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Author: Tania Biswas

Laboratory User Guide

INTRODUCTION

The Water Sciences Laboratory (WSL) is an analytical facility within the Nebraska Water Center and the Daugherty Water for Food Global Institute. It was originally funded through the Institute of Agriculture and Natural Resources (IANR). In 1990, IANR initiated a $400,000 renovation of an existing University of Nebraska-Lincoln East Campus building to provide a laboratory for specialized analyses, including trace levels of agrichemical compounds and environmental isotopes. The 6,000 square foot facility consists of six laboratories, several offices, a conference room, and staff/graduate student areas.

The location and working environment promote collaborative research on water-related projects involving professors and students from a wide variety of disciplines including Agronomy, Biological Sciences, Geology, Chemistry, Biological Systems Engineering, Entomology, Civil Engineering, and the School of Natural Resources. Students, staff and faculty using the facility may be familiar with available analytical services and equipment but must also understand procedures and protocols to be followed if they intend to work in the Laboratory. This guide is intended to provide an overview of the equipment, safety precautions, general procedures, and methods used, as well as expectations for all users of the facility. Routine standard operating procedures (SOPs) for lab management, general laboratory protocols, and analytical protocols are referenced in other parts of this guide. Very often, these protocols and procedures are simply a matter of good laboratory practice, to be followed in any analytical laboratory. This guide was created in order to promote uniformity and to preserve the quality control (QC) procedures for all data produced at this facility.

LABORATORY TRAINING

All users, including staff and students, must become familiar with the proper use and care of these resources by reading all applicable standard operating procedures (SOPs) after completion of the initial training. Training of individuals on specific instrumentation depends upon the level of use. Any individual using WSL equipment is responsible for its calibration and general upkeep. It is the user's responsibility to learn and understand the proper procedures to be followed when using equipment, document all use in equipment notebooks, and to immediately notify staff of any needed repairs or maintenance. In general, all WSL users must have documented training for the following:

- Laboratory, chemical, and compressed gas safety
- Laboratory record keeping
- Proper use and locations of common lab equipment (balances, micropipettes, reagents)
- Proper use of refrigerators and freezers throughout the building
- Locations and proper handling and restocking of commonly used supplies and solvents

- Proper handling and replenishment of compressed gas supplies
- Proper cleaning of glassware, equipment, and solvent disposal
- General laboratory organization and housekeeping

The training sequence for new WSL users and staff includes the following (all training must be signed off on and finished prior to working in the laboratory), and will be completed in order:

1. EHS Core Safety Training, available through instructor or on-line at http://ehs.unl.edu/training/online, and includes:
    - Core – Injury and Illness Prevention Plan (IIPP)
    - Core – Emergency Preparedness Training
    - Core – Bloodborne Pathogens
    - Core – Chemical Safety Training (4 units)
    - Personal Protection Equipment (PPE)
2. Additional EHS training modules determined by the **Training Needs Assessment** for EHS-Related Topics.
3. General WSL standard operating procedures (WSLSOP) and EHS Safe Operating Procedures (EHSSOP). This includes training related to analytical balances, micropipettes, and glassware, as well as other SOPs relating to the specific duties of the user.
4. Method specific WSL standard operating procedures (WSLSOP) related to the equipment and methods they will use.
5. A scheduled appointment with the Lab Manager to assess laboratory techniques, complete one or more proficiency tests, and complete a written exam.

Additional training for lab and data management at the WSL must also be completed prior to working in the laboratory. SOPs relating to this information can be acquired from the Lab Manager.

Safety Equipment

Eyewash stations are located in rooms 103C, 107, 203 and 205. A safety shower is located in the corrosion control lab (Room 205). Personal protective equipment (PPE), including eye protection, lab coats, face shields and ear protection, is ordered and provided to WSL users who need these items for the methods and procedures followed in the laboratory. At a minimum, eye protection is required of all WSL users. For more information, see the EHS SOP regarding Emergency Eyewash and Shower Equipment.

SAMPLE SUBMISSION

All client or student samples must be submitted with a properly completed Sample Submission Form. If necessary, they may also be accompanied by a Chain of Custody Form which legally documents transport and submission. Ensuring that these forms are filled out properly and completely will facilitate the movement of the samples through the laboratory workflow.

For more information, please refer to the Sample Submittal Form SOP.

LAB MANAGEMENT

As with many general safety procedures, the following habits and practices will maintain safe and high quality working conditions. These recommendations are designed for accident prevention, and all WSL users are responsible for seeing that these housekeeping policies are followed.

- All areas must be kept as clean as the work allows, and each WSL staff or student user is responsible for maintaining the cleanliness of his/her area.
- Regents and equipment items will be returned to their proper place after use, including while processing samples.
- Used glassware is always labeled during use. After use, they are transferred to cleaning areas, and are not allowed to accumulate on work areas.
- No chemicals or solutions are ever to be stored in fume hoods, on benches or tables, or on the floor.
- Use closed door chemical storage cabinets suitable for the material to be stored.
- All work done in fume hoods shall be performed in the "Safety Zone", (10 cm minimum from the sash). Close hood sashes at the end of the day.
- Stored items or equipment shall not block access to the fire extinguisher(s), safety equipment, or other emergency items.
- Hallways, passageways, and access to emergency equipment and/or exits must be kept dry and not obstructed in any fashion, including storage, equipment, phone or other wiring.
- All benches, other working surfaces, and floors will be cleaned at least weekly.
- Leave the balances and their surroundings clean.
- Monitor waste containers and notify EHS if they are full, in order to facilitate their removal.
- Be sure that all electrical equipment is powered off at the end of the day, and all equipment intentionally left on (i.e. ovens, block heaters) must be labeled with user and status (on for drying, etc).
- The oven in Room 203 should only be used for drying chemicals, or samples used for the Volatile Suspended Solids (VSS) or Total Suspended Solids (TSS) protocols.
- Reserve all instrument usage on QReserve.

QReserve and Sign-Up

All instrument usage in the Water Sciences Laboratory is scheduled through a website service called QReserve. Users are able to reserve usage of a piece of equipment during a specified day and time of their choosing. All students, staff, and faculty using the lab must reserve all of their instrument usage on QReserve. Please follow the steps below to sign up with QReserve:

1. Navigate to https://www.qreserve.com.
2. Create an account using your e-mail address and a password of your choosing.
3. On the Memberships page, search for the Water Sciences Laboratory. A picture of the outside of the building will confirm that the site is the correct one.
4. Use the join code **wsl16** to join the Water Sciences Laboratory QReserve site as a user.

For more information on how to use QReserve, please refer to the Equipment Reservation SOP.

Laboratory Glassware and Sample Containers

Most glassware is shared by all WSL users and it is critical that strict handling, cleaning, and storage procedures are followed. All WSL users are responsible for proper use, cleaning, and storage of glassware and sample containers. For detailed information pertaining to cleaning glassware and sample containers, please refer to the following SOPs:

- Laboratory Glassware Cleaning and Storage
- Plastic Cleaning for Trace Element Analysis
- Teflon Cleaning for Trace Element Analysis
- Cleaning of Sample Containers

Ordering and Receiving Supplies

Purchased equipment, supplies, reagents, standards, and other testing materials must be of sufficient quality so as not to adversely affect analytical results. Scientific vendors are regarded as resources or extensions of the analytical laboratory, and thus must adhere to the same standards of quality. The WSL has access to, and experience with, a wide variety of scientific manufacturers. Orders for equipment and supplies are generally placed either through an online procurement (EShop) system, or directly with the vendor if they are not serviced through EShop. All laboratory supplies, chemicals, and consumables are ordered by the Laboratory Director and Staff. WSL users are responsible for notifying the director or staff immediately when individual items are running low. Decisions on equipment repairs and replacement must be evaluated with respect to current and future use, along with availability of improved or lower cost alternatives. Staff may request chemicals and consumables through Quartzy.

When a chemical or consumable is received:

- List the item on the appropriate inventory list.
- Record the date and your initials on the packing slip.
- Mark the item as received on Quartzy.

Monthly Maintenance

Monthly maintenance for laboratory instruments and housekeeping occurs throughout the month, and laboratory users may be assigned particular maintenance tasks. Students and staff should sign and date each task as it is completed on the checklist located on the bulletin board outside Room 204. A copy of this checklist can be found in Box under Supporting Files.

24-hour Building Access

New lab users, who will need to use the facility outside of normal work hours (Mon-Fri: 8 am to 5:30 pm), can request 24 hours access to the WSL building by providing the following information to the Lab Manager:

1. Full name
2. NU ID
3. Primary Affiliation

Access will be granted after the new user has completed the basic lab training.

LAB POLICIES

Lab Space, Equipment, and Labware

- **NO** lab user can perform lab work (e.g. operate an instrument, prepare chemical solutions) before completing the required **basic and analytical proficiency training**.
- **ALL equipment** must be reserved on **Qreserve** before use (see 'Equipment Reservation' SOP). Laboratory staff have priority on equipment for analyzing client samples.
- Return chemicals and lab equipment (micropipettes, etc.) to **the correct location** after use.
- **Clean bench/work space** after use. Leave the balances and surrounding areas clean. Notebooks, papers, chemicals, glassware, etc. must all be removed from the area and put in the proper location.
- **Label** samples (including sub-samples) with batch sheet number and lab IDs. Keep sample containers from the same batch together, in a rack or tray, in the cold storage units.
- **ALL** glassware containing chemicals or solutions must be properly labeled with the chemical(s) name(s), concentration, your name, and the date while in use.
- **ALL** dirty labware (stainless steel spatulas, stir bars/rods, glassware, Teflon, etc.) must be placed in the Citronox soap baths, or filled with Citronox (especially for volumetric flasks, large graduated cylinders and beakers, etc.) and kept on the dirty side of the sink (the right-hand side) after use. After placing your own labware in the Citronox bath, wash an equal number of glassware that have been soaking in the bath, as a common courtesy (see 'Cleaning Labware' SOP).
- Every time any instrument is used, write down all relevant information in the **lab notebook**, such as the name of user, date, method run, any reagents or standards prepared, any troubleshooting issues faced, and maintenance performed.
- **Notify** staff when the **supply of any item runs low.** Since there can be many lab users requiring the same consumables, (chemicals, pipet tips, vials, gloves, etc.) the available stock may run low much faster than expected. A good rule to follow is to alert a staff member when there is less than half, left.
- **NO laboratory/analytical equipment can be removed** from the lab for any reason. The only exception to this rule is field equipment and sample containers.
- **ALL full time** lab users, will assist with general housekeeping duties and maintenance of instruments, routinely used by them in their research projects.

Lab User Code of Conduct

- The lab is a place where people from different backgrounds co-exist and work together to maintain a peaceful, helpful, and encouraging working environment. Please be respectful of everyone's personal space and privacy.
- Do not engage (monopolize) staff time to assist with researcher or student run projects after training has been completed. Limited assistance will be provided by lab staff as necessary.
- Do not perform other lab user's work for them at the expense of your own tasks.
- The use of any form of intimidation that might frighten or discourage a user from working in the lab will not be tolerated. If you see anyone acting in this manner, please report it to the Lab Manager or Director.

- Do not interrupt or interfere with new users or lab interns during their training procedures and/or instrument operation. If you see anyone doing so, report it to the Lab Manager.
- If you see another lab user performing any lab procedure incorrectly, or in an unsafe manner, report it to the Lab Manager.
- For reporting any general misconduct, lab space misuse, or equipment damage at the facility, it is advisable to include visual evidence.

ANALYTICAL PROTOCOLS AND METHOD VALIDATION

Unless otherwise specified, all procedures and analytical methods performed at the WSL are taken from a standard published reference for water or soil analyses such as Standard Methods for the Examination of Water and Wastewater (APHA, 1992), EPA Methods for Chemical Analysis of Water and Wastes (USEPA, 1983), or Methods of Soil Analysis (ASA, 1982). Many specialized analyses are performed at the WSL, such as pesticide residue analyses by isotope dilution and determination of 15N in nitrate. These protocols and all routinely performed procedures are documented in the Water Sciences Laboratory Standard Operating Procedures (WSLSOP). All Laboratory users should become familiar with the SOPs related to the analyses and methods they will be using prior to performing any work in the Laboratory. This Users Guide should help students, staff, and researchers to decide which SOPs pertain to their work, as well as introduce the resources available in this facility. Undergraduate students are regularly employed to assist WSL staff in labor intensive sample preparation methods, routine analysis, and general laboratory procedures. Students are trained in standard laboratory methods, and if time allows, permitted to explore other more specialized analytical procedures. Laboratory assistants always work under the direct supervision of WSL staff. Work must be thoroughly checked by the supervisor prior to allowing the student to produce data for WSL clients.

QUALITY ASSURANCE

Quality assurance is fundamental to the production of analytical data for scientific research. General quality assurance procedures used at the Water Sciences Laboratory are detailed in the WSL Quality Assurance Manual, written to conform to USEPA guidelines. Laboratory staff maintain and operate all equipment, train others in its use, and document methods used through preparation of standard operating procedures (SOPs). Other analytical equipment available to support water related research is also maintained, operated and validated through the use of SOPs and quality controls, including regular analysis of round robin samples and performance evaluations.

For more information, please refer to the Quality Assurance Manual.

DATA MANAGEMENT

Laboratory Notebooks

Bound laboratory notebooks are the preferred means for creating a permanent record of data, instrument readings, project activities, chain of custody reports, and experiments in the research laboratory. Validation of these records is accomplished by including the entry date and analyst signatures, or initials. Secondary review, signatures and dates provide further corroboration in accordance with most quality assurance protocols. A three-ring binder with consecutively numbered removable pages or

chronologically dated pages may be used as a means for keeping instrument print-outs, bench forms, sample forms, etc. in addition to the laboratory notebook. Pre-formatted tables and solution preparation sheets may be taped or pasted into notebooks if signed and dated by the technician. Avoid using temporary written records (paper towels, scraps of paper, loose leaf notebooks, etc.) and make all written notes in bound laboratory notebooks.

A laboratory notebook provides a permanent record of first hand observations, as well as a medium for performing calculations and organizing one's work. Quality record-keeping is indicated when someone other than the person making the entries can locate and interpret pertinent information. The following practices are recommended for keeping and maintaining laboratory notebooks.

General Notebook Etiquette

- Enter all data directly into the notebook in **ink** as it is acquired. Do not record data elsewhere for transfer to the notebook.
- Record sufficient information, so that someone else can understand at a glance what has been observed or performed. All relevant procedures, reagents, apparatus, sketches, conditions, references, etc. should be included.
- Title each page. Clearly label all entries with appropriate headings and units. Set up a data entry page prior to an experiment or procedure to facilitate direct entry of data.
- Record routine observations in less detail, unexpected or unusual observations in more detail. Repetitive information can be entered once in each book and referred to by page number.
- Delete entries by drawing a single line through them, so that they can still be read. Make corrections adjacent to the deleted entry, initial, and date.
- Write your full name and date on each entry of the notebook as it is completed. Mark blank pages or substantial portions of pages with a large "X" to indicate that they were intentionally left blank and cannot be used for additional data entry at a future date.

Types of Information to Record

An analyst or technician will need to keep track of the chemicals and solutions that are to be used for each protocol. If troubleshooting a method is required, often the first step is to evaluate or remake the chemical reagents.

Preparing Solutions:

- Record the chemical information including the name, manufacturer, lot number, and the amount used for the preparation of the solution.
- If using a prepared solution within your prep, note the solution name and the WSL RefID number (explained below).
- Each solution should have a label with the WSL reference ID number which links the laboratory notebook with the solution. This ID includes the lab notebook number, the page number, and the solution listing. For example, the first solution prepared in lab notebook WSL414 on page 46 would have the WSL RefID WSL-414-46A.

Preparing Standards:

- Record information about the stock standard, including the chemical name, concentration, and the WSL RefID.
- Note the concentrations for the working standards, the volume of stock standard required, and the volume of diluent.

Running an Instrument:

- Record information about the protocol and batch.
- List the directory location for data output and sample and/or batch file names.
- Record instrument data such as the air pressure, column pressure, etc.

Digital Output

Many of the instruments in the lab store digital data output for a run and the samples in a batch. All digital files should use the 9 digit protocol number (00_00_00.000) and date (YEARMODA) (ex: 18_01_01.003_20170119) naming convention when storing data or creating directories. This information should also be recorded in the laboratory notebook for quick reference.

REAGENT WATER

The reagent water at the WSL is distilled and deionized (DDW; surpassing ASTM Type 1) and may be organic-free (DDO) if drawn and stored properly. This high-purity water is expensive to generate and should not be wasted. It is produced by first distilling softened tap water using a Purewater C50 electric still located in Room 204. Distilled water for the building is stored in a stainless steel storage tank and is provided on demand to four laboratories in the building. Distilled water from faucets labeled with "DI" may be used in some laboratory applications which do not require high-purity, such as a pre-rinse for glassware. In the Wet Chemistry Lab (Room 203), a Barnstead Diamond cartridge system "polishes" distilled water by deionization and removal of colloids and trace organics to obtain ultra-high purity reagent water. Additional polishing systems are installed in the ICP-MS laboratory (Room 204B) and stable isotope mass spectrometry laboratory (Room 103C). Reagent water produced from this combination of distillation and polishing is suitable for the most critical applications, including HPLC. Inorganic impurities are continuously monitored in the polishing system by a water purity meter. Cartridges are changed if water resistance falls below 17.0 megaohm-cm, or if reagent blanks indicate cartridge bleed. Distilled deionized water (DDW) can be stored in 19-liter polyethylene carboys at various locations throughout the WSL. DDW is suitable for most analytical applications at the Laboratory except for trace-level organic analyses. Reagent water stored in polyethylene carboys cannot be considered organic-free since plasticizers can be leached from the container. If the reagent water is to be stored and used as organic-free (DDO), then it should be drawn and stored in pre-combusted glass containers.

For more information, please refer to the General Water SOP.

COMPRESSED GASES

Many instruments require compressed high purity specialty gases, while other lower cost gases are used for pneumatic actuators and evaporative concentration of extracts. Chromatographic or zero-grade helium is used in the Chromatography Laboratory as carrier gases for gas chromatographs. Zero-grade

nitrogen is used in the Wet Chemistry Laboratory for the carbon analyzer. Research-grade nitrogen, carbon dioxide, carbon monoxide, hydrogen, and argon are used in the Isotope Ratio Mass Spectrometry Laboratories for isotope gas standards. Because carbon monoxide is toxic, it is only used in a ventilated enclosure in Room 203C. Industrial-grade nitrogen is used for operating pneumatic valves on analytical equipment, and for concentrating organic solvent extracts. Industrial-grade oxygen and propane are used for glass-blowing. Acetylene is used in Room 203 as fuel for the atomic absorption (AA) spectrophotometer. Gas cylinder pressure should be checked often while using an instrument. If the supply pressure of any cylinder falls below 400-500 psi, notify the Director or staff member so that a replacement may be ordered. All cylinders MUST be clearly labeled with a gas cylinder tag marked "Full", "Part-full", or "Empty". Always remove the regulator and use a hand truck when moving a cylinder. Replace the empty cylinder as soon as a full one is available, and be sure that it is secured.

For more information, please refer to the EHSSOP Compressed Gas Cylinders in Laboratories.

FUME HOODS

There are six fume hoods located in the Water Sciences Laboratory, including one stainless steel hood designated solely for the use of perchloric acid. Hoods are periodically checked by Environmental Health and Safety (EHS) for face velocity. All hoods should be left on at all times unless otherwise instructed. Use of all corrosive acids at the WSL is restricted to the Corrosion Control Laboratory (Room 205). The perchloric acid hood, with its integral wash-down system and all stainless steel construction, may only be used for perchloric acid work. Fume hoods located in the Wet Chemistry Lab, and the two Isotope Ratio Mass Spectrometry Labs may be used to remove fumes when working with organic solvents. Do not use organic solvents in the Corrosion Control Laboratory (Room 205).

For more information, please refer to the EHSSOP Laboratory Hoods/Cabinet Identification and Use.

ANALYTICAL BALANCES

Perhaps the most important general purpose equipment used for most analytical work, is the analytical balance. The quality of all calibration standards prepared for quantitative analytical work depends on the continued accuracy and performance of these balances. Because it is a delicate and critical instrument, use of the Mettler Toledo XS205 Analytical Balance is restricted to trained and pre-approved WSL staff preparing calibration standards. Analytical balances should only be used for accurate weighing, such as in the preparation of calibration standards, or in gravimetric analyses when the weight must be made to the nearest 0.1 mg. All other types of weighing should be performed using one of the top-loading electronic balances.

For more information, please refer to the Balances SOP.

EQUIPMENT

Emerging Contaminants, Pesticides, and Other Organics

New methods based on mass spectrometry are among the most sensitive available for accurate measurement for very low concentrations of contaminants in water. The facility currently supports two gas chromatography (GC/MS) and two liquid chromatography tandem mass spectrometry (LC/MS)

systems, along with both manual and automated extraction systems, for organic contaminant analysis. The new GC/MS includes a purge and trap autosampler for volatiles, and a CombiPAL autosampler for automated solid phase microextraction and headspace sample introduction methods. Recently-developed methods include analysis of pharmaceuticals, steroid hormones, cyanotoxins, and herbicides in water and wastewater. Additional chromatography equipment includes a Waters 2695 HPLC with photodiode array and fluorescence detectors, older 600 HPLC with UV visible detectors, Trace Analytical Reduced Gas analyzer for low level hydrogen gas detection, and other gas chromatographers with thermal conductivity, electron capture and flame ionization detection.

Trace Metals and Stable Isotopes

An inductively coupled plasma-mass spectrometer (ICP-MS) is available for trace element and heavy metal analysis. The instrument can be interfaced with a liquid chromatograph system and hydride generator for elemental species determination. A microwave system is available for semi-automated digestion of samples. Stable isotope mass spectrometry, used for isotope fingerprinting of contaminants such as nitrate and in studying chemical reactions and metabolic processes of contaminants, is supported by two dual inlet and two continuous flow mass spectrometers with automated sample preparation systems.

Other Analytical Support

Routine laboratory analyses are supported by a variety of equipment. A Seal Analytical AQ2 discrete chemistry autoanalyzer and Lachat Quikchem 8500 flow injection autoanalyzer (FIA) are both available for automated water quality testing. An OI Analytical Model 1100 total organic carbon (TOC) analyzer uses persulfate oxidation for measurement of dissolved organic carbon. A Perkin Elmer AAnalyst 400 atomic absorption (AA) spectrophotometer and Lambda 25 UV-vis spectrophotometer can be used for analysis of a variety of metals, cations, and other substances dissolved in water. Samples and reagents are measured using electronic top-loading balances and analytical balances. A variety of meters are available for electrochemical measurements utilizing pH, redox, and ion selective electrodes (ISE). Other laboratory equipment includes ovens, a muffle furnace, a shaker water bath, a wrist-action shaker, a laboratory mill, ultrasonic probe, several centrifuges, and a wide variety of vacuum pumps and high-vacuum equipment.