDE_GENOP_706_Occurrence_Reporting_Procedure.doc

The unit institutes preventive actions including having maintenance agreements on equipment, routine monitoring of equipment, performing QC on media and reagents, preforming QC samples, DOCs, and

conducting internal and external audits. The specific SOPs are located in the final folder. The department works with other departments, WSLH teams, other governmental agencies, external auditors and internal staff to improve processes.

Departures from Documented Policies and Procedures

The department follows the procedure described in the general chapter (Section 1.21.3) for departures from documented policies and procedures.

5.12. Quality Control Limits Procedures

The characteristics of data that measure accomplishment of a specified purpose can be expressed in terms of representativeness, comparability, precision, accuracy and completeness. Analytical SOPs have specifics on quality control limits used by the department. Analytical methods with present/absent results give the percentage of false negatives and false positives.

Representatives

- 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.

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.

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."

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.

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.

5.13. Internal and External PT Studies

The Water Microbiology Department purchases external performance evaluation (PE) or proficiency testing (PT) samples approximately once every 12 months for every certified method where PE/PT samples are available. The PE/PT samples are assigned randomly among trained analysts. The PTs are performed in house with no contact with other labs or provider of studies. The PE/PT samples are given laboratory numbers in Horizon and results are entered into Horizon. Once the results are final in Horizon a report is sent electronically to the supervisor and a paper copy is kept in a filing cabinet in the supervisor's office.

The electronic results from the PE/PT vendor for PE samples or PT samples are located in the following folder: M:\EHD\ESS(4900)\ESS Micro(4920)\ QAQC\Proficiency Results

If there is a failure, an Occurrence Report is generated and the cause investigated. Any failing results are e-mailed to the QA office (i.e., Camille Danielson, Lisa Berkan).

Every analyst in the Water Microbiology Department annually performs DOC (Demonstration of Capability) testing on every method the analyst is certified to perform. The analyst uses quality control samples from IDEXX or flow counted organisms from ATCC culture strains maintained for quality control. The analysts are given blind cultures or samples to test made by a supervisor for each potable water or non-potable water method. Methods where numbers are required, the results are considered acceptable if the analyst's results are within 2 standard deviations when compared to all the other analyst results or within the 95% confidence level for most probable number methods and colony counts. The DOC for *Cryptosporidium/Giardia* is an OPR with acceptable recovery according to the method. If the analyst's 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. PE/PT samples may also be used for DOCs.

New analysts are not allowed to perform testing until they have acceptable results with an Initial Precision Recovery (IPR) for each method. The following describes the DOC and IPR procedures in more detail:

O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO GENOP 010 rev 1_Inital & Ongoing DOC.doc

The results for the DOCs are located in the following folder: M:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\DOC documentation

External PT Programs Schedule

Program Name	Method/Technology	Micro SOP #	Schedule
Water	SM9222D – Fecal Coliform	SOPs 310, 314	March

Pollution Control Micro – Phenova (non-potable water)	SM9223B (Colilert/Colilert-18 Quanti-tray) – Total Coliform/*E. coli* Fed Reg. Enterolert- *Enterococci*	SOP 300 SOP 356	
Water Supply Micro – Phenova (potable water)	SM9221 B + F – Total Coliform/*E. coli* Fed Reg., Readycult – Total Coliform/*E. coli* SM9223B, Colilert/Colilert-18 - Total Coliform/*E. coli* Fed Reg, Colitag - Total Coliform/*E. coli* SM9223 B, Colisure - Total Coliform/*E. coli* SM9215 B, PCA– HPC Fed Reg., Simplate – HPC MPN **Source Water** SM 9223 B, Colilert/Colilert-18 - Total Coliform/*E. coli* SM9223 B , Colisure - Total Coliform/*E. coli*	SOP 302 SOP 360 SOP 300 SOP 301 SOP 300 SOPs 304 & 305 SOP 312 SOP 300 SOP 300	March
WI Proficiency Program (potable water)	SM9221 B + F – Total Coliform/*E. coli* Fed Reg., Readycult – Total Coliform/*E. coli* SM9223B, Colilert/Colilert-18 - Total Coliform/*E. coli* Fed Reg, Colitag - Total Coliform/*E. coli* SM9223 B, Colisure - Total Coliform/*E. coli*	SOP 302 SOP 360 SOP 300 SOP 301 SOP 300	March
Cryptosporidium/Giardia PT (source water)	EPA 1623 – *Cryptosporidium/Giardia*	SOP 600	April, October

Internal PT Programs Schedule

QC type	Method/Technology	Micro SOP #	Schedule
WI Proficiency Program (QA	SM9223B, Colilert/Colilert-18 - Total Coliform/*E. coli* SM9223 B, Colisure - Total Coliform/*E. coli,*	SOP 300 SOP 300	March

reference)	Fed Reg., Colitag – Total Coliform/*E. coli* Fed Reg., Readycult - Total Coliform/*E. coli*	SOP 301 SOP 360	
DOCs – Surface Water (non-potable water)	SM9222D – Fecal Coliform SM9223B (Colilert/Colilert-18 Quanti-tray) – Total Coliform/*E. coli* Fed Reg. Enterolert- *Enterococci*	SOPs 310, 314 SOP 300 SOP 356	December/January (may vary by analyst but will be performed annually)
DOC – Drinking Water (potable water)	SM9221 B + F – Total Coliform/*E. coli* Fed Reg., Readycult – Total Coliform/*E. coli* SM9223B, Colilert/Colilert-18 - Total Coliform/*E. coli* Fed Reg, Colitag - Total Coliform/*E. coli* SM9223 B, Colisure - Total Coliform/*E. coli* SM9215 B, PCA– HPC Fed Reg., Simplate – HPC MPN **Source Water** SM 9223 B, Colilert/Colilert-18 - Total Coliform/*E. coli* SM9223 B , Colisure - Total Coliform/*E. coli*	SOP 302 SOP 360 SOP 300 SOP 301 SOP 300 SOPs 304 & 305 SOP 312 SOP 300 SOP 300	December/January (may vary by analyst but will be performed annually)

5.14. Internal Audits

A systems internal audit will be conducted by divisional QA staff or a supervisor annually.

Internal audits will be performed on each analytical method on an annual basis. These audits will be performed by the department QA Officer or certified analysts.

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 checklists from EPA 815-B-97-001 including the supplemental Cryptosporidium/Giardia checklist. The auditor will give the checklist to the supervisor for the water

microbiology department with suggested corrections. The paper copy is kept in a filing cabinet in the supervisor's office. . The Departments will make the corrections in a reasonable time frame.

After all the audits are performed the supervisor will write the Management Review for the departments.

Method	**WI SOP**	**Audit Schedule**
SM9222D – Fecal Coliform SM9223B (Colilert/Colilert-18 Quanti-tray) – Total Coliform/*E. coli* Fed Reg. Enterolert- *Enterococci*	SOPs 310, 314 SOP 300 SOP 356	End of December/January
SM9221 B + F – Total Coliform/*E. coli* Fed Reg., Readycult – Total Coliform/*E. coli* SM9223B, Colilert/Colilert-18 - Total Coliform/*E. coli* Fed Reg, Colitag - Total Coliform/*E. coli* SM9223 B, Colisure - Total Coliform/*E. coli* SM9215 B, PCA– HPC Fed Reg., Simplate – HPC MPN **Source Water** SM 9223 B, Colilert/Colilert-18 - Total Coliform/*E. coli* SM9223 B , Colisure - Total Coliform/*E. coli* EPA 1623 – *Cryptosporidium/Giardia*	SOP 302 SOP 360 SOP 300 SOP 301 SOP 300 SOPs 304 & 305 SOP 312 SOP 300 SOP 300 SOP 600	End of December/January

5.15. Traceability of Measurement

The department follows the procedure described in the general chapter (Section 1.13)

Standards, Commercial Media, Chemicals, Reagents and Dyes

All standards, commercial media, chemicals, reagents and dyes are dated when received to monitor the shelf life. They are disposed of when the shelf life of the manufacturer is exceeded unless the expiration date can be reliably extended through method verifications and documented. All chemicals for

microbiology are ACS (American Chemical Society) or AR (Analytical Reagent) grade. All dyes for microbiology are certified by the Biological Stain Commission for bacteriological use. A certificate of analysis is kept in an electronic format for the water microbiology department at: M:\EHD\ESS(4900)\ESS Micro(4920)\ QAQC\Certificate of Analysis. A paper copy is kept for the media/glassware department in a binder located in the media room.

M:\EHD\ESS(4900)\ESS Micro(4920)\Purchasing and Supplies\Water Micro Chemical Inventory

M:\EHD\ESS(4900)\ESS Micro(4920)\Purchasing and Supplies\WM Order list.xlsx

Dry Powder Media

Once a bottle is opened an expiration date of 6 months is given to the Water Microbiological medium. The NELAC auditor approved the medium does not have to be discarded if the lab can show the medium is still working correctly after six months. The lab compares bottles of commercial media opened more than six months with media opened less than six months. If the counts fall within 2 standard deviations, the media may be used for analysis for another six months or until the manufacturer's expiration whichever is longer. For presence/absence media the media must recover equal to or less than 10 organisms after six months. The media is disposed of when the shelf life of the manufacturer is exceeded. R:\EHD\ESS(4900)\ESS Micro(4920)\QAQC\\Water and Media T-Test Results

Prepared Media

The media is prepared to the recipes located in the media recipe folder. O:\SOP\EHD\EHD Glassware Media\Final\Media Recipe SOP. The prepared media recipe/bench sheet has the manufacturer's name and lot numbers of reagents, chemicals and powders used in the preparation. The prepared media is given a lot number of the date prepared followed by the media expiration date in the following format: mm/dd/yy – mm/dd/yy.

Analytical Traceability

The equipment and media used in analytical testing are documented in Horizon in the Aux Data field according to the following SOP: O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO GENOP 411 Chemware Process for Analytical Testing.docx. Analytical testing requiring a bench sheet has the information documented on the bench sheet. Equipment temperatures are recorded in a temperature logbook. Holding times are automatically calculated in Horizon for both receipt and incubation. The temperature of receipt is recorded by sample receiving staff when samples are received.

5.16. Method References

The analytical methods used for regulatory purposes are published and approved in the Federal Register and/or the WI Administrative Code. The department performs analytical testing for the Safe Drinking Water Act (Total Coliform Rule, Groundwater Rule and Long Term 2 Enhanced Surface Water Treatment Rule), Clean Water Act and WI agencies regulated programs (Department of Natural Resources, Division of Health Services and Department of Agriculture, Trade and Consumer Protection).

The majority of the approved methods for the department are EPA methods or from *Standard Methods for the Examination of Water and Wastewater*. The specific reference is listed in the reference section of the individual technical SOPs. The following is a list of sources for approved reference methods:

- American Public Health Association, American Water Works Association, Water Environment Federation, Standard Methods for the Examination of Water and Wastewater, 21st edition, 2005
- American Public Health Association, American Water Works Association, Water Environment Federation, Standard Methods for the Examination of Water and Wastewater, 22nd edition, 2012
- Method 1623: Cryptosporidium and *Giardia* in Water by Filtration/IMS/FA, December 2005, EPA-815-R-05-002
- Readycult, Chromocult, MI Agar, Simplate, Federal Register, Oct 29, 2002, Volume 67, Number 209, Rules and Regulations, pp65888-65902
- Readycult, Coliforms 100 Presence/Absence test for Detection and Identification of Coliform Bacteria and *E.coli* in Finished Waters". November 2000, Version 1.0, available from EM Science (an affiliate of Merck KggA, Darmstadt, Germany, 480 S Democrat Rd, Gibbstown, Nj 08027-1297
- Federal Register: July 21, 2003 (Volume 68, Number 139, pp 43271-43283). Environmental Protection Agency: Guidelines Establishing Test Procedures for the Analysis of Pollutants; Analytical Methods for Biological Pollutants in Ambient Water
- Federal Register: February 13, 2013 (Volume 78, Number 30)] [Notices] [Page 10269-10365], National Primary Drinking Water Regulations: Revisions to the Total Coliform Rule; Final Rule.
- EPA 600/8-78-017, Microbiological Methods for Monitoring the Environment, Water And Wastes, December 1978
- American Society of Testing and Materials, Annual book of ASTM Standards, Section 11, Water and Environmental Technology, vol. 11.02. 1993.

5.17. Standard Operating Procedures

The table of contents for GENOPS, SOPS, QA METHODS and IOPs located:

O:\SOP\EHD\Division Wide\Final\001 TOC Internal Documents.xlsx

There are separate tabs for Glass Washing/Media SOPs and Water Microbiology SOPs.

The tables of contents include unique SOP identifiers, titles, revision numbers, effective dates, review dates, and hyperlinks to the actual documents.

5.18. Test Methods for accredited Methods

By matrix, list regulatory (reference) method, WSLH SOP number, and title.

Drinking Water

Regulatory Method	**Description**	**WSLH Method**
SM9215B	Heterotrophic Plate Count	304 & 305
SM9221B	Multiple Tube Fermentation	302
SM9221E	Fecal Coliform	312
SM9223/National Field - AEM	Colilert/Colilert -18	300
SM9223/ Federal Register	Colisure	300
Federal Register	Colitag	301
SM9221F/Federal Register	*E.coli* (tube MUG) EC-MUG	312
Federal Register	Readycult	360
Federal Register/SM9230D	Enterococci	356

Non-Potable Water

Regulatory Method	**Description**	**UWSLH Method**
SM9222D/EPA 600/8-78-017	Fecal coliform	314
Federal Register/SM9230D	Enterococci	356
SM9223B	Total Coliform/*E.coli*	300
EPA 1623	*Cryptosporidium/Giardia*	600

Organic Chemistry

Table of Contents

6.1. Personnel

Table 1: Education and Experience

Name	**Title**	**Degree**	**Yrs Exp.**
Angela Albrecht	Chemist	B.S. Environmental Studies	13
Brenda Anderson	Chemist Senior	B.S. Biology	14
Sarah (Sadie) Ayala	Chemist Laboratory Technician	B.S. Wildlife Ecology	0
Kyle J Burke	Chemist Associate	B.S. Chemistry	6
Shaun Carrol	Project Associate Chemist	B.S. Chemistry	4
Donna Johnsen	Chemist Senior	B.S. Chemistry	35
Robel Kebede	Chemist Senior	B.S. Microbiology	6
Erin Mani	Chemist SR	B.S. Chemistry	18
Erin Meinholz	Chemist	B.S. Microbiology	19
Matthew Mireles	Chemist	M.S. Chemistry	5.5
Dave Rogers	Chemist Advanced	BS Water Resources/Chemistry Minor	27
Alex Schwartz	Chemist	BS in Biochemistry	2.5
Jenna Smith	Chemist Associate	B.S. Environmental Science	3

6.2. Training

6.2.1. **Organic Department's Training**

Personnel's training in Organic Chemistry is essentially accomplished by an apprenticeship-type program. A newly hired trainee is assigned to work under the guidance of a qualified organic chemist. The trainer is responsible for teaching the trainee general laboratory operations and routine methods and procedures that are needed to perform most of the section's analytical work. However, any chemist in the section may assist in the training at any time as needed, to streamline production and promote quality.

During the training period, the trainee will also learn how to perform preventative maintenance and troubleshooting techniques and will learn, complete methods and practices necessary to analyze most routine particulate samples. However, because of the varied nature of the samples, no trainee will be able to learn all aspects of sample analysis during the training period. The department recognizes that training is always ongoing and that certain client expectations and requests will always require consultation with the supervisor and other chemists to resolve procedural questions.

This initial training is completed within one year. However, this time may be extended as needed and is dependent on the trainee's abilities and the laboratory's situation. The trainee will only be able to independently report out data after he/she has completed an Initial Demonstration of Capability (IDC) as stated in EHD QC 115 and the "Analyst Method Training Form, Organic Chemistry Dept." on the method he/she is being trained.

Annual training requirements are continuing Demonstration of Capability (DOC) for each analysis an analyst is required to perform. All organic personnel are required to review the follow documents annually:

The NELAC QC Manual,
Data Integrity, Ethics, Data Documentation, EHD GENOP 0029,
Chemical Hygiene Plan, AD SAFEY GENOP 102 and
Emergency Action Plan. AD SAFEY GENOP 102

All employees who work with tissues, serum and blood must read OSHA Bloodborne Pathogens Exposure Control Plan SOP (Labwide Safety GENOP 302) and attend a Bloodborne Pathogen training session annually. Chemists new to working with clinical matrices must complete the University of Wisconsin bloodborne pathogens course, instruction to take the course at located at http://slhicmsprod/regulatory-compliance/safety-topics/bloodborne-pathogens-for-laboratory-and-research/. Additional training specific to the tasks may also be provided.

All employees who work with devices containing radioactive sources such as PO-210 anti-static devices or electron capture devices must review annually the EHD GENOP 041 Antistatic Devices Containing a Radioactive Material. O:\SOP\EHD\Division Wide\Final.

6.2.2. **Training Documentation**

All reported training is recorded in an Access Database located at M:\EHD\QC\ESS ORG and WOHL Training.mdb.

6.2.3. **Forms**

Demonstration of Capability Form: O:\SOP\EHD\Division Wide\Final\EHD QA 115_DOC CALC FORM_rev 5.xls. For analysis that has more than one analyte Initial Demonstration Capability (IDC) spreadsheets have been developed and are located at M:\EHD\ESS(4900)\ESS Org(4940)\ESS Org QA\Initial Demo of Capability IDC\IDC Templates\. The completed spreadsheet should be attached to the EHD QA 115 DOC CALC Form.

Analyst Method Training Form, Organic Chemistry Dept.: M:\EHD\ESS(4900)\ESS Org(4940)\Method Related Documents\Method Support Documents\Organics Analyst Method Training Form.doc.

Figure 1: Analyst Certification Statement

Figure 2: ESS Quality Manual Review Signature Page

Figure 3: Emergency Action Plan Signature Page

Figure 4: Data Integrity Procedure for the Wisconsin State Laboratory of Hygiene's Environmental Health Division (EHD) Plan Signature Page

Figure 5: Chemical Hygiene Plan Signature Page

Figure 1

Analyst Certification Statement
For Current Technical Methods

Analyst Name: _____

Method: (Method number, SOP#, Rev#, and Analyte, or Class of Analytes or Measured Parameters)

I, the undersigned, CERTIFY that I have read, understood, and agreed to perform the most recent version of the above test method.

_____ _____
Analyst Date

Figure 2

ESS Quality Manual Review Signature Page

I have reviewed the Environmental Health Division Quality Policy. In addition, I have familiarized myself with the location and contents: Chapter 1-20 and the analytical section you been assigned. For reference, the current QA Manual can be found in this directory:
General Section: O:\SOP\EHD\Division Wide\Final\NELAC QA Manual rev 17 2019\NELAC QA Manual rev 17 2019 1 general chapter.doc.

Organic: O:\SOP\EHD\Division Wide\Final\NELAC QA Manual rev 17 2019\NELAC QA Manual rev 17 2019 6 Organic Chem.docx

Employee: _____

Annual Review Signature/Date

Date	**Signature**	**Date**	**Signature**

Figure 3

Emergency Action Plan Signature Page

I have reviewed the Ag Drive Emergency Action Plan. I agree that by my supervisor safety officer has discussed all pertinent items with me. I also understand that I may ask for further information or clarification on these items at any time during my employment in the section. The SOP is located at: O:\SOP\Safety\Final\AD_SAFETY_GENOP_101_ Emergency Action Plan.doc

Employee: _____

Annual Review Signature/Date

Date	Signature	Date	Signature

Figure 4

Data Integrity Procedure for the Wisconsin State Laboratory of Hygiene's Environmental Health Division (EHD)Plan Signature Page

I have reviewed the Data Integrity Procedure for the Wisconsin State Laboratory of Hygiene's Environmental Health Division (EHD). I understand and agree to follow all pertinent items in the policy. I also understand that I may ask for further information or clarification on these items at any time during my employment. The document is located at: O:\SOP\EHD\Division Wide\Final\EHD GENOP 029 SOP Data Integrity Procedure.doc

Employee: _____

Annual Review Signature/Date

Date	**Signature**	**Date**	**Signature**

Figure 5

Chemical Hygiene Plan Signature Page

I have reviewed the Ag Drive Chemical Hygiene Plan and agree that my supervisor or safety officer has discussed all pertinent items with me. I also understand that I may ask for further information or clarification on these items at any time during my employment in the section. The plan is located at: O:\SOP\Safety\Final\AD_SAFETY_GENOP_102_Chemical Hygiene Plan.doc

Employee: _____

Annual Review Signature/Date

Date	Signature	Date	Signature

6.3.Document Control System

6.3.1.**SOPs**

Organic Standard Operating Procedures (SOPs) are located at O:\SOP\EHD\ESS\Enviro Organic\Organic and Air Chem\. The current approved SOPs are found in the final folder under four categories: General (GENOP), Instrument Operating (IOP), Method Manual, and Quality Assurance (QA). There is also another directory under "Final" called "Reference Method". These reference methods are saved because they have been cited in an SOP, O:\SOP\EHD\ESS\Enviro Organic\Organic and Air Chem\Final\Reference Method\.

Any SOP being updated but has not been QC reviewed or Supervisor approved is found in the 'in progress" folder O:\SOP\EHD\ESS\Enviro Organic\Organic and Air Chem\Draft\in progress\. While the analyst is updating the method they are required to fill out "SOP Checklist, ESS Organic Chemistry Dept." form to ensure that all required SOP section are addressed. The form is located at M:\EHD\ESS(4900)\ESS Org(4940)\Method Related Documents\Method Support Documents\SOP checklist.doc. When the analyst has finished updating a method, the SOP will be moved to the "Awaiting Peer Review". An analyst familiar with the method is provided the completed "SOP Checklist" and will review the newly revised SOP.

After the review is completed, the SOP is moved to the "Proposed" folder located at O:\SOP\EHD\ESS\Enviro Organic\Organic and Air Chem\Draft\Proposed\. After the SOP has been moved, an email is sent to QC personnel informing them that an SOP is ready for review and the analyst will drop off a completed "SOP Checklist" to QC. QC personnel are required to review all SOPs to assure that they meet EHD GENOP 102 and 103 and any other agendas' accreditation requirements. When new method MDLs are being updated in an analytical method, QC personnel must work with Horizon Admin personnel to ensure the revised method and newly entered Horizon data have the same effective date before the method can be approved.

Following QC review the SOP is moved to the "for approval" folder, only QC personnel are allowed to move SOPs into this folder. The Organic supervisor will be informed by email that a SOP is ready for the final approval and can be found in O:\SOP\EHD\ESS\Enviro Organic\Organic and Air Chem\Draft\for approval\. The supervisor either approves or sends the SOP back for more work.

When the final approval process is completed by the supervisor, the document is dated and QC personnel notified that the document is ready to be moved into the final directory. Before the new revision is moved over into the final directory, the current SOP is watermark in red with "ARCHIVED (with date archived)". The document is saved with its revision number and archived date included in the document's filename. All archived documents are retained at O:\SOP\EHD\ESS\Enviro Organic\Organic and Air Chem\Archived\. The Archived directory has the same four categories as the Final directory. Under each of the four categories there are subdirectories named for the unique SOP number and all archived revisions for that SOP are in that directory/folder. As soon as the old reversion been copied to the "Archived" directory, the new revision is copied to the final directory and the signature page printed for personnel to sign. The signed signature pages

are kept with QC personnel and are available for review. All approved documents are updated in the table of contents, this can found at O:\SOP\EHD\Division Wide\Final\001 TOC Internal Documents.xlsx. Open the document, click on the "Update" box, find the "Organic Chem & Air" tab and click on it to open spreadsheet. If it is a new document, enter it into the sheet or if it is a new revision find the SOP and update with new information. The final step is print out a current revision certification statement for each analyst who does the analysis to sign.

All SOPs (analytical, administrative, instrumental and quality control) are reviewed every 2 years except any SOPs that pertain to the Safe Drinking Water Act (SDWA) which are reviewed annually. All SOPs may be revised before the required 1 or 2 years when changes take place based on instrument/technology changes, regulatory changes, etc. All changes will result in a new revision of the SOP. Each revision will have a new revision number, and an effective date that lists when the SOP was replaced.

6.3.2. **Logbooks**

The laboratory utilizes logbooks for various purposes, including instrument maintenance, instrument result transcription or run logs, cataloging of standards and their preparation, reagents 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 uniquely labeled for easy association with their purpose. Physical logbooks are assigned a unique record number, which is recorded in a database under a table named "ESS Lab Records and it is located at M:\EHD\QC\Archived Records.mdb. Some analysts prefer to maintain electronic logbooks to record information. These are located at M:\EHD\ESS(4900)\ESS Org(4940)\Logbooks\.

6.3.3. **Spreadsheets and Forms**

The spreadsheets and forms are listed in the 001 TOC Internal Documents located at O:\SOP\EHD\Division Wide\Final\001 TOC Internal Documents.xlsx. All the Organic Chemistry is documents are listed under the tab: Organic Chem & Air. The spreadsheet has a hyperlink to each document's location.

All spreadsheets and forms are required to have document control that includes name of document, issuer of document, page # of total pages, and issued date. See EHD GENOP 040 SOP, Protecting Excel and Access Data, for procedure on how to protect the laboratory's spreadsheets. When applicable forms are attached to a SOP that has document control it protects the form from being changed. If it is not possible to include a form in a SOP, all the required document control will be added to the forms. Spreadsheets and forms used in analysis are printed out and the printed copy constitutes the official document.

6.3.4. **Reference**

Links to reference sources are located at O:\SOP\EHD\Division Wide\Draft\In Progress\TOC External Documents.xls under "Organic Chemistry" & Air tab.

6.3.5. **Other Documentation**

IDC studies are assigned Horizon OC numbers and the data is entered into a spreadsheet located at M:\EHD\ESS(4900)\ESS Org(4940)\ESS Org QA\Initial Demo of Capability IDC\. The batch file with the IDC data is kept in cabinets across SC213 along with client sample reports.

Initial MDL studies are conducted when there has been signification change in a method or a new method is being developed. MDL verification studies are done annually to confirm current MDL concentration and samples are run quarterly over the calendar year. All MDL samples are assigned Horizon QC numbers when run. When all the samples are run, they are entered into a spreadsheet. These spreadsheets are located at M:\EHD\ESS(4900)\ESS Org(4940)\MDL Procedure\. Each set of MDL data is also included in the appropriate analytical SOP in the form of MDL 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.

6.4. Records Retention, Control, and Storage

Hardcopies of all worksheets, calibrations, and raw data are currently kept on site for three years. After three years, all hardcopies will be transferred to the State Records Center and the Center will keep the records for the length of time stated in Retention/Disposition Authorization Number (RDA). 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 are kept for the same length of time as hard copies, see Retention/Disposition Authorization Number (RDA) more information. Electronic data, including methods, data files, and sequence logs, are backed up at least every night, archived on suitable media, and are retained the same as the other above-mentioned media.

6.5. Sample Handling Procedures

6.5.1. **Sample Receiving**

The samples and paperwork are delivered to room 217, aisle 3 behind the glassware sink. There is a sign marking the location. Shipping and Receiving personnel are to inform staff that the samples have been delivered before leaving the area. Lab staff checks the condition of the sample, e.g., check for air bubbles for VOC samples, and check the temperature of receipt. Some samples, e.g. TOC, will be checked for pH to verify that the pH is <2. Samples received in unacceptable condition are moved into proper storage, but the paperwork is delivered to the lab supervisor who will notify the client of the reason for sample rejection and arrange for re-collection. All samples are moved to their appropriate storage location, which is indicated when the chemist records a “Transfer” in the Horizon database. All pertinent sample information is then entered into the Horizon database. The Horizon database automatically tracks sample status, entry, and modification times per result (and per sample) basis. The complete Organic Chemistry procedure is found at ESS ORG GENOP 0028, Sample Receipt Protocol.

6.5.2.**Sample Handling**

Once a sample has been received and the data entry procedure completed, the sample is retrieved from storage by the analyst and undergoes analysis. Each testing procedure is described in detail in the individual method SOP.

6.5.3.**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 stored in refrigerators, walk-in coolers, freezers, or other designated areas of the laboratory, the storage location and disposal of samples is recorded in Horizon.

Routine non-enforcement samples are retained until results for those samples are released and may be kept longer if necessary. Samples used for environmental enforcement action will be retained until the proper enforcement entity authorizes their disposal. The only exception to this are those samples whose nature makes it meaningless to keep them (e.g., VOCs in water). These samples are retained for a minimum of two weeks past their holding times and then disposed.

Samples are disposed of in a manner that is consistent with the nature of the sample and all applicable rules and regulations. Waste disposal guidelines are described in the University of Wisconsin Laboratory Safety Guide and the Department Sample Disposal SOP, ESS ORG GENOP 0029.

In some cases, requests for sample return or sample shipment to another site are made by the client. The client will specify the conditions for transfer (ice packs, dry ice, etc.). Prior to shipment, an inventory of samples to be shipped is made and a copy of the chain of custody is made to accompany the sample shipment. For dry ice shipments, special documentation and labeling required by the courier service needs to be arranged with shipping and receiving personnel. Safety concerns for dry ice shipments are considered, such as asphyxiation hazards and excessive pressure build in enclosed spaces or containers.

6.5.4. **Enforcement Samples**

Chain-of-Custody Procedures

Chain-of-Custody (chain-of-possession) is a mechanism for documenting 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 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 the sample collector and chain of custody procedures used by field personnel are described in the Wisconsin Department of Natural Resources Field Procedures Manual.

Transfer of Law Enforcement Samples to the Laboratory

Enforcement samples may be transported to the laboratory by two means:

a. The sample collector may keep the samples in his/her physical possession and deliver them directly to the laboratory.

b. The collector may seal the samples in a field pack (shipping container) and ship the samples to the laboratory by U.S. mail.

Physical Transfer

Department personnel will receive law enforcement samples, identify each sample container with a unique laboratory number and the date received, and complete the chain of custody record initiated by the sample collector. The COC record includes the following information:

a. field identification number,
b. laboratory number;
c. description of the sample;
d. the name, title, work station and telephone number of the person delivering the samples;
e. the time (hour and minute) and date the samples are received in the laboratory;
f. the disposition of any unused portion of the samples remaining after the analyses
g. the signature of the laboratory employee receiving the samples
h. 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.

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 his/her name and date on the tape where the ends overlap. When the field pack is received at WSLH, Shipping and 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.

Handling of Law Enforcement Samples in the Laboratory

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 cold rooms (rooms 119C, 217A, 217B, 219C and I and H freezers in room 217E).

The analyst is responsible for maintaining sample possession 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 any 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 cold room.

Currently only authorized personnel with the use of key-card activated electronic locks, have access to the enforcement cold rooms. In addition, key-cards will enable Capitol Police and Security to maintain an electronic record of all entries to enforcement cold rooms.

Upon completion of all tests, a sample disposition form 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 cold room 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 a minimum of two weeks past the holding time and then disposed. These samples are not retained 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.

6.6. Data Review and Reporting

Batch completion consists of entering data from the analytical run, as well as any other required data such as but not limited to, the date of analysis, dilution factors, temperature, instrument ID, analyst ID, all qualifiers and comments. The analyst will generate the Quality Control Batch Review report for the purposes of auditing the batch. The analyst will then forward the batch to peers and/or supervisors for review. Once reviewed, the data are stamped, dated, and initialed before the reviewer validates the analytical batch to release the clients' reports. During the review process, all aspects of the sample are reviewed (see ESS ORG QA 0008, Data Auditing, for more detail). Any corrections are made in consultation with the analyst and only then are the results released.

Results are available in pdf format, which may be sent to the applicable parties. For SDWA samples, the final report is emailed or mailed to the customer. For enforcement samples, the original chain of custody and TRF are mailed to the customer. In addition, results are available to various external agencies (primarily the WDNR) in an electronic format.

6.7. Procedures for Accepting New Work/Review of Requests, Tenders and Contracts

Organic Chemistry follows section 1.15 in the General Chapter of the QC Manual.

6.8. Laboratory Facilities

The Organic Chemistry laboratory comprises five separate laboratory spaces. Due to the low-level nature of the analyses, the GC/MS volatiles lab room 220 (750 sq. ft.), the air analysis lab room 219 (990 sq. ft.), the PCB and pesticide clean room 218 (660 sq. ft.) and Air Chemistry & Immunochemistry room 215 and 215A (418 sq. ft.) are separated from the rest of the facility by doors. Room 215A is a humidity and temperature control room used for the weighting of air particulate filters. The general organic chemistry lab room 217 (1650 sq. ft.), contains space suitable for pesticide, herbicide, PCB, TOC and other analysis and is separated from the rest of the facility by doors. 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 complemented 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.

6.9.Instrumentation and Equipment

6.9.1.**Instrument Summary for Organic Chemistry**

Item	Manufacturer	UW ID Number	Serial Number	Model	Room	Date Acq.	Description	Horizon Number
Roots Meter	Dresier	na	7459402	5M125	215	Before 1999	Calibrate DNR Orfaces	ACROOT
Spectrophotometer	OHMICRON	U581940	117	RPA-1	215	1992	Triazine	OC0117
Spectrophotometer	Strategic Diagnostics Inc		1064	11141 – RPAII Photometric Analyzer	215	12/21/2016	immunoassay reader	OC0118
Automated Weighing System	Bohdan	U016506	122612	AWS-1503	215A	1999	Air Chemistry Sample	na, uses balance number
Solid Phase Extraction Station	Gilson	U650422, U016466	ACN 001768396 Tray 260f7n124u z-arm 261c1q072u	GX-271, Detector 112, Pump 307, Syringe pump 402	217	2007	Research & GPC Sample Preparation	na
Solid Phase Extraction (SPE)	PromoChrom Technologies Ltd.	U037488	201906004	SPE-03	217	08/07/2019	Sample Preparation	OCSPE1
Solid Phase Extraction (SPE)	PromoChrom Technologies Ltd.	NA	202001003	SPE-03	217	2/2020	Sample Preparation	OCSPE2
GCMS	Agilent	U0636202 U016459	US10218127 US10462148	6890N/5973	217	>2014	Research	OCMS10
TurboVap	Biotage	U034457	181000417	LV	217	2018	Various Sample Preparation	na

GC/ECD	Agilent	U641252	GC – CN10638080, Autosampler – US04209553	6890N (G1530N), G2614A	217	Acquired from WOHL 3/2020	Halo Acetic Acids	OCEC6
GC/ECD	Agilent	U636159 U016463	US10308009	6890N/5973	217	2003	Two mega-bore columns used for PCB Aroclor and pesticide analysis	OCEC3
GC/ECD	Agilent	U636158	US10308008	6890N	217	2003	Two capillary columns used for PCB and pesticide analysis.	OCEC5
GC/FID	Agilent	U027013	CN14243040	7890	217	2014	A capillary column GCs used for glycols, petroleum fingerprints, and flashpoint alcohol analysis	OCFID4
TOC analyzer	Teledyne Tekmar	U032981	Detector - 15-1600-000 US17306010 LSS Boat – 15-2400-000 US17373018	Lotix & LSS Boat Sampler	217	1/7/2018	Used for TOC in sediment and TOC/DOC/DIC in water	OCTOC3
TOC analyzer	Sievers	U641076 U016471	6041102	900 Portable & Autosampler	217	2006	Used for TOC in TOC/DOC/DIC in water	OCTOC1
Flash Point Analyzer	Stanhope-Seta	U024845	1035938	Setaflash Series 3	217	2015	Waste analysis	OCFP2
Flash Point Analyzer	Koehler Pensky-Martens	na	R07002736B	Close Cup Flash Tester	217	2011	Waste analysis	OCFP1
GC/ECD	Agilent	U644134 U016457	CN10910031	7890A	218	2009	Used for Low-level PCB and pesticide analysis	OCEC1

GC/MSD	Agilent	U641478	US10442004 US43130465	6890/5973N	218	2004	Used for Atmospheric Pollution Tracers	OCMS6
GC/MSD	Agilent	U641477 U008506 U024793	US10343057 US33230069	6890/5973	218	Nov-14	8270 PAHs	OCMS9
GC/MSD	Agilent	U641149 U016443	CN10636106 US62724106	HP6980/5975	219	2006	Used for air analysis for air toxics (TO-15)	OCMS2
Cryogenic Concentrator	Entech	na	1143	7200	219	2013	Used for air analysis for air toxics (TO-15) High	na
Auto Sampler Tree	Entech	na	1410	7016D	219	2013	Used for air analysis for air toxics (TO-15) High	na
Canister Cleaning System	Entech	U641658 U016441	1220	3100A	219	2003	The instrument is used to clean gas containers after analysis for air toxics	na
EC/OC Analyzer	Sun Set Labs	U630125	101	Series I	219	1999	Two used for organic Carbon Analysis	OECOC1
EC/OC Analyzer	Sunset Laboratory Ink	UO35726	459-228	5L	219	12/2018	Used for Organic Carbon Analysis	OECOC3
GC/MSD	Agilent	U636291 U016448	US10307014 US21884845	HP6890/5793N	219	2003	PBDEs in serum Analysis	OCMS3
GC/MSD	Agilent	U646107 U018573	CN10531064 US11012716	7890A/5975C	220	2011	EPA 524.2	OCMS7
P & T Concentrator	Teledyn-Tekmar	na	US12209003	14-9800-100	220	2011	This is being used with the GC/MSD for EPA 524.2	na
P & T Concentrator	Teledyn-Tekmar	na	US12208003	14-9800-100	220	2014	This is being used with the GC/MSD for EPA 8260/624	na

Purge & Trap Autosampler/ Concentrator	Tekmar	na	US12220003	Aquatek100	220	2011	Sample prep for 524	na
Purge & Trap Autosampler/ Concentrator	Tekmar	na	US13282008	Aquatek100	220	2014	Sample prep for 8260/624	na
GC/MS	Agilent	U021410	CN13483030 US1347L217	6890N/5977A	220	2014	Sample analysis 8260/624	OCMS8
UPLC	Waters	U651766 U008505	F11CHA 836G	Acquity	220	2013	Research	na
MS/MS	Sciex	U642279	V21120710	API 4000	220	2004	Research	OCLMS1
QTrap	Sciex	U018578	AU24991012	5500 QTrap	220	2011	Research	QTRAP
Digital Dilution System	Entech Instruments Inc.	NA	AM404	PG7-50.00-PSIA	219	4/17/17	TO15 Standard Making System	OCDDS2
Microwave	Milestone	4036382	19013729	MA174-002	217	3/13/19	Microwave Extraction	OCMic1
LC-MS/MS	Agilent/SCIEX	U028646/ U030981	DEBA200712 (High Speed Pump), DEBA401017 (Multicolumn Thermostat, MCT), DEBAQ01506 (Multisampler)/ CJ20651702	1290 Infinity II/Triple Quadrupole 4500	220	2016	Shared 4500 for PFAS	LC E
1260/1290 LC Stack	Agilent	NA	JPAAA01635	G4225A	220		Degasser	NA
			DEACB04222	G1312B	220		BinPump	
			DEBAC05015	G1316C	220		TCC	
			DEBAK11491	G1330B	220		Thermostat	
			DEACO03060	G1367E	220		Hip ALS	

Chromatograph	Agilent	U646281 U018581		1260 Infinity II	220	2016	Research	na
			JP02415172	G1379B	220		Degasser	
			DEABM00995	G1312B	220		CapPump	
			DEAAK03274	G1316A	220		COLCOM	
			DEBAK02263	G1330B	220		1260 Therm	
			DEAAX00666	G1367E	220		1260 ALS	

Instrument maintenance, calibration, and verification procedures, can be found in ESS ORG Method SOP or ESS Instrumental Procedure IOPs, see section 6.17 for a listing of the SOP available.

6.9.2.**Supporting Equipment**

Supporting Equipment like balances, volumetric dispensing equipment, thermometers and other such devices shall be calibrated or verified at minimum annually and be traceable to National Bureau of Standard (NBS). All Supporting Equipment records for calibration or verification are kept in the Organic Chemistry Department. The SOP for supporting equipment can be found in Section 6.18.

Refrigerators/Freezers

Item	Manufacture	Model	Room	Date Acq.	Description	Unique ID
Freezer F	SN: Z17T181519ZT	R411XA16	220	before 2014	Glycols and 524 standards	RM220F
Refrigerator K	SN: Z17T181519ZT	R411XA16	220	before 2014	524 samples	RM220K
Freezer Q	Marvel Scientific SN:201012313125H	17CAF001	219	before 2014	Research Filters	RM219Q
Rm. 219C Walkin	Norlake Scientific		219	before 2014		RM219C
Rm. 219D Walkin	Norlake Scientific		219	before 2014		RM219D
Freezer E	Lab-Line Instruments Inc, SN 322	3552, Explosion Proof Fridge-Cab	217	before 2014	LCMS Standards	RM217E
Freezer P	Fisher Scientific SN:1422070873481	97-928-1, Explosion Proof	215	before 2014	GC/MS Standards Freezer	RM217P
Freezer MF	Frigidaire SN: LA22409216	FRT21C5AW5	215	before 2014	Air Chemistry Freezer	RM215MF
Refrigerator MR	Frigidaire SN:LA22409216	FRT21C5AW5	215	before 2014	Air Chemistry Refrigerator	RM215MR

Freezer N	GE SN: HG174296	FUM21DPDRWH	215	before 2014	Air Chemistry Freezer	RM215N
Rm. 217B	Norlake Scientific		217	before 2014	enforcement walkin	RM217B
Rm. 217C	Norlake Scientific		217	before 2014	sample walkin	RM217C
Freezer H	Lab-Line Instrument Inc., SN: 0203382275	FV21M2WKFA, Explosion Proof Fridge-Cab	217E	before 2014	fish and serum freezer	RM217H
Freezer A	Lab-Line Instrument Inc. SN: 12481705	13-987-525F, Explosion Proof Fridge-Cab	216D	before 2014	Standards	RM216D-A
Freezer B	Baxter, SN: U13E212026	EPF2118ABA, Explosion Proof Fridge-Cab	216D	before 2014	stock standards	RM216D-B

Ovens

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
Muffle Furnace	Thermolyne	30400	218	before 2014		ORG MF
Drying Oven	Lindberg Blue M SN: X14T-521025-XT	Gravity Oven G01300A-1	218	before 2014	used to dry TOC vials for research samples	ORG DO
Oven G	Fisher Scientific U596821 SN: 30500073	Isotemp Oven Model 655G	217	before 2014		ORG G
Oven D	Fisher Scientific, SN: 1579070356038	637G	217	before 2014	Fluorosil and silica gel oven	ORG D
Oven C	Thermo Scientific SN: 602976	Precesion 6520	217	before 2014	used to dry soil/sediment samples	ORG C

Balances

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
Balance	Denver Instrument Company SN: T0114507	TO-403	218	before 2014		OC4507
Balance	Adventurer SN: 1203500330P	Ohaus	217	before 2014		OC0330
Balance	Denver Instrument Company SN: 25955735	MXX-412	217	before 2014		OC5735
Balance	Mettler Toledo SN: D39364 U521914	AD163	216D	before 2014		OC9364

Balance	Sartorius SN:37060389 U552892	SART A2003	215	before 2014	Used in TSP and PM_{10} analysis	ACSART
Balance	Mettler Toledo SN: 115432153	MT5	215	11/19/1996	Microbalance	ACMT5
Balance	Mettler Toledo SN: P09378	PB302	217E	before 2014		OC9378
Balance	Mettler Toledo SN: 1120143018	MX5	215	before 2014	Microbalance and Automated Weighing System (AWS)	ACMX5

Rotovaps

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
Rotovap	Buchi	R210/B-491 (bath)	217	before 2014		RVP001
Rotovap	Buchi U598465	R-114/B-490 (bath)	218	before 2014	Air Tracers	RVP002
Rotovap	Buchi	R110	218	before 2014		RVP003

Soxhlet Banks

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
Soxhlet Bank	Lab-Line Instruments Inc.	5000	217	before 2014		SXA217
Soxhlet Bank	Lab-Line Instruments Inc.	5000	217	before 2014		SXB217
Soxhlet Bank	Lab-Line Instruments Inc. U602836	5000	217	before 2014	backup soxhlet bank	SXC217
Soxhlet Bank	Lab-Line Instruments Inc. U602836	5000	217	before 2014	backup soxhlet bank	SXD217
Soxhlet Bank	Lab-Line Instruments Inc. U614250	5000	218	before 2014		SXA218
Soxhlet Bank	Lab-Line Instruments Inc. U561993	5000	218	before 2014		SXB218

RO Water Systems

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
RO Water System	ELGA U646714 & U008508	PureLab Ultra	219	before 2014	RO System	RO219A

RO Water System	ELGA U646714 & U008508	PureLab Ultra MK2-US	218	2006	RO System	RO218A

Vortex Mixer

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
Vortex-Genie Mixer	Scientific Products SN:13274	K-550-G	218	before 2014		VGM001
Vortex-Genie Mixer	American Scientific Products SN:007911	S8223-1	217	before 2014		VGM002
Vortex-Genie Mixer	American Scientific Products SN:013627	8223-1	217	before 2014		VGM003
Vortex-Genie Mixer	Fisher Scientific	G-560	215	before 2014		VGM004

Centrifuge

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
Centrifuge	Thermo Scientific U024899	Sorvall ST 40	217	2015		ORG009
Centrifuge	Eppendorf SN:2607	5424	217	before 2014		ORG010
Centrifuge	DYNAC	UW ID:496452, SN: 16324	216	before 2014	WOHL centrifuge for serum extractions	

Miscellaneous

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
Tube Rotator	Scientific Equipment Products SN:2494	NA	218	before 2014	serum extractions	ORG001
Blow Down Apparatus	Organomation Associates Inc		218	before 2014	Blow down Air Tracers Work	ORG002
Shaker	Lab-Line Instruments Inc. SN: 0284	Multi-wrist shaker 3587	217	before 2014	used in the extraction of Haloacetic acids	ORG005
Shaker Table	Lab-Line Instrument Inc SN: 0590-8377	4625	215	before 2014	used in the extraction of organic acids	ORG007
Blender	Waring	Commercial Blender, SN:15024	217E	before 2014	grinding fish tissue	ORG012
Blender	Tekmar	A-10, SN: 263168	217	before 2014	grinding sediment for TOC	ORG011
Sonicator	Bransonic	52, SN:06157	217	before 2014		ORG013

Sample Homogenizer	Biospec Products	Tissue Terror: 985370-395	217	before 2014	Research Tissue Extraction	ORG015
Rotary TCLP extractor	Assoc. Design Mfg.	FS9 NSI 34RH	118, closet	before 2007	Tumbler	Instrument #68
Desiccating Dehumidifier	Munters SN: 5580	HC-300	Hallway Closet outside RM215	5/29/2014	Dehumidifies RM215 Air Chemistry Weighting	ORG016
pH meter	Fisher Scientific	Accumet AE150	215	3/8/2017	WI DNR Lake Herbicide Monitoring Sample Preparation	ORG017
Sonicator	Fisher Scientific	FS9, SN:228299	220	4/11/2017	VOC sample preparation for OC17601 and OC15512	ORGFS9
Flow Meter	Agilent	G6691A, SN: MY18120564	217	7/23/2018	Stored by and used by the Lotix TOC Instrument in 217	ORG018
Vacuum Manifold	Supelco	57044	217	~2008	SPE Column Vacuum Manifold	OCVac1
Evaporative Humidifier	AIRCARE	EA1208	215A	2016	Evaporative Humidifier with Adjustable Humidistat	na
Vacuum Manifold	Supelco	57265	217	~2008	24 Port SPE Column Vacuum Manifold	OCVac2

Pipettes

Item	Manufacture	Model	Room	DateAcq.	Description	Unique ID
TOC Pipette	Thermo Scientific	Finnipipette F1 (10-100ul)	217	Feb-15	TOC research	LH66733
Research Pipette	Eppendorf	Reference	217	<8/5/2015	Research 100-1000uL	486680
TOC Pipette	Eppendorf	Research Plus variable volume	217	Jul-14	TOC in Sed, PCB/PEST IS	H26398D
Air Chemistry	Eppendorf	Repeater	215	<1/1/2015	Air Chemistry	750
Air Chemistry	Eppendorf	200ul	215	<1/1/2015	Air Chemistry	335758
Air Chemistry	SCILOGEX	10-100mL	215	7/30/2018	Air Chemistry	YL183AE00-27520
Research Pipette	Gilson	100-250uL	217	<3/18/2019	Research 100-250 uL	S12062M
Research Pipette	Gilson	10-95uL	217	<03/18/2019	Research 10-95 uL	U12178B
Research Pipette	Gilson	5-25uL	217	<3/18/2019	Research 5-25 uL	S124166

Research Pipette	Gilson	2-10uL	217	<3/18/2019	Research 2-10 uL	W13060E
Research Pipette	Eppendorf	Reference 2	217	11/7/2019	Research 100-1000 uL	J11656I
Research Pipette	Gilson	100-1000 ul	217	8/20/2019	PFAS Research 100-1000 µL	QJ05352
Research Pipette	Rainin	AutoRep M 250-1000ul	217	.11/15/2019	Repeat Pipette 250-1000 ul	02J39611

6.10. Laboratory Supplies and Chemicals

6.10.1. **Verification of Chemicals, Supplies and Vendors**

ESS Organic follows the same procedure as stated the section 1.18.3 to 1.18.5 to verify and evaluate supplies, approval criteria for vendors and the approved vendor list but ESS Organic does need to add more vendors to the list as needed for specific analysis needs. Other vendors may be used due to analytical or availability needs.

6.10.2. **ESS Organic Specific Vendors**

AB Sciex
Abraxis
ACE Glass, Inc.
All-Foils, Inc.
Apel Riemer Environmental
Biotage
Cambridge Isotope Laboratories
C/D/N Isotopes
City Chemical
Energy Management Consultants (EMC)
Enzo Life Sciences
GasTech
GE Analytical
Gilson®
Ionics Instruments
Koehler Instrument Company
Modern Water
MTL
Parker Hannifin Corporation
Pressure Biosciences
Sievers Instrumentation
Sunset Laboratories
Teledyne Tekmar
Thermo Electron North America LLC
Uline
UTAK Labs
Valco Instruments Company Inc.
Wellington Labs
Wenniger Compressor
Zorn Compressor

6.10.3. **Sample Containers**

Virtually all samples received by the laboratory arrive in appropriate 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 Screw-capped Tube

Sample preservation tubes are obtained from an approved provider, currently Fisher Scientific. Vials containing 2 ml of 1:1 hydrochloric acid are used in the field preservation of VOCs by EPA 524.2 for the 40mL vials.

Quart Mason Jars

Only brand new jars are used and they are sent through an established 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 has been discontinued.

One Liter, 250-ml and 125-ml Amber Bottles

Brand new one-liter amber bottles with Teflon lined septa are sent out with no checks. The laboratory buys certified bottles and the Certificate of Analysis will be retained in the Shipping and Receiving Department for the one-liter bottles. Organic chemistry will keep the Certificate of Analysis for the 125- and 250-ml bottles.

Forty- 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. The Certificate of Analysis for both 40- and 60-ml vials will be kept in the Organic Chemistry Department.

Polypropylene Plastic Containers

Two hundred and fifty milliliter (250 ml) conical tubes with Trizma preservative are used to collect drinking water samples for PFAS. Non-potable water PFAS samples are collected in 250 ml bottles with no preservative. Hundred milliliter (100 ml) digestion tubes are used to collect soil samples for PFAS analysis.

All newly ordered containers are assigned a batch number and tested for PFAS contamination before being made into sampling kits. The clean container analysis is located at M:\EHD\ESS(4900)\ESS Org(4940)\Method Related Documents\PFAS\ under media collection type.

Gas Canisters

The 1.8-, 2.7-, 6-, and 15-liters canisters used for VOCs in air analysis are cleaned and re-used. After analysis is complete, the containers are cleaned by the repeated application of humid air and vacuum. For each batch of twelve 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. The acceptable cans are then 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 would be made to choose the “dirtiest” can in the batch for checking.

6.10.4. **Chemicals**

Standards

The laboratory buys standards of the highest purity, which are obtained from an approved list of suppliers from Sections 1.18.5 and 6.10.2. 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.

Gas Standards are UHP (99.999%) or better and may be equipped with additional traps (e.g., activated carbon, moisture) to improve their performance.

Reagents, Solvent, Gases & Dry chemicals

Any background contamination in secondary reagents can seriously affect the quality of an analysis. As a result, only the highest purity reagents, 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.

6.11. General Quality Control Procedures

6.11.1. **Quality Control Data Gathering and Documentation**

QC Data

All QC data that is tracked is being recorded in the Horizon program. The program is interactive and designed to collect QC results and their associated biographical data in a database format. Horizon allows for adding, and modifying QC results, and identifies each result with a unique code.

For each set of QC results, the analyst will prepare a QA report generated by Horizon or the current laboratory LIMS system that will contain all biographical information and all results. In addition to the electronic input of the QC data, a hard copy of the report will be retained with the appropriate sample results.

The Horizon program is accessed through the WSHL internal web Internet located at http://slhicmsprod/, and clicking on the Horizon box. ESS ORG QA 0001, Horizon Laboratory Information Management System (LIMS) and Quality Assurance, describes how to Login and enter data into the QC database. Several tables are described below

a. Table 1 is the Queues Code used in each area of analysis

b. Table 2: The organic Acodes and method codes for each of the ESS ORG METHOD SOPs, along with a description are found here.

c. Table 3: The Matrix Codes and a description of each.

d. Table 4: Sample Type Codes, along with description, QC type and Duplicate Link (if there is a link to the Sample Type code).

e. Table 5: The three types of calculation done in the QC database and the formula used.

Detection Limit Data

Analytical parameters include method detection limit (MDL), limits of quantification (LOQ), method biases (including instrumental and matrix factors), method accuracy, precision, and equivalency. The statistical MDL and the LOQ are determined using the procedure outlined in Definition and Procedure for the Determination of the Method Detection Limit, Revision 2, Title 40 Code of Federal Regulations Part 136 (40 CFR 136, Appendix B), December, 2016. MDL/LOQ data is collected when a new method is first being developed and verified after that unless there is a major change in the method than a new MDL study shall be done. See Section 6.3.5, Other Documentation, for where detection limit studies are stored. Horizon Detection Limits are located under Operations/Detection Limits where Edits and Inquiries can be made.

6.11.2. Corrective and Preventative Action Procedures

Corrective Action Procedure

Corrective action must be taken after any QC failure and anytime an analyst observes an instrument problem or analytical problem. When corrective action is needed within the run, the following questions will be addressed:

a. What was the source of the problem (what was the failure)?
b. What corrective action was performed (what was done to fix the problem)?
c. What was done to verify the corrective action (how do you know the fix worked)?

The corrective action must be documented within the analytical run and/or in the appropriate instrument logbook. Procedures for taking corrective action in the laboratory are often instrument specific. Corrective action may include repairing, adjusting, or servicing instrument hardware or software. Corrective action may also include sample handling or treatment issues. Other corrective actions would relate to correcting any analyst errors that may have taken place. The analyst will perform corrective action unless an outside service call is required. If outside service is required, the Department Supervisor will be consulted.

When a specific problem has been identified and corrective action is validated as working or not then any samples that may have been affected by the problem needs to be addressed by a subsequent action. Possible subsequent actions include: not reporting result, qualifying results, reanalyzing (and/or re-digesting) a single sample, reanalyzing an entire QC group, reanalyzing all samples from the last acceptable QC sample, and re-standardizing and reanalyzing all samples within the analytical run

There are occurrences which affect more than one analytical batch, departments and failed proficiency or blind samples. These occurrences are documented in the lab-wide "Occurrence Management System. All occurrences entered in this system follow the procedures listed below.

a. LABWIDE GENOP 706, Occurrence Reporting Procedure located at O://Labwide SOP Policies\Final\LABWIDE_GENOP_706_Occurrence_Reporting_Procedure.docx.

b. LABWIDE GENOP 707, Occurrence Management System Policy located at O://Labwide SOPPolicies\Final\LABWIDE_GENOP_707_Occurrence_Management_System_Policy.docx.

As noted in 6.13 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 Proficiency sample results fall outside the acceptable range, the analyst will complete an Occurrence Management form. This 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.

Qualifiers Used in the Sample Comment Field

* - QC FAILURE. NO confidence in the results.

*A – Lab accident – no results reported.

*AI – Aroclor identification is not possible.

*AP – Canister was received at atmospheric pressure.

*AT – The results are not confirmed by an alternate technique.

*B – Compound detected in lab blank.

*BC – Sample container(s) received broken – no work done.

*C – Calibration Exceeds quality control limit.

*CCCL - The Continuing Calibration Check (CCC) does not meet the lower QC limit.

*CCCU - The Continuing Calibration Check (CCC) does not meet the upper QC limit.

*CCVL - The Continuing Calibration Verification (CCV) does not meet the lower QC limit.

*CCVU - The Continuing Calibration Verification (CCV) does not meet the upper QC limit.

*D – The LOD is not achievable due to dilution.

*DB – Congener detected, but at a level that is less than two times the blank value.

*DNQ – Compound detected but not quantitated.

*DOC – Sample was filtered in the lab.

*DOC1 – Acid preserved sample was filtered in the laboratory.

*DOC2 – Sample was filtered more than 48 hours past collection time.

*DUP - The Relative Percent Difference (RPD) for the sample and sample duplicate does not meet the QC limit.

*E – The dry weight concentration for this compound is indeterminate.

*ES – Sample was run using expired standard.

*F – Analysis not possible due to foaming.

*FP – Flash point cannot be determined on a solid.

*FPPM – Run by Pensky-Martins method EPA 1010A, 2004

*FPSF – Run by Setaflash method EPA 1020B, 2004

*FRB – Compound detected in field reagent blank (FRB).

*FRBL - The Field Reagent Blank (FRB) does not meet the lower QC limit.

*FRBU - The Field Reagent Blank (FRB) does not meet the upper QC limit.

*HCl – Hydrochloric Acid Expired, pH <2

*I – Interference.

*ICVL - The Initial Calibration Verification (ICV) does not meet the lower QC limit.

*ICVU - The Initial Calibration Verification (ICV) does not meet the upper QC limit.

*IP – Sample contains/may contain polybrominated biphenyl #153 (PBB-153).

*IS – The internal standard QC limit is exceeded.

*LAS – Large air space in vials – rejected – mailer resent.

*LCSD - The Relative Percent Difference for the LCS and the LCSD does not meet the QC limit.

*LCSDL - The Laboratory Control Spike Duplicate (LCSD) does not meet the lower limit for percent recovery.

*LCSDU - The Laboratory Control Spike Duplicate (LCSD) does not meet the upper limit for percent recovery.

*LCSL - The Lab Control Spike (LCS) does not meet the lower QC limit.

*LCSU - The Lab Control Spike (LCS) does not meet the upper QC limit.

*LFBL - The Lab Fortified Blank (LFB) does not meet the lower QC limit.

*LFBU - The Lab Fortified Blank (LFB) does not meet the upper QC limit.

*LOL – The % water test was not applicable to the sample because of the lack of liquid in the sample.

*LRB - Compounds detected in the Lab Reagent Blank (LRB).

*MRLL - The Method Report Limit (MRL Check) does not meet the lower QC limit.

*MRLU - The Method Report Limit (MRL Check) does not meet the upper QC limit.

*MSB - Background levels in matrix spike are too high to assess matrix effects.

*MSD - The Relative Percent Difference for the MS and MSD does not meet the QC limit.

*MSDL - The Matrix Spike Duplicate (MSD) does not meet the lower limit for percent recovery.

*MSDU - The Matrix Spike Duplicate (MSD) does not meet the upper limit for percent recovery.

*MSL – The matrix spike does not meet the lower QC limit.

*MSU – The matrix spike does not meet the upper QC limit.

*NA – Result not available due to poor chromatographic response.

*NI – Sample not received on ice.

*NIAS – Not iced - large air space – rejected – mailer resent.

*NPP – Sample not properly preserved – no work done.

*NR – Not received in WSLH vials – rejected – mailer resent.

*NWD – Insufficient sample – no work done.

*PCL - The TOC Performance Check does not meet the lower QC limit.

*PCU - The TOC Performance Check does not meet the upper QC limit.

*PF – Paint filter test not needed – Sample was visually confirmed to be a liquid.

*PWM – Sample was preserved with methanol in the laboratory.

*QCSL - The Quality Control Standard (QCS) does not meet the lower QC limit.

*QCSU - The Quality Control Standard (QCS) does not meet the upper QC limit.

*RL – Report limit not achievable due to dilution.

*RPD – The relative percent difference for the matrix spike and matrix spike duplicate does not meet the QC limit.

*RSD – Replicate precision exceeded expected range.

*SA – Sample was acidified with H2SO4 in the laboratory.

*SAP – Sample was acidified with phosphoric acid in the laboratory.

*SI – Interference occurred with the surrogate compound. No surrogate recovery determination is possible.

*SNV – Sample not valid – no work done.

*SRL – Surrogate recovery does not meet lower QC limit.

*SRML - The Standard Reference Material (SRM) does not meet the lower QC limit.

*SRMU - The Standard Reference Material (SRM) does not meet the upper QC limit.

*SRU – Surrogate recovery does not meet upper QC limit.

*SSL - The Second Source Standard (SS) does not meet the lower QC limit.

*SSU - The Second Source Standard (SS) does not meet the upper QC limit.

*SW – Sample weight exceeds 35.0g – no work done.

*TB – Compound detected in trip blank

*TS – Traceability Sticker not returned, pH of sample <2

*U – Results are approximate, above upper calibration range.

*VOC – This sample was analyzed for Volatile Organic Compounds (VOCs) using a qualitative technique and is intended to be a screening tool to provide a general profile for the presence of VOCs. The concentrations of the VOCs present should be considered approximate.

*WCG – Water content is greater than 40% by weight. *WCL – Water content is less than 40% by weight.

*WS – Sample was received without methanol preservation or in an appropriate coring device. VOC results may be biased low.

*pH<2 – Sample was acidified in the lab with HCl to pH<2.

Preventative Action Procedures

Any scheduled instrument maintenance such as the Mettler's annual balance check, refer to EHD GENOP 015, Annual Balance Calibrations and Maintenance Checkups, for the procedure. Any other schedule maintenance will be recorded in ESS ORG Instrument Operation Procedure (IOP), see Section 6.17 for location.

6.11.3. 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/or time to 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 (work list, lab sheet, etc.). For program requests, the deviation would be stated 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 Section 6.11.2).

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.

6.12. Quality Control Limits Procedures

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) variance 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 statistically will be expected in

this area. However, any sample data associated with a 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-T) 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.

QC Limits are reviewed annually to determine to changes need to be made. If this is done, EHD QA 113, Horizon QC Limits Evaluations & Updates will be employed.

Precision Control Limits

Precision is defined as the degree of mutual agreement among individual measurements made under defined conditions (US EPA, 1979). Precision limits are calculated by the relative percent difference (RPD). For many parameters, precision may be concentration dependent. In these instances, control limits are established for a series of sequential concentration ranges. These limits are either updated yearly, when the determinative method requires, or when the test conditions change. Precision sample pass if they are between zero and three standard deviations.

If a precision result exceeds the UCL, two possible paths are followed. After appropriate corrective action has been performed, 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. Test 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 6.11.2. for possible QC flags.).

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. Sample pass if the

percent recovery is within ± 3 standard deviations. If the sample is out of control, a similar procedure is followed as described in the precision section.

6.13. Internal and External PT Studies

6.13.1. **External PT Programs Schedule**

ESS ORG QA 0006, Proficiency Testing Sample Procedures, describes what programs and proficiency the Organic group participates in.

6.13.2. **Internal PT Programs Schedule**

ESS ORG QA 0002, Blind Samples, describes how often and what type of samples are analyzed on a quarterly basis.

6.14. Internal Audits

Internal Audits follow the procedure described in EHD QA 120, Division Internal Auditing Procedure and organic chemistry supplements it with ESS ORG QA 0003, Auditing Procedure. Organic chemistry will use the internal audit template located at O:\SOP\EHD\Division Wide\Final\Method Internal Audit Checklist Template for EHD - Chemical testing_rev 3.doc

6.15. Traceability of Measurement

Standards

The highest purity standards are obtained for analysis. 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 located at M:\EHD\ESS(4900)\ESS Org(4940)\Standards Log\Certificates of Analysis\. 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 (10 years, unless otherwise indicated).

All standard solutions, including any necessary serial dilutions, are recorded in the proper standard forms located at M:\EHD\ESS(4900)\ESS Org(4940)\Standards Log\ 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 forms located at M:\EHD\ESS(4900)\ESS Org(4940)\Standards Log\Completed Standard Forms\. 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, and expiration date. The stability of all solutions is carefully monitored. They are re-standardized and/or prepared at a frequency determined by the appropriate analytical method.

Most standards use the documentation procedure from .ESS ORG GENOP 0036, General Chemistry Standard Log book when making standard solution.

Reagent Chemicals

All reagents that are utilized as part of sample analysis will be documented to the extent necessary to determine how a reagent was prepared and what was used in its preparation. A specific procedure can be cited, but that citation must appear in a notebook/ worksheet with the analyst's initials and date. All containers shall be labeled with the contents, a receipt and expiration date. All parent reagents will obtain a copy of its Certificate of Analysis (COA), these are located at M:\EHD\ESS(4900)\ESS Org(4940)\Standards Log\Certificates of Analysis\Bulk Chemical COAs\.

Reagents include purchased solutions and those solutions that are mixed or formulated by laboratory personnel.

Pure reagents that are used to make solutions shall be documented.

Solutions used in an analytical batch shall be traceable back to the pure reagents used.

If any solution (parent) used to make another solution (daughter) shall be traceable back to the parent solution and the pure reagents used to make the parent.

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 carrier 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 Airgas 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 employs 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.

Dry Chemicals

The primary dry chemicals used in the laboratory and their proper preparations are documented in the analytical method and preparation procedure. All containers shall be labeled with the receipt and expiration date. For each primary dry chemical lot the laboratory will obtain a copy of its Certificate of Analysis (COA), these are located at M:\EHD\ESS(4900)\ESS Org(4940)\Standards Log\Certificates of Analysis\Bulk Chemical COAs\

Florisil and silica gel preparation document is in the analytical method and preparation procedure. Below are SOPs used for Florisil and silica gel:

ESS ORG GENOP 0032, Silica Gel Calibration and Usage

ESS ORG GENOP 0033, Florisil Preparation Procedure

6.16. Method References

Analytical methods used by the Wisconsin State Laboratory of Hygiene Organic Chemistry Departments are documented in the "Department Methods Manual". The methods are derived from documents such as:

- US Fish and Wildlife, "Technical Papers of the Bureau of Sport Fisheries and Wildlife, No. 65, Handbook of Procedures for Pesticide Residue Analysis, U.S. Dept. of the Interior, Fish and Wildlife Service, Bureau of Sport Fisheries and Wildlife".
- USEPA Federal Register.
- USEPA, Office of Water and Waste Management, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", EPA publication SW-846, Third Edition, Final Updates I (1993), II (1995), IIA (1994), IIB (1995), III (1997), IIIA (1999), IIIB (2005), IV (2008), and V (2015).
- USEPA, "Analysis of Pesticide Residues in Human and Environmental Samples", US EPA Health Effects Research Laboratory Envir. Tox. Div., Research Triangle Park, N. C.
- USEPA, "Handbook for Analytical Quality Control in Water and Wastewater Laboratories", US EPA/600/4-79/019, 1-1, (1979).
- AOAC, "Official Methods of Analysis of the Association of Official Analytical Chemists", Thirteenth Edition, (1980)
- FDA, "Pesticide Analytical Manual", U.S. Dept of Health and Human Services, Food and Drug Administration, Revised June (1980)
- USEPA, "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water", July (1978)
- USEPA, "Methods for the Determination of Organic Compounds in Drinking Water," US EPA/600/4-88/039, December (1988)
- USEPA, "Methods for the Determination of Organic Compounds in Drinking Water," Supplement 1,2 and 3 US EPA/600/4-90/020, July (1992)
- USEPA, "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020, United States Environmental Protection Agency, Revised March 1983 and 1979 where applicable.
- ASTM, "1993 Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, (1993)
- APHA et. al., "Standard Methods for the Examination of Water and Wastewater", 20th ed. (1998)
- USEPA, Technology Transfer Network Ambient Monitoring Technology Information Center, Air Toxic – Monitoring Methods, http://www.epa.gov/ttn/amtic/airtox.html.
- There is also a Reference Method directory located at O:\SOP\EHD\ESS\Enviro Organic\Organic and Air Chem\Final\Reference Method\. The most recent method references are being saved there.

6.17. Standard Operating Procedures

All current and archived SOPs are listed in the table of content located at: O:\SOP\EHD\Division Wide\Final\001 TOC Internal Documents.xlsx under the "Organic Chem & Air" Tab.

6.18. Accredited Methods (NELAC, EPA, DNR)

6.18.1. **Drinking Water**

Regulatory Method	**Description**	**WSLH Method #**	**Accreditation**
EPA 524.2	Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Mass Spectroscopic Detection - EPA Method 524.2 - Revision 4.1 - (August 1995)	ESS ORG Method OC13251	WDNR, EPA
EPA 552.3	Determination of Haloacetic acids In Drinking Water By Liquid-Liquid Extraction, Derivatization and Gas Chromatography With Electron Capture Detection - EPA Method 552.3 - Revision 1.0 (July 2003)	ESS ORG Method OC12531	WDNR, EPA
EPA 537.1	Analysis of Perfluorinated Compounds in Drinking Water by HPLC-MS/MS-EPA Method 537.1, Version 1.0, November 2018	ESS ORG Method OC12731	NELAC

6.18.2. **Non-Potable Water**

Regulatory Method	**Description**	**WSLH Method #**	**Accreditation**
EPA 8260B	Volatile Organic Compounds by Purge-and-Trap using Capillary Column Gas Chromatography/Mass Spectrometry (GC/MS) - EPA Method 8260B - Revision 2.0 - (December, 1996)	ESS ORG Method OC13601	WDNR
EPA 8270E	Polynuclear Aromatic Hydrocarbons in Water by GC/MS analysis - SW846 Method 8270	ESS ORG Method OC16721	WDNR
SM5310B	Total Organic Carbon in Water	ESS ORG Method OC16602etal	NELAC, WDNR
EPA 8015C	Glycols in Water by Gas Chromatography Modified EPA Method 8015C – February 2007, Revision 3	ESS ORG Method OC16121	WDNR

6.18.3. **Solid and Chemical Materials**

Regulatory Method	**Description**	**WSLH Method #**	**Accreditation**
EPA 1010A	Flash Point – EPA 1010A (ASTM D93-80) Pensky Martin (OC17201)	ESS ORG Method OC17201 & OC17202	WDNR
EPA 1020B	Flash Point – EPA 1020B (ASTM D3278-78) Setaflash (OC17201)	ESS ORG Method OC17201 & OC17202	WDNR
EPA 9095	Paint Filters Liquids (OC17202)	ESS ORG Method OC17201 & OC17202	WDNR
EPA 1311	TCLP Analysis for VOCs	ESS ORG Method OC17402	WDNR
EPA 8260B	VOCs in Waste and Waste Oils	ESS ORG Method OC17601	WDNR
EPA 8260B	VOCs in Soil	ESS ORG Method OC15512	WDNR
EPA 9060A	Total Organic Carbon in Sediment by the Slurry Method	ESS ORG Method OC15604	NELAC, WDNR
EPA Method 8082A	PCB Aroclors in Soil/sediments	ESS ORG Method OC15103, OC15108, OC15109, and OC15129	WDNR
EPA Method 8082A	PCBs in Oil	ESS ORG Method OC17301	WDNR
SW846 Method 8270E	Polynuclear Aromatic Hydrocarbons in Soil and Sediment by GC/MS	ESS ORG Method OC15804	WDNR

6.18.4. **Air Sampling and Testing**

Regulatory Method	**Description**	**WSLH Method #**	**Accreditation**
TO-15	Toxic Organic Compounds in Ambient Air by Gas Chromatography/Mass Spectrometry in SIM/SCAN - Modified EPA Method TO-15	ESS ORG Method OC19161	NELAC

OPERATIONS: HORIZON DATA MANAGEMENT (HDM), SAMPLE RECEIVING, & CUSTOMER SERVICE

Table of Contents

7.1. Personnel

Table #1: Education and Experience

Name	**Title**	**Degree**	**Yrs. Exp.**
Kathleen Dax-Klister	Supervisor-Horizon Data Management	BS Environmental Science (Biological Resource Management emphasis) BS Animal Biology	9
Barbara Gaffney	IS Network Support Tech	HS Diploma	28
Jill Hamann	Environmental Program Associate	BA Environmental Studies	7
Sherry Kalamarz	Environmental Program Associate	HS Diploma	12
Tracy Hanke	EHD Operations Manager	BS Chemistry	26
DeVante Nelson	Environmental Program Associate	BS Life Sciences Communication	2
Matthew Strebel	IS Data Services Professional -	HS Diploma	10
Tony Gaffney	Environmental Program Associate	HS Diploma	2
Colton Forester	Environmental Program Associate	BS Biology and Outdoor Conservation, Natural Resources	1

7.2. Training

Please see section 1.10 of this QA manual for training requirements common to all EHD employees. In addition, each new employee will be taken through the Horizon Data Management (HDM) New Employee Training Checklist (M:\EHD\HDM\Training\New Employee HDM Training Checklist Rev1.doc).

Method specific training for HDM includes the following steps:

- Read the SOP's for data management methods.
- Observe an experienced employee perform the methods.
- Perform the methods while being observed by an experienced employee.
- Perform the method on own, with review by experienced employee until mistakes/questions are minimal.
- Perform the method on own.

These steps are documented, dated and initialed in the Horizon Data Management Method Training forms (M:\EHD\HDM\Training\Method Training Form ver1.doc). Forms are filed in the employees training folder.

7.3. Document Control System

Please see section 1.11 of this QA Manual for information about location and control of lab-wide and division level SOPs.

For the Horizon Data Management (HDM) department, all SOPs and documents are kept in electronic format on O:\SOP\EHD\HDM\Final and will be available to all staff members as "read-only" documents. Employees may print out if they wish to have their own copy at their workstation. Copies will be made available to customers or to regulatory agencies if requested.

SOPs are revised when necessary due to software changes, regulatory changes, etc. Each new revision will have a new revision number, an effective date, and will list the SOP it is replacing. Any archived documents 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. For detailed information regarding the control of documents, please see EHD HDM GENOP 001, "How to Write an SOP."

7.4. Sample Handling Procedures

Please see section 1.16 of this QA Manual for sample acceptance information on the divisional level. Also included is sample login and tracking information and sample ID generation in Horizon LIMS.

The Horizon Data Management Department is responsible for receiving and logging in Environment Health Division samples. See EHD HDM GENOP 116 Sample Receiving and Login.

HDM staff also does the Check-in of Inorganics and Metals Samples. The SOP for this procedure can be found in O:\SOP\EHD\HDM\Final.

Please see EHD GENOP 033, "Sample Acceptance Policy," which includes what to do if sample acceptance criteria are not met.

A spreadsheet was developed that lists required preservatives, minimum volumes, hold times, and acceptable bottle types for common Inorganic and Metals water tests: O:\SOP\EHD\ESS\Inorganic\Final\Bottle Types_Tests_Preserv_Vol_Inorg4910 one page.xlsx. More specific information can be found in the "Sample Handling and Preservation" sections of analytical SOPs.

Inorganic Chemistry/Metals sample disposal procedures, including documentation in Horizon as well as physical disposal techniques, are detailed in ESS INO GENOP 110, "Sample Disposal Protocol."

7.5. Data Review and Reporting

The Horizon Data Management Department deals with the demographic data associated with a sample (i.e. collector name, location, collection date, and sample auxiliary data). Horizon Data Management staff will also enter field data written on a labslip if applicable. The entry and review of the demographic data is detailed in the Departmental SOPs located in O:\SOP\EHD\HDM\Final.

After demographic and field data are entered and reviewed, the sample will report automatically once all analytical schedules have been finalized. Results are sent to appropriate parties either electronically, by fax or by mail, or any combination of the three. Report recipients and reporting options are designated within each client in Horizon.

7.6. Laboratory Facilities

The Horizon Data Management Department is spread throughout the Agriculture Drive Building including rooms 203 (water microbiology data entry), 118 (inorganic chemistry, organic chemistry and metals data entry), 114 (customer service and Supervisor's Office), 134 (Sample receiving room).

The Horizon Data Management Group uses the ChemWare Horizon Laboratory Information System (LIMS).

7.7. Instrumentation and Equipment

The following equipment is located in Rm. 134 and is used in the sample receiving process.

One fume hood: FH1-134, Air Sentry.
Used for adding acids to un-preserved samples.

Infrared Thermometer: Control Company
Ray 7 Serial #150402024
Ray 9 Serial #181021754
Ray 10 Serial #191975679

Zebra GK 420t label printers – provided by WSLH Office of Information Systems.

7.8. Laboratory Supplies and Chemicals

There are no laboratory supplies or chemicals used for the data entry portion of Horizon Data Management. The receiving function uses the following:

Bulk nitric acid – supplied by ESS Inorganics Department

Nitric acid and sulfuric acid vials – supplied by ESS Inorganics Department.

pH Paper – supplied by ESS Inorganics Department.

Drinking straws – ordered through UW MDS.

1x1", 1x2.5" labels - Nev's Ink

7.9. Preventative Action

On a weekly basis the following Horizon queues are reviewed by HDM staff: EDD, REC, REP, ADMN, BILL, CSRV, FLD, MAIL, TECH, UNRP. Staff looks for samples in these queues that are not completed yet but should be, samples that need further attention, and as general way to keep on top of what is "hanging out in the system".

7.10. Corrective Action

The guidelines for Customer Communications and Corrective Action in the HDM department are outlined in O:\SOP\EHD\HDM\Final\EHD HDM GENOP 114 ver 1 - Customer Communications and Corrective Action.doc.

7.11. Internal Audits

An annual internal audit will be conducted by QA staff using the Horizon Data Management System QA Checklist as well as the divisional systems checklist.

7.12. Standard Operating Procedures

Horizon Data Management Departmental SOPs are located in O:\SOP\EHD\HDM\Final.

Shipping

Table of Contents

8.1.Personnel

Table: Education and Experience

Name	**Title**	**Degree**	**Yrs Exp.**
Marten Metoxen	Env Program Associate	HS Diploma	13
William Beck	Inventory Control Coordinator	HS Diploma	29
Barb Woehrl	Shipping and Mailing Supervisor	BS Biology	24

8.2.Training

See general chapter 1.10.
Selected Shipping staff are certified to ship hazardous materials.
UW Madison-Office of Biological Safety-Bio HazMat Shipping Training:
US DOT 49 CFR part 172.704 and IATA DGR Sect. 1.5

Hazardous Materials Shipping Training: 49 CFR 172 Subpart H and IAT DGR 1.5

Annual Bloodborne Pathogens Training, on site.

HIPAA training on site.

Other training as appropriate not required.

8.3.Document Control System

Please see section 1.11 of this QA Manual for information about location and control of lab-wide and division level SOPs.

SOPs are revised when necessary due to software changes, regulatory changes, etc. Each new revision will have a new revision number, an effective date, and will list the SOP it is replacing. Any archived documents will also be available if required. Each item will be clearly labeled and dated so that it is apparent when the document was enforced.

Hard copies of UPS/Speedee reports are kept in drawer in room #132.

All posted purchasing receipts are kept in room 136 for five years then destroyed.

A copy of the monthly mail charges are filed in room 136, original is sent to Henry Mall Accounting services. Files are kept for 5 years, then destroyed.

8.4.Sample Handling Procedures

EHD Shipping GENOP 010 Loading Dock Receiving

8.5.Laboratory Facilities and Equipment

There is a loading dock and loading dock office, room 136. The ICC (Inventory Control Coordinator) and other shipping staff use this area to receive all incoming samples and supplies. Room 136 has two computer work stations and one Neopost mail machine used for all outgoing letter mail.

This room also stores unopened boxes of nitric and sulfuric acid in a cabinet. The cabinet is labelled.

On the dock itself are two flatbed carts and a hand jack. The shipping department has 5-6 wheeled carts for incoming and outgoing shipments of samples and for delivery of supplies.

Shipping room 132 is used for preparation of sampling kits, storage of sampling supplies and the shipping center. There are two computer stations, one with UPS, SpeeDee and USPS shipping software and one networked computer for misc. use.

There is a strapping machine for packaging boxes.

Room 134 Sample receiving room: used for processing environmental samples for the lab.

8.6. Laboratory Supplies and Chemicals

Bottles and other supplies are ordered by the Inventory Control Coordinator (ICC). The order is placed into an electronic purchasing program. The ICC receives the bottles and notifies the QA Coordinator. 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 50 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. The bottle types that are routinely used are listed below. In addition, other types of bottles and sample containers are checked as needed.

Plastic Quart

Plastic 1 liter

Plastic 500 mL

Plastic 250 mL

Plastic 60 mL

Plastic 150 ml sterile

Plastic 250 ml sterile

Other supplies include Kleenex, absorbent wipes and copy paper, mailers and cardboard boxes, labels, bags and office supplies.

Quality checks on bottles begin with the shipping staff. See section 12.0 of O:\SOP\EHD\ESS\Water Micro\Final\ESS MICRO QA 212 rev 3_Sample Bottle Sterility Calibration Fluorescence.doc, for shipping staff responsibilities.

Vendors Include but are not limited to:

Associated Bag
Dairyland PackagingUSA
Fisher
Grainger
Uline
Nevs Ink
MDS
Industrial Glassware
L&L Foods
Robin II

8.7.Internal Audits

The Shipping dept is part of the lab audit for accreditation for the WSLH.

See other depts for audit schedules.

8.8.Standard Operating Procedures

Shipping and Receiving SOPs are located at: O:\SOP\EHD\EHD CS SR Final and Draft.

8.9.Kit Production

The shipping department also creates testing kits and ships these kits to customers.

EHD Shipping GENOP 014, Kit Preparation (in progress)

EHD Shipping GENOP 003, Organic Chemistry mailer/Kit Preparation

EHD Shipping GENOP 004, Opening and closing receiving room

EHD SR GENOP 005 Water Microbiology SDWA Mailer

EHD Shipping GENOP 010, Loading dock receiving

EHD Shipping GENOP 011, Training form

EHD Shipping GENOP 012, Purchasing

EHD Shipping GENOP 013, SOP

EHD Shipping GENOP 015, Misc. dock activities

EHD Shipping GENOP 017, TECL Bottle request instructions

EHD Shipping GENOP 020, Customer communication and corrective action

8.10. Corrective Action

The Shipping dept will follow HDM sop:

O:\SOP\EHD\HDM\Final\EHD HDM GENOP 114 ver 1 - Customer Communications and Corrective Action.doc