North Carolina Department of Environmental Quality, Division of Water Resources,

Water Sciences Section, Intensive Survey Branch (ISB)

STANDARD OPERATING PROCEDURES MANUAL:

Physical and Chemical Monitoring

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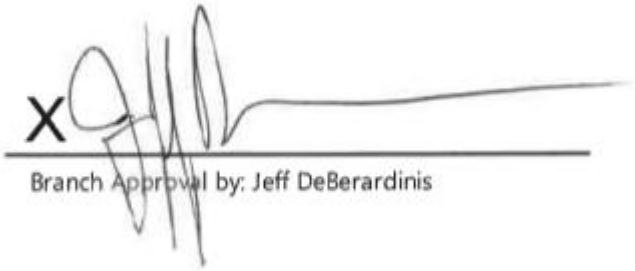
X 

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Reader Verification Page

I have read the ISB Standard Operating Procedures. I understand that these Standard Operating Procedures are written down to document the correct sampling procedures, techniques, and record keeping that all ISB staff should follow. By understanding and following the Standard Operating Procedures I am helping to reduce the inconsistencies and errors that can occur in water quality monitoring programs. By understanding and following the Standard Operating Procedures I am also contributing to producing quality data that can be used to make valid and sound decisions by others in North Carolina.

If I have any questions, I can contact the ISB Supervisor. A signed copy should be submitted to the QA Coordinator.

Signature

Title

Date

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INTENSIVE SURVEY BRANCH SOP REVISION LOG

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INTRODUCTION

This manual contains the standard operating procedures (SOP) employed by the North Carolina Division of Water Resources (DWR) to evaluate water quality. It is intended to encompass all aspects of routine physical and chemical water quality monitoring with the occasional sediment samples. Therefore, this manual is to be considered a working, dynamic guideline for DWR personnel. Efforts to improve current procedures will continue, and the manual will be revised periodically, as needed.

The primary goal of the manual is to promote the use of procedures that are consistent and reliable during field operations. All employees of the DWR staff are expected to be familiar with and to utilize these procedures as appropriate tools for water quality data collection. Because the procedures have been presented to cover a broad range of applications encountered in water quality monitoring, modifications may be necessary for specific conditions. Deviations from the procedures outlined in this manual, however, should be documented at time of collection.

These standard operating procedures apply to surface water, wastewater, and sediment. The manual details procedures for sample collection and handling, as well as methods for parameters that must be measured in situ.

Procedures are referenced at the end of each section. In addition, all references are compiled in Section XIII. Mention of trade names or commercial products does not constitute endorsement or recommendation for use by the Division of Water Resources.

These standard operating procedures will assist the Division of Water Resources in its efforts to monitor the waters of the state with increased accuracy and confidence.

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I. CONSIDERATIONS FOR WATER QUALITY SAMPLING

The purpose for collecting water samples is to obtain a representative portion of the surface water body (i.e. lake, river, or stream) being evaluated. Valid results depend upon:

- Ensuring that the sample obtained is a true representative of the surface water body being evaluated.
- Employing proper sampling, handling, and preservation techniques.
- Properly identifying the collected samples and documenting their collection in permanent field records.
- Maintaining sample chain-of-custody procedures, if necessary.
- Protecting the collected samples by properly packing and transporting (shipping) them to the appropriate laboratory for analysis.

1. GENERAL WATER QUALITY SAMPLING CONSIDERATIONS

The following factors and procedures shall be considered and/or implemented in planning and conducting all water quality sampling operations. All of these factors and procedures should be considered in view of the specific objectives and scope of each individual field investigation. It is advisable to discuss sampling with the DWR Chemistry Lab during the planning process to verify and coordinate methodologies, analytical capabilities and timing of sample submittal.

Selection of parameters to be measured

The parameters to be measured are usually dictated by the purpose of an investigation and should be selected based upon required monitoring conditions (NPDES permits for example) or upon the investigator's knowledge of the problem.

Dissolved and particulate sample fractions

A sample is generally composed of dissolved and particulate fractions. When it is necessary to analyze samples for individual fractions, filter samples in the field using appropriate equipment and techniques. (i.e. dissolved phosphorous).

Required sample volumes

The volume of sample obtained should be sufficient to perform all the required analyses with an additional amount collected to provide for any quality control needs such as split samples or repeat examinations. DWR Laboratory sample submitting guidance document can be found at the Sample Submittal Guidelines – DWR Water Sciences Section Chemistry Laboratories

Sample handling

After collection, all samples should be handled as little as possible. All personnel should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, personnel should ensure that the ice does not submerge the sample containers, thereby preventing cross-contamination. This is extremely important, especially if the samples are to be used in an enforcement action. Alternatives that can be used to prevent contamination include the use of frozen water containers instead of ice, double wrapping the sample containers in trash bags surrounded with ice or placing the samples back in the station Ziplock bag prior to submerging in ice.

Special precautions for sampling trace amounts of contaminants

Most contaminant compounds are detected in the range of parts per billion or parts per trillion; therefore, extreme care must be taken to prevent contamination of samples.

The following precautions shall be taken when trace contaminants are of concern:

- When sampling surface waters, the aqueous sample should always be collected prior to any sediment sample collection. Sample collection should always be performed using clean equipment and proper collection techniques.
- Sample collection activities should proceed progressively from the least contaminated area to the most contaminated area (if this fact is known).
- When possible, samples should be collected facing upstream to avoid contamination from sampling activities.

Procedures for identifying potentially hazardous samples.

Samples that are either known or thought to be hazardous should be identified clearly on both the sample tag and field sample sheet. Also Use ArcGIS Survey123's geopoint and multimedia features to capture location-specific data and observations in real time. see **ArcGIS Survey123**
Information explaining the hazard, i.e., corrosive, flammable, poison, etc., shall also be listed. If a sampling hazard is identified, only continue if a properly trained staff member is present and if appropriate safety equipment is available. Follow procedures found on the Water Sciences Section Fish Kill web page when sampling fish kill events

Collection of auxiliary data

Record all auxiliary data using the ArcGIS Survey123 "Intensive Survey Lakes Field Form" at the time of sample collection. Required fields include flow measurements (enter numeric value in the designated field), site photographs (attach via the form's camera or file upload option), meteorological conditions (select from predefined options or enter manually), additional observations (use the text field for notes). The form automatically records the sampling location via a geopoint question. Verify the GPS coordinates match the intended site before submission. Data is synced to the ArcGIS platform in real time (if online) or stored locally for later upload (if offline), replacing paper field records.

Time records

All records of time shall be kept utilizing local time in the military (2400 hour) time format and shall be recorded to the nearest five (5) minutes unless more precise measurements are dictated. This shall be documented on the sample label, the field sheet and using the ArcGIS Survey123 "Intensive Survey Lakes Field Form.

Transporting and shipping of samples

Samples may be hand delivered to the appropriate laboratory, or they may be shipped by common carrier. Chain of custody may be necessary during and after sample collection. All personnel must be aware that certain samples could be classified as hazardous materials and as such, could be regulated by the U.S. Department of Transportation under the Transportation Safety Act of l974. These regulations are contained in Title 49, CFR, Parts ll0-ll9 (An example would be concentrated acid, azide, etc.). A copy of these regulations is available online at: Code of Federal Regulations.

2. SURFACE WATER SAMPLE SITE SELECTION

Selection of a surface water sampling location for water quality studies is based on many factors. These include but are not limited to, study objective, water use, point source discharges, nonpoint source discharges, tributaries, changes in stream characteristics, types of stream bed, stream depth, turbulence, presence of structures (weirs, dams), accessibility, safety concerns, and personnel. When such sampling locations are in estuarine systems, tidal effects must be considered when determining sampling locations. When assigning a station, the naming convention of river basin, unique identifier (creek/lake), and/or 3-digit numerical number should be used (NEUXX000).

Before sampling is conducted, a site assessment should be conducted to locate suitable sampling locations. For lotic systems bridges and piers are normally good choices as they provide ready access and permit water sampling at any point across the width of the water body. When sampling from bridges, samples should be taken from the upstream side; however, this may alter the nature of water flow and cause sediment deposition. Additionally, bridges and piers are not always located in desirable locations with reference to waste sources, tributaries, etc. Wading for water samples is not recommended in lakes, ponds, and slow-moving rivers and streams. However, when wading for sample collections in slow-moving water bodies, it is best to work from downstream stations to upstream sampling points, especially when samples are taken in close proximity. In slow-moving lentic or deep-water systems, a boat is usually required for sample collections and sampling should allow for the possible presence of stratification. Assessment of the lake sampling location should utilize the deepest point of the given section that is well mixed and provides the best representative data. Generally, on smaller lakes this can be found near the dam. On larger lakes with multiple sampling locations, samplers can take advantage of channels or underwater contours to maximize depth.

All samples should be taken within 100m of the fixed site location based on GPS coordinates.

3. SAMPLE COLLECTION TYPES

Grab sample

A grab sample is a sample collected over a period of time not exceeding 15 minutes. A grab sample is normally associated with water or wastewater sampling. However, soil, sediment, liquid hazardous waste samples, etc., may also be considered grab samples; no particular time limit would apply for the collection of such samples. These samples are used to characterize the medium at a particular point in time; and are generally associated with instantaneous water or wastewater flow data.

Conditions when a grab sample is conducted:

- Water or wastewater stream is not continuous (e.g., batch discharges or intermittent flow)
- Characteristics of the water or waste stream are known to be constant
- Sample is to be analyzed for parameters whose characteristics are likely to change significantly with time (i.e., dissolved gases, bacteria, etc.)
- Sample is to be collected for analysis of a parameter such as oil and grease where the compositing process could significantly affect the observed concentrations
- Data on maximum/minimum concentration are desired for a continuous water or wastewater stream
- When NPDES permit effluent monitoring specifies grab collections

Grab sample collection methods

- A grab sample is collected at 0.15 m below the water surface. Gloves should be worn for personal safety and to prevent sample contamination.
- Direct- A sample bottle is placed 0.15 m below the water surface while pointing the bottle mouth up current or towards the bow of a boat when lake sampling.
- Intermediate Grab Sampling Device- These devices are any type of sampling device that holds the sample prior to pouring it into a sample bottle and are used when sampling from a bridge or area that the water cannot be reached. The collection end is placed 0.15 m below the water's surface with the open end facing upstream or up current. An example is a Polyethylene Dipper (Figure 1) or other custom-made devices.

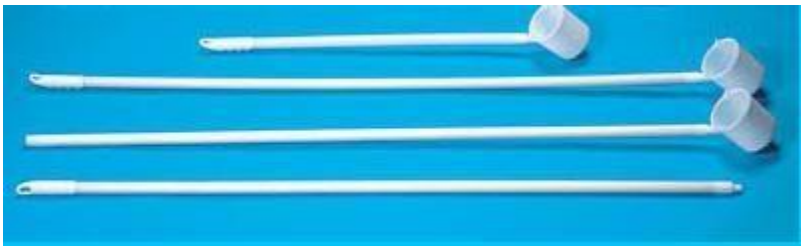


Figure 1. Polyethylene dipper typically used by DWR

Parameters that are always grab samples:

- Metals
- Phenol
- Sulfide
- Oil and grease
- Volatile organics
- Bacterial
- Chlorine residual
- Other dissolved gases
- PFAS

Composite sample

Composite samples are used when average concentrations are of interest and are associated with average flow data (where appropriate). Composite sampling is employed when the water or wastewater stream is continuous, or it is necessary to calculate mass/unit time loadings or when analytical capabilities are limited.

Timed integrated

A timed integrated composite sample contains discrete samples taken at equal time intervals over the compositing period. A timed composite may be collected continuously. A timed composite is collected continuously or with constant sample volume and a constant time interval between samples.

Flow proportional integrated

A flow proportional composite contains discrete samples, taken proportional to the flow rate over the compositing period. Proportional composites are

collected with constant sample volume and constant time interval between samples proportional to stream flow.

Area Integrated

Area integrated composite samples are collected over a predetermined area of a waterbody, usually from the same depth. Samples are collected then composited into one representative sample.

Vertical spatial composite (Photic Zone Sampling)

Vertical spatial samples are composite samples (a.k.a. photo zone, depth-intergraded samples) taken within the photic zone. The photic zone is found between the surface and twice the Secchi reading (Section III.6). Samples are collected by lowering and raising an integrated depth sampling device such as a Labline water sampler (Figure 2) at a steady speed to obtain a representative water sample within the photic zone. Prior to sampling, the Labline should be rinsed 3 times with station water to avoid sample contamination. Decontaminate with phosphate-free detergent between water bodies as needed. Graduated measuring line used to determine the depth of the sampler should be measured starting from the point where water enters the water sampler.

Figure 2. Labline sampler for photic zone (vertical spatial) composites

Split sample

A split sample is a sample that has been portioned into two or more containers from a single collection device. Portioning assumes adequate mixing to assure the split samples are, for all practical purposes, identical. Devices such as churn splitters should be rinsed with ambient site water prior to field use for composite split samples, cleaned with phosphorous free cleaner after use and rinsed with deionized water before storage. Composite sample volume in the splitter should allow for ¾ of the total aliquot to be split with ¼ remainder. This prevents aeration of the sample during dispensing. Sample agitation should be performed for 2 minutes prior to sample split to ensure homogeneity of the composite. The spigot or valve should be purged prior to dispensing the first sample. Churning should be continuous while samples are being dispensed from the spigot. As the composite volume in the churn is reduced, churning rate should increase.

Duplicate sample

Duplicate samples are collected simultaneously from the same source, under identical conditions but in separate containers. This measures the accuracy/precision/repeatability of samples.

Replicate sample

Two or more samples representing the same population characteristic, time, and place, which are independently carried through all steps of the sampling and measurement process in an identical manner. Replicate samples are used to assess total (sampling and analysis) method variance. Replicate samples should be taken 5 minutes after the regularly scheduled sample.

Control sample

A control sample is collected upstream or above a source or site to isolate the effects of the source or site on the particular ambient medium being evaluated according to the study plan for that particular project.

Background sample

A background sample is collected from an area, water body, or site like the one being studied but located in an area known or thought to be free from the pollutants of concern. Background samples should be taken from well-mixed areas, not necessarily midstream to represent normal conditions.

Sample aliquot

A sample aliquot is a portion of a sample that is representative of the entire sample.

Scoop sample

A scoop sample is one that is taken in a non-quantitative way for identification only, such as a surface skim, a filamentous clump or rock scrape. All aquatic macrophyte samples are taken as scoop samples.

Physical Water Quality Measurements (In-Situ Field Measurements)

Physical parameter measurements recorded by a field meter such as a Hydrolab, YSI, or In-situ multiparameter probe. Parameters that are considered physical water quality samples or parameters are:

- Depth (m)
- Temperature (°C)
- Salinity (ppt)
- Conductivity (µS/cm)
- pH
- Dissolved Oxygen (mg/L)

These may be measured at various depths depending on the water body and needs of the study being performed.

4. AUTOMATIC SAMPLERS

The Instrumentation Specialties Company (ISCO) model 2700 (Figure 3), model 3700, and two model 6700 wastewater samplers are portable devices designed to collect up to 24 separate sequential samples or perform composite sampling.

More complex sampling such as multiplexing, storm spaced sampling, interfacing with a variety of equipment such as flow meters, field printers, and lap top computers can also be accomplished with the 3700 model. Both sampler models must be supplied with 12 VDC power from one of four sources: an ISCO AC power pack, an ISCO nickel-cadmium battery pack, an ISCO sealed lead acid battery, or an external 12 V direct current source (such as an automotive or marine battery).

Refer to the ISCO 2700 and 3700 instruction manuals for detailed description of operating procedures. **It is important to verify the configuration of these samplers prior to placing them in the field** (Instrument Specialties Company 1988,1991).

Figure 3. ISCO automated samplers

5. MANUAL SAMPLING

Manual sampling is usually employed when collecting grab samples and immediate *in-situ* field analyses samples. However, it may also be used, in lieu of automatic equipment, over extended periods of time for composite sampling. All samples should be taken within 100m of the fixed site location.

Manual Sampling Technique - Flowing Water

The best method to manually collect a sample is to use the actual sample container. This eliminates the possibility of contaminating the sample with an intermediate collection container. **The actual sample container must always be used for collecting oil and grease and bacterial samples**.

Sampling with an intermediate

If the water or wastewater stream cannot be physically reached, an approved intermediate sampling device may be used. Approved intermediate sampling devices include Labline samplers or Van Dorn type samplers. When a sample needs to be collected in the sample container such as grease or oil, a cage sampler can be used for the out-of-reach locations (Figure 4).

Figure 4. Cage sampler used in the DWR Ambient Monitoring Program

Surface Grab Sample by Hand

Field personnel should wear gloves when sampling. Being careful not to disturb the nearby sediment, quickly submerge properly cleaned collection vessel (bottle or intermediate sampling devise) into the water or wastewater stream to <0.2 m below the surface of the water with the container facing into the flow of the water. Ensure that the mouth of the container is far enough below the surface to not accidentally collect debris, scum, etc. that is floating on the water. Rinse out the container via this procedure at least twice before the sample is collected (exceptions to this rinsing procedure may exist if preservatives are present in the sampling container and for certain analyses such as oil and grease). When collecting the sample, carefully cap the container underwater if possible. This minimizes contamination from airborne matter. Be careful not to touch the underside of the cap.

If an intermediate sampling device is used, the container employed to collect the initial sample must be rinsed three times with sample water and must be constructed of a material that meets requirements of the parameter(s) being investigated.

From EPA 1669. “Surface samples are collected using a grab sampling technique. The principle of the grab technique is to fill a sample bottle by rapid immersion in water and capping to minimize exposure to airborne particulate matter.”

Sampling Utilizing a Pump

Some types of analyses require the use of a pump when sampling. If a pump is used, it is imperative that it be pre-purged and all components of the pump that come into contact with the liquid be properly cleaned to ensure the integrity of the sample.

Surface Grab Sample from Bridge

Field personnel should wear gloves. For normal sample parameters, metal bridge samplers and bottles do not need to be field rinsed before sampling.

Attach the appropriate sample containers to the bridge sampler using several rubber bands (Figure 4). It may help to include a few twists in the rubber band on either side of the bottle so that the bottle is more secure. Be sure to arrange the bottles so that weight will be fairly distributed once the bottles are full. The

bridge sampler may start spinning as it is lowered; it can be helpful to slowly sink a few inches of the bridge sampler in the water to stop the spinning before fully lowering the entire sampler below the water. Lowering the entire sampler below water without stopping spinning momentum may cause bottles to come loose. Be sure to avoid hitting the bridge with the sampler, particularly as the bridge sampler is being raised back up from the water.

When sampling from bridges, samples should be taken from the upstream side of the bridge, when possible, to avoid contamination from metal bridges and/or debris trapped by the bridge structure.

Manual Sampling Technique

Again, the best method to manually collect a sample is to use the actual sample container. This eliminates the possibility of contaminating the sample with an intermediate collection container. The actual sample container must always be used for collecting oil and grease and bacterial samples.

Surface Grab Sample from Boat

Field personnel should wear gloves. Quickly submerge the sample container to <0.2 m below the surface of the water while pointing the container mouth up current or towards the bow of a boat. Ensure that the mouth of the container is far enough below the surface to not accidentally collect debris, scum, etc. that is floating on the water. Cap the container underwater, if needed. This minimizes contamination from airborne matter. Carefully cap the container without touching the underside of the cap. Avoid sampling near boat bilge pump discharges, gasoline vents, and boat motors. Be sure to leave the correct amount of headspace for the sample you are collecting.

Photic Zone Sampling

Field personnel should wear gloves. Photic zone samples are a type of vertical spatial composite sample used to collect a representative sample of the entire photic zone. Photic zone samples are also sometimes referred to as depth-integrated samples. The photic zone is the uppermost portion of the water column between the surface and the lowest depth that sunlight reaches. DWR estimates photic zone depth as twice the Secchi depth reading.

If the bottom depth is less than the photic zone depth, then the bottom depth is reported as the sampling depth for photic zone samples. Where the bottom depth is less than the photic zone depth, the depth-integrated sampling

devices should be lowered to the bottom carefully to not disturb the bottom sediment.

Photic zone samples are collected by lowering and raising a depth-integrated sampling device at a steady speed to obtain a representative water sample within the photic zone. Depth-integrated sampling devices are sometimes called Lablines, which was the name of a specific device produced in the past by a specific manufacturer that is no longer in business (Figure 2). However, the term “Labline” is still commonly used by DWR staff to refer to this style of sampler.

Prior to sampling, the depth-integrated sampling device should be rinsed 3 times with station water to avoid sample contamination from previous stations. Using surface water to rinse the labline is sufficient to remove any water that may have been in the labline from the previous site.

To collect a sample, slowly lower the depth-integrated sampling device at a steady rate down to the photic zone depth (twice the Secchi depth reading). Then slowly raise the depth-integrated sampling device at the same steady rate up through the water column. Bubbles should emerge from the depth-integrated sampling device as the device fills up with a composite sample water between the surface and the photic zone depth. The speed of lowering/raising the sampler should be sufficient so that the depth-integrated sampling device is almost (but not completely) full when it is brought back to the surface. This ensures the entire photic zone is sampled.

Field Replicate Sampling

Field replicates are two or more samples representing the same population characteristic, time, and place, which are independently carried through all steps of the sampling and measurement process in an identical manner. Replicate samples are used to assess total (sampling and analysis) method variance. ISB only collects replicate water samples. Field meter and Secchi disk measurements are not replicated. Ten percent of environmental samples are assigned a field replicate. Replicates are assigned on a monthly basis with an effort to create a diverse sampling subset representative of the total sampling pool.

All replicates and environmental samples are reviewed annually, and Relative Percent Difference (RPD) is calculated for replicate and environmental sample pairs. The ISB Program are the only people who qualify replicate and associated environmental samples. The WSS Chemistry Laboratory reports

the replicate and environmental sample results, but they do not qualify the data based on replicate performance criteria. They may qualify the data based on receipt, analysis, and verification guidelines in their QA/QC documents. The ISB Program matches the replicates with the associated environmental samples. They calculate RPD and look at the results in relation to the PQL. The RPD is calculated for all environmental and replicate sample pairs. An environmental sample is considered to fail QC performance criteria for duplicates or replicates when all the following conditions are true:

- The environmental sample result is greater than 5 times the Practical Quantitation Limit (PQL).
- The associated QC sample result is greater than 5 times the PQL.
- The calculated RPD between the environmental sample result and QC sample result is greater than 25%.

An environmental sample that fails duplicate or replicate performance criteria is qualified with the qualifier code “J2”.

6. SPECIAL SAMPLE COLLECTION PROCEDURES

Priority pollutants

Priority pollutant detection limits are usually in the range of parts per billion, thus extreme care avoiding contact with skin or unapproved materials must be exercised to ensure sample integrity.

All containers, composite bottles, tubing, etc., used in priority pollutant sample collection should be cleaned as described in Section VI.

When possible, the sample should be collected directly into the appropriate sample container. If the material to be sampled cannot be physically reached, an intermediate collection device may be used. This device should be a Teflon, glass or stainless-steel vessel or Teflon tubing via a peristaltic type of pump. The device should be cleaned as described in Section VI.

When an automatic sampler is employed for priority pollutant collection, the procedures described in Section I concerning collection of organic and metal samples with automatic samplers should be used.

Bacterial sampling

Samples for bacterial analysis should always be collected directly into the prepared glass or plastic sample container. Everything possible (e.g., "wear gloves, avoid touching the rim") must be done to avoid contamination through physical contact with the inside of the cap or bottle and mouth of the bottle.

1. Hold the bottle near the base.
2. With cap still on, plunge the bottle, neck downward, below the surface and turn until the neck points slightly upward. The mouth should be directed toward the current.
3. Uncap the bottle without rinsing. Collect sufficient sample volume for analysis, as specified by the laboratory.
4. Recap immediately while underwater.

Immiscible liquids/oil and grease

Oil and grease may be present in wastewater as a surface film, an emulsion, a solution, or as a combination of these forms. **The designated sample container must always be used for collecting oil and grease samples.**

As it is very difficult to collect a representative oil and grease sample, due to variable distribution in wastewater, the inspector must carefully evaluate the location of the sampling point. The most desirable sampling location is the point where greatest mixing occurs (e.g., near turbulence or discharge points) Quiescent areas should be avoided. Because losses of oil and grease will occur onto the sampling equipment, the collection of a composite sample is impractical. Collect individual samples at set intervals and analyze them separately to determine average concentrations

Volatile Organics Analyses (VOA)

Samples to be analyzed for volatile organics should be stored in the appropriate vials to prevent contamination and loss of sample. To verify proper sample container requirements, consult the DWR Chemistry Laboratory website.

The current methodology calls for 40 ml screw cap septum vials with a Teflon silicone disk in the cap. The disks should be placed in the caps (Teflon side down) in the laboratory prior to the initiation of the sampling activities. Extra disks should be carried during field sampling in case of loss of the disks previously placed in the caps.

When there is no chlorine present in the sampled waterbody a 40ml VOA vial pre-preserved with 1:1 HCL by the Central Laboratory should be used for collection. A VOA sample should be preserved with ascorbic acid and 1:1 HCL whenever there is chlorine present or if it is not known if chlorine is present.

7. WASTEWATER SAMPLING

General considerations

Important procedures for obtaining a representative wastewater sample include:

- Collecting the sample at a location where the wastewater is mixed. Therefore, the sample should be collected near the center of the flow channel, at a depth between 0.4 - 0.6 m total depth, where the turbulence is at a maximum and the possibility of solids settling is minimized. Skimming the water surface or dragging the channel bottom should be avoided.
- Doing cross-sectional sampling when sampling from wide conduits or within a mixing zone. Dye may be used as an aid in determining the most representative sampling point(s).
- If manually compositing a sample, thoroughly mix individual samples before pouring the individual aliquots into the composite container.

Site selection

Where applicable, wastewater samples should be collected at the location specified in the NPDES permit.

Influent

Influent wastewaters are preferably sampled at points of highly turbulent flow in order to ensure adequate mixing. When possible, influent samples should be collected upstream from side stream returns.

Effluent

Effluent samples should be collected at the site specified in the permit, or if no site is specified, at the most representative site downstream from all entering wastewater streams prior to discharge into the receiving waters.

Pond and lagoon sampling

Generally, composite samples should be employed for the collection of wastewater samples from ponds and lagoons. Even if the ponds and lagoons have a long retention time, composite sampling is necessary because of the tendency of ponds and lagoons to short circuit. However, if dye studies or past experience indicate a homogenous discharge, a grab sample may be taken as

representative of the waste stream; but in all cases, sampling should be consistent with permit requirements.

Sampling techniques

All techniques are covered in Section IV ISB Standard Operating Procedures and in the NPDES Compliance Sampling Inspection Manual.

Additional guidance can be found on the EPA website.

II. FIELD MONITORING

1. DATA SHEETS

A field sheet is used to submit a sample(s) to the DWR Chemistry Laboratory. They are generated in Labworks or EQuIS by ISB staff. Survey 123 is also used to collect metadata and notes from field operations. Tablets have access to the Survey 123 app where the "Intensive Survey Lakes Field Form" may be found, or online.

Field Data Sheets

Pre-logged field sheets are generated via Labworks by ISB staff with much of the information filled out ahead of time including the lab sample number, location code, DWR region, river basin, location description, priority, water matrix, location type and analytes requested. These sheets have spaces for information to be filled out by the sampler including collector, date and time of collection, Secchi depth, sample depth, ambient surface water temperature, and preservative lot numbers.

You may also find additional blank forms.

Use a black indelible ink pen to mark on the sheets.

- Blue ink will not scan well into Laserfishe
- Do not use Wite-out® or pencils on field data sheets
- If errors occur when filling out the field data sheet, **corrections can be made by crossing a single line through the error and entering the correct information. All corrections must be initialed and dated**. If the correction is to indicate that you did not sample for an analysis on the sheet, notate with "NS" in addition to crossing out the analysis and initialing and dating the correction.

Write legibly and within the allotted space.

A separate form is used for sediment, soil and tissue, as well as any parameters that are to be processed at the Asheville Regional Laboratory. Access the DWR Chemistry website to acquire the appropriate lab form.

Contract labs will have their own forms; consult lab prior to sampling for any special requirements.

Be sure to secure lab sheets(s) in a watertight container before shipping (usually a zip bag).

Survey 123

Survey 123 is available to Samplers as an app and on sampling tablets or on the ARCGIS website.

The "Intensive Survey Lakes Field Form" is used to collect metadata and notes/ comments relating to each sampling site.

Recorded parameters include: waterbody, station code, and lab number, date and time of sample collection, Secchi depth, ambient water surface temperature, level calibration checkbox, parameters collected, weather information, replicate sample information, and additional parameters collected

If an algal bloom is detected (pH of 8 or above, and/ or DO above 110%) samplers will check the appropriate box on this form and complete an additional Survey 123 form "Internal Reporting Fish Kill and Algal Bloom"

Field meter (multiparameter sonde) calibration forms are to be entered on the form "ISB AMS RAMS Field Meter Calibration Sheet" upon completion of out-cal procedures.

VuSitu and the FTP File Storage

Physical water quality data are no longer hand-recorded to data sheets in the field. These data are currently recorded using meter-specific applications on a Bluetooth-enabled tablet and uploaded to an FTP server where they can later be downloaded, reviewed and pre-processed by the ALMP coordinator. Following this pre-processing, data are again reviewed, further processed and ultimately uploaded to Labworks or EQuIS by the Environmental Specialist II responsible for data management within ISB (Database Administrator).

Physical water quality data collected by the multiparameter sondes (meters) are recorded via the VuSitu app on the field tablet.

- Samplers will begin recording the meter's deployment through the water column at the surface reading.

- A surface reading will be marked at 0.15-0.20 meters and every subsequent 1m increment up to 10 meters in depth. Readings will only be marked following stabilized readings at the desired depth.
- After the 10m increment, samplers will mark readings at 5m increments (15, 20, 25, and 30 meters)
- If at any point the meter reaches the bottom of the lake, samplers will raise the sonde slightly to avoid sediment interference, mark the bottom depth once reading has stabilized, and stop the recording process.
- Recordings are only taken on the “downcast”
- Files are saved first on the tablet’s Main Storage, then the TurboClient app is used to upload the files to the FTP.

North Carolina Division of Water Resources
Laboratory (Water Sciences Section)

Water Sample Collection & Submittal Form

Visit ID: (optional) | Tag ID

Lab Use Only:
Laboratory Sample Number:
Date Received: M/D/Y
Time Received: (24 hr format)
Received By:
Delivery Method: State Courier / Hand Delivered / Other:

Location Description:
Location Code:
County:
Collector:
DWR Region: (based on county)
DWR Office: (or agency name)
River Basin:
Date (m/d/y): (begin/end)
Time (24 hr): (begin/end)
Notes:

Priority:	Water Matrix:	Location Type:	
Ambient	Surface	River/Stream	Lake
Routine	Ground	Estuary	Canal
Compliance	Waste	Stormwater	Influent
COC	Blank	Monitoring Well	Filter Blank
Emergency	Solution	Effluent	Trip Blank
QA	Foam	Field Blank	Water Supply
Method Development		Other:	

Sampling Method: Grab / Composite / Other
Chlorinated / De-chlorinated in Field
Filtered in Field
Dissolved analysis: Enter "DIS" in check-boxes for parameters
Sample Depth:
Secchi Depth:
Login file:
Temperature on Arrival (°C):

Lab Comments:

Lot #s / Collector Comments:

Field Parameters *(optional)* : Preservative : Y | Water Temp (°C): | pH (s.u.): | Dissolved Oxygen (ppm): | Conductivity (µmhos/cm): | Salinity (ppt):

Microbiology Parameters (Chemically Preserved)	Preservative
Coliform: Fecal MF	B - C
Coliform: Total MF	B - C
TOC - Total Organic Carbon	A - G
Microbiology Parameters (Not Chemically Preserved)	**Preservative**
Alkalinity, as CaCO3, to pH 4.5/8.3	A
BOD: Biochemical Oxygen Demand, 5-day	A
cBOD: Carbonaceous BOD, 5-day	A
Specific Conductance, at 25 °C	A
Turbidity	A
Other Parameters	**Preservative**
pH	Y
Nutrients Parameters (Chemically Preserved)	**Preservative**
Ammonia as N (NH3-N)	A - C - D
Nitrate-Nitrite as N (NO3+NO2-N)	A - D
Total Kjeldahl Nitrogen as N (TKN)	A - D
Total Phosphorus as P (TP)	A - D
Nutrients Parameters (Not Chemically Preserved)	**Preservative**
Nitrite as N (NO2-N)	A
Nitrate as N (NO3-N calculated)	-
Orthophosphate as P (PO4) (must be field filtered)	A - Z
Cyanotoxins	**Preservative**
Microcystin	A

Wet Chemistry Parameters (Chemically Preserved)	Preservative
COD: Chemical Oxygen Demand	A - D
Cyanide, Total	A - H - P
Hexavalent Chromium (Cr6+)	A - I
Oil and Grease, HEM, Total Recoverable	A - D
Phenols, Total Recoverable	A - D - M
Sulfide	A - J
Wet Chemistry Parameters (Not Chemically Preserved)	**Preservative**
Bromide	A
Chloride	A
Fluoride	A
Sulfate	A
Chlorophyll a	A
Color: ADMI	A
Color: Platinum Cobalt	A
MBAS (surfactants)	A
Residue: Total (*Total Solids*)	A
Residue: Volatile/Fixed, Total	A
Residue: Suspended (*Suspended Solids*)	A
Residue: Volatile/Fixed, Suspended	A
Residue: Total Dissolved (TDS)	A
Silica	A
Tannin & Lignin	-

Metals Parameters	Preservative
Aluminum (Al)	E
Antimony (Sb)	E
Arsenic (As)	E
Barium (Ba)	E
Beryllium (Be)	E
Cadmium (Cd)	E
Calcium (Ca)	E
Chromium (Cr), Total	E
Cobalt (Co)	E
Copper (Cu)	E
Iron (Fe)	E
Lead (Pb)	E
Lithium (Li)	E
Magnesium (Mg)	E
Manganese (Mn)	E
Mercury (Hg)	E
Molybdenum (Mo)	E
Nickel (Ni)	E
Potassium (K)	E
Selenium (Se)	E
Silver (Ag)	E
Sodium (Na)	E
Strontium (Sr)	E

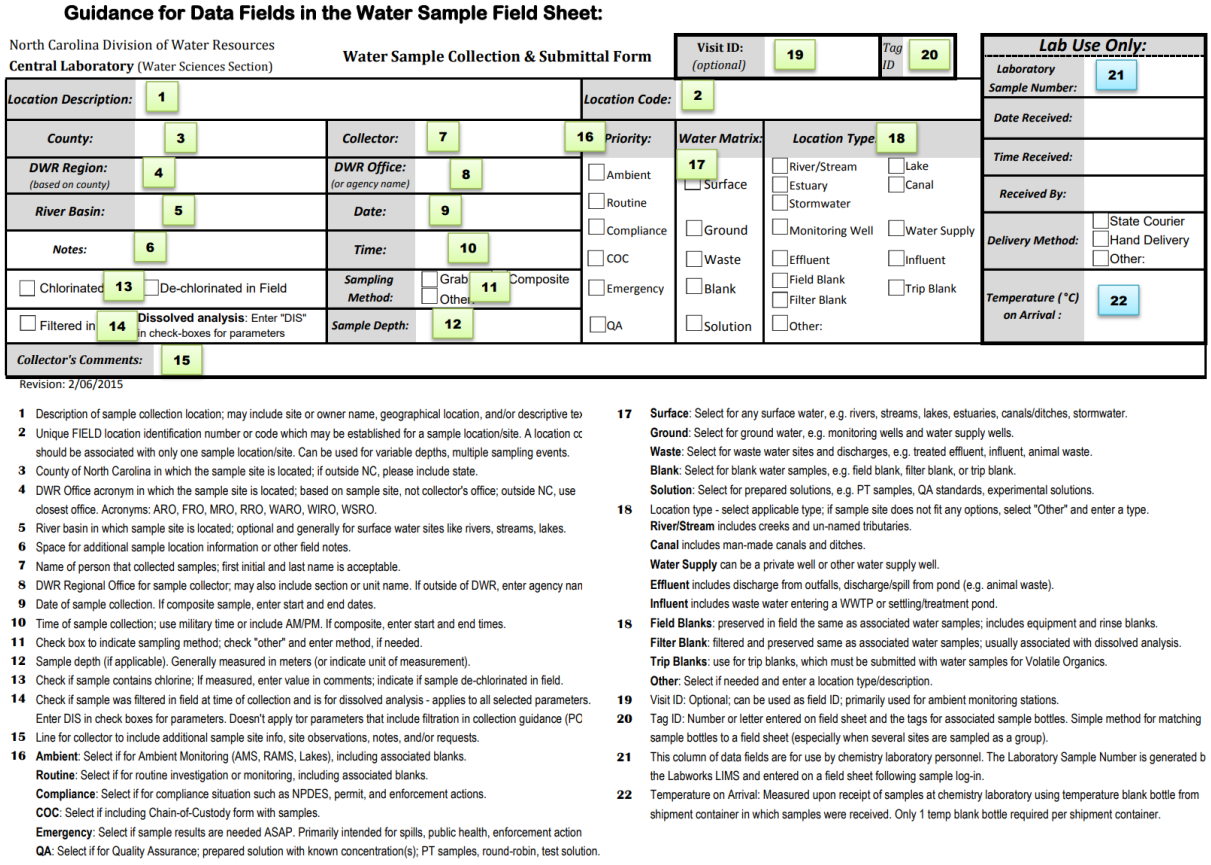
Metals Parameters Con't	Preservative
Thallium (Tl)	E
Tin (Sn)	E
Titanium (Ti)	E
Vanadium (V)	E
Zinc (Zn)	E
Mercury 1631, low-level	-
Boron (B)	E
Hardness, Total as CaCO3 - by titration	E
Organics Parameters	**Preservative**
Acid Herbicides	A - C
Organochlorine Pesticides	A - C
Organonitrogen Pesticides	A - C
Organophosphorus Pesticides	A - C
PCBs (polychlorinated biphenyls)	A - C
Semi-Volatile Organics (BNAs)	A - C
TPH Diesel Range	A - C
Volatile Organics (VOA)	A - C - F - L
1,4-Dioxane	C - L
TPH Gasoline Range	A - C - F - L
Perfluorinated Compounds (PFAS)	A - T
Biological	**Preservative**
Phytoplankton / Algae	A - R

Preservative Legend (circle above as needed): **(A)** cool ≤6°C, **(B)** cool <10°C, **(C)** 0.008% $Na_2S_2O_3$ [when chlorine is present], **(D)** H_2SO_4 to pH <2, **(E)** HNO_3 to pH <2, **(F)** HCl to pH <2, **(G)** H_3PO_4 to pH <2, **(H)** 6N NaOH to pH >10<11, **(I)** $(NH_4)_2SO4$ pH=9.3-9.7, **(J)** zinc acetate & NaOH to pH to >9, **(L)** leave no headspace, **(M)** ferrous ammonium sulfate [when chlorine is present], **(P)** ascorbic acid [when chlorine is present], **(R)** Lugols, **(T)** Trizma [when chlorine is present], **(Y)** analyzed within 15 minutes of sample collection, **(Z)** filtered in field within 15 minutes using 0.45um pore size

Required: 1) Collector Initials and Date, 2) Circle each preservative option utilized next to each parameter.

Revision: 4/3/2023

Figure 5. Blank Surface Water Field Sheet



Guidance for Data Fields in the Water Sample Field Sheet:

North Carolina Division of Water Resources
Central Laboratory (Water Sciences Section)

Water Sample Collection & Submittal Form

Visit ID: *(optional)* 19 | Tag ID 20

Lab Use Only:
Laboratory Sample Number: 21
Date Received:
Time Received:
Received By:
Delivery Method: State Courier / Hand Delivery / Other:
Temperature (°C) on Arrival : 22

Location Description: 1 | Location Code: 2
County: 3 | Collector: 7 | Priority: 16 | Water Matrix: 17 | Location Type 18
DWR Region: *(based on county)* 4 | DWR Office: *(or agency name)* 8
River Basin: 5 | Date: 9
Notes: 6 | Time: 10
Chlorinated 13 / De-chlorinated in Field | Sampling Method: Grab / Composite 11 / Other
Filtered in 14 **Dissolved analysis**: Enter "DIS" in check-boxes for parameters | Sample Depth: 12
Collector's Comments: 15

Priority: Ambient, Routine, Compliance, COC, Emergency, QA
Water Matrix: Surface, Ground, Waste, Blank, Solution
Location Type: River/Stream, Estuary, Stormwater, Lake, Canal, Monitoring Well, Water Supply, Effluent, Influent, Field Blank, Filter Blank, Trip Blank, Other:

Revision: 2/06/2015

1 Description of sample collection location; may include site or owner name, geographical location, and/or descriptive te
2 Unique FIELD location identification number or code which may be established for a sample location/site. A location cc should be associated with only one sample location/site. Can be used for variable depths, multiple sampling events.
3 County of North Carolina in which the sample site is located; if outside NC, please include state.
4 DWR Office acronym in which the sample site is located; based on sample site, not collector's office; outside NC, use closest office. Acronyms: ARO, FRO, MRO, RRO, WARO, WIRO, WSRO.
5 River basin in which sample site is located; optional and generally for surface water sites like rivers, streams, lakes.
6 Space for additional sample location information or other field notes.
7 Name of person that collected samples; first initial and last name is acceptable.
8 DWR Regional Office for sample collector; may also include section or unit name. If outside of DWR, enter agency nan
9 Date of sample collection. If composite sample, enter start and end dates.
10 Time of sample collection; use military time or include AM/PM. If composite, enter start and end times.
11 Check box to indicate sampling method; check "other" and enter method, if needed.
12 Sample depth (if applicable). Generally measured in meters (or indicate unit of measurement).
13 Check if sample contains chlorine; If measured, enter value in comments; indicate if sample de-chlorinated in field.
14 Check if sample was filtered in field at time of collection and is for dissolved analysis - applies to all selected parameters. Enter DIS in check boxes for parameters. Doesn't apply tor parameters that include filtration in collection guidance (PC
15 Line for collector to include additional sample site info, site observations, notes, and/or requests.
16 **Ambient**: Select if for Ambient Monitoring (AMS, RAMS, Lakes), including associated blanks.
Routine: Select if for routine investigation or monitoring, including associated blanks.
Compliance: Select if for compliance situation such as NPDES, permit, and enforcement actions.
COC: Select if including Chain-of-Custody form with samples.
Emergency: Select if sample results are needed ASAP. Primarily intended for spills, public health, enforcement action
QA: Select if for Quality Assurance; prepared solution with known concentration(s); PT samples, round-robin, test solution.
17 **Surface**: Select for any surface water, e.g. rivers, streams, lakes, estuaries, canals/ditches, stormwater.
Ground: Select for ground water, e.g. monitoring wells and water supply wells.
Waste: Select for waste water sites and discharges, e.g. treated effluent, influent, animal waste.
Blank: Select for blank water samples, e.g. field blank, filter blank, or trip blank.
Solution: Select for prepared solutions, e.g. PT samples, QA standards, experimental solutions.
18 Location type - select applicable type; if sample site does not fit any options, select "Other" and enter a type.
River/Stream includes creeks and un-named tributaries.
Canal includes man-made canals and ditches.
Water Supply can be a private well or other water supply well.
Effluent includes discharge from outfalls, discharge/spill from pond (e.g. animal waste).
Influent includes waste water entering a WWTP or settling/treatment pond.
18 **Field Blanks**: preserved in field the same as associated water samples; includes equipment and rinse blanks.
Filter Blank: filtered and preserved same as associated water samples; usually associated with dissolved analysis.
Trip Blanks: use for trip blanks, which must be submitted with water samples for Volatile Organics.
Other: Select if needed and enter a location type/description.
19 Visit ID: Optional; can be used as field ID; primarily used for ambient monitoring stations.
20 Tag ID: Number or letter entered on field sheet and the tags for associated sample bottles. Simple method for matching sample bottles to a field sheet (especially when several sites are sampled as a group).
21 This column of data fields are for use by chemistry laboratory personnel. The Laboratory Sample Number is generated b the Labworks LIMS and entered on a field sheet following sample log-in.
22 Temperature on Arrival: Measured upon receipt of samples at chemistry laboratory using temperature blank bottle from shipment container in which samples were received. Only 1 temp blank bottle required per shipment container.

Figure 6. Field sheet completion guidance. Downloaded as a part of the DWR [sample submission guidance document] on 10/23/2025

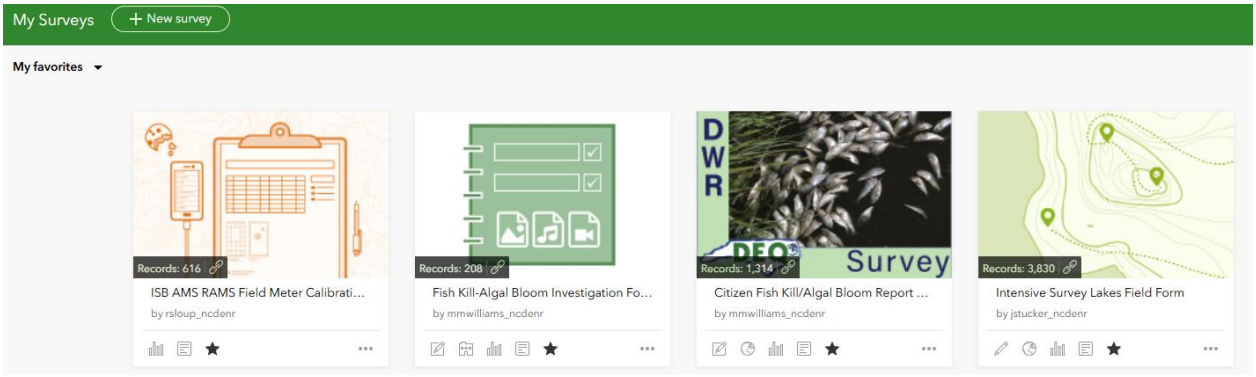


Figure 7. Select Survey123 Forms. From left to right: ISB/AMS/RAMS Field Meter Calibration Sheet, Fish Kill and Algal Bloom Investigation Form, Fish Kill and Algal Bloom Report Form, Intensive Survey Lakes Field Form

2. LABELS AND TAGS

Labels and tags must be completed with pens that are indelible ink or permanent ink, specifically the ink should not run or smear when wet. Many ballpoint pens use indelible ink conforming to ISO 12757-2, which means that they are resistant to acid, water, alcohol and UV radiation. Examples of pens safe to use on labels and tags include ballpoint pens and sharpies. Some gel pens claim to be permanent ink, but gel pens should not be used on labels and tags as the writing will smear when wet.

Do not use pencils or whiteout to complete labels and tags.

Information on a sample label and/or tag should include:

- Location Code/Water Body or Location/Station ID
- Date & time of collection
- Analyses to be conducted (parameter)
- Types of preservative(s) used
- Collector Name should be listed as first initial and full last name (e.g., “J. Doe”).

If preservative is listed on the tag is used, then the preservative should be initialed with first and last initial to indicate preservative was added.

The dates and times of collection, location code, collector name, and all other information on the container labels must match the field sheets.

Any deviations from the specified information pre-populated on labels should be noted on the labels and/or field sheet.

> **If a mistake is made while writing out labels, tags or field sheets, the mistake should be crossed out with a single line, then initialed (first and last initial) and dated.**

There are several different labels and tags available to field personnel. The ALMP Coordinator may send out pre-printed water-proof adhesive labels to the corresponding field personnel. **Labels and field sheets can also be printed by field personnel from WSS Ambient Lakes Monitoring Program SharePoint site.**

If possible, waterproof labels such as Avery 5522 should be used. The ALMP uses Avery 5522 labels (Waterproof Labels with Ultrahold® Permanent Adhesive, Sure Feed™, TrueBlock®, Laser, 700 Labels, 1-1/3" x 4" (5522)).

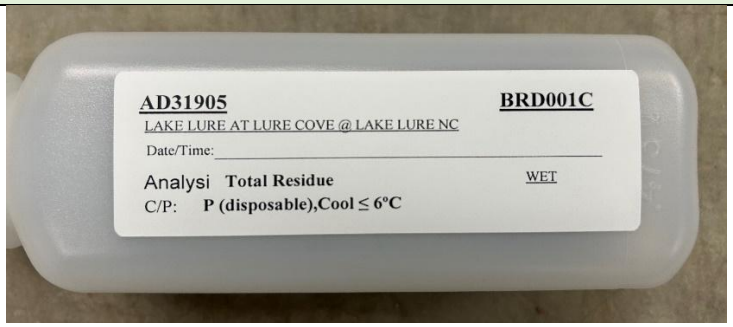
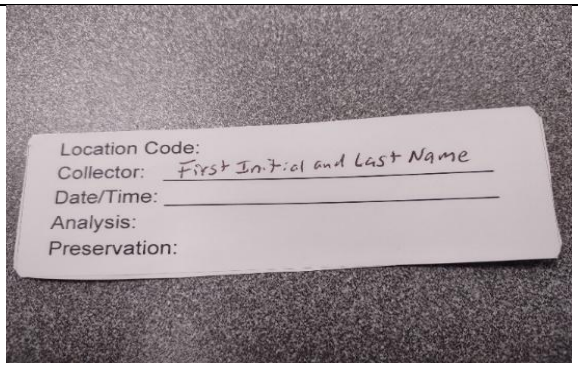
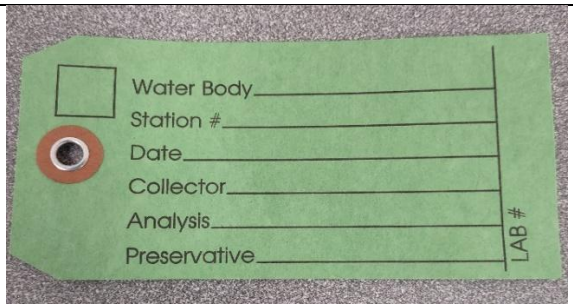
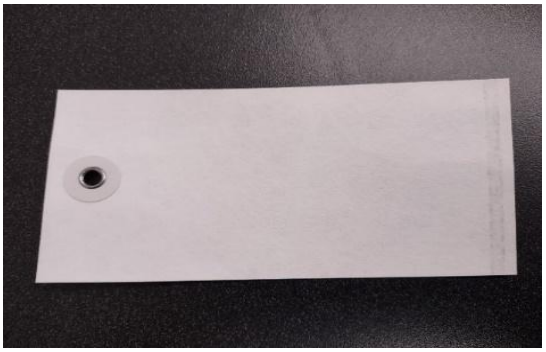
Label or Tag type	Label or Tag picture
ALMP-Generated / Pre-Printed Label	
Generic Adhesive Label	
Green Tag	
White Tag	4.75 x 2 3/8 #5 White tag, Tyvek 

Figure 8. Common labels and tags for sampling

Adhesive Labels vs. Tags

The WSS Chemistry Laboratory requests that adhesive labels should not be applied to reusable bottles such as those for chlorophyll-a (brown 500 mL wide-mouth plastic bottle), fecal coliform (sterilized and pre-preserved 250 mL plastic bottle), and cyanide (dark 250 mL plastic bottle). Reuseable bottles used for chlorophyll-a (brown 500 mL wide-mouth plastic bottle), fecal coliform (sterilized and pre-preserved 250 mL plastic bottle), and cyanide (250 ml plastic bottle) parameters require the attachment of tags with rubber bands (not adhesive labels).

Adhesive labels can be applied to disposable plastic and single-use bottles. Parameter bottles that can be labeled with adhesive labels include alkalinity, color, fluoride, chloride, bromide, sulfate, hardness, metals, nutrients, oil and grease, pesticides, semi-volatiles, and turbidity.

1,4-Dioxane, sulfide, and VOAs require multiple vials per sample at each station and must be secured together in a manner that keeps the individual sample multiple-vial set together and protected.

Vials should be placed in bubble wrap bags to prevent contamination from water in the cooler and breakage. Ziploc bags may be used if bubble wrap bags are not available. Ziploc bags may be used to protect the vials from water/ice contamination but may not provide protection from breakage. The adhesive protective tape should be removed from the bubble wrap bag and the top folded over to seal the bubble wrap bag. Use both Ziploc bags and bubble wrap bags if possible.

Three options for labeling VOA, 1,4-dioxane, and sulfide vials are:

- Adhesive label attached to a tag. Tag is attached to the vials with a rubber band. Set of vials with rubber banded tag with label is placed in a Ziploc bag and then into a small bubble wrap bag. Bubble wrap bag should be sealed by removing the protective adhesive strip and fold the top over to secure the vials in the bubble wrap bag.
- Adhesive label attached to a tag. Tag with the vials is placed in a Ziploc bag and then the Ziploc bag with the vials and tag placed into a small bubble wrap bag. Bubble wrap bag should be sealed by removing the protective adhesive strip and fold the top over to secure the vials in the bubble wrap bag.
- Adhesive label attached to a Ziploc bag. Vials are placed in a Ziploc bag with the label attached to a Ziploc bag. The vials and Ziploc bag with the label

attached to the Ziploc bag is then placed into a bubble wrap bag. Bubble wrap bag should be sealed by removing the protective adhesive strip and fold the top over to secure the vials in the bubble wrap bag.

If no adhesive label is available, field personnel must write on a blank label or fill out a tag with the appropriate information.

Figure 9. Labeling Multiple Vials as a Set with Rubber Band, Tag, and Label

Photic Zone Composite Sampling vs. Grab Samples

Routine ALMP samples are either photic zone or surface grab samples depending on the site and ALMP directions. Most ALMP samples are photic zone samples except for hardness, PFAS, and 1-4 Dioxane, which are typically sampled at sites near water intake structures or dams. Hardness sample bottles are marked with an “S” or “X” on the lid of the sample bottle to indicate surface grab collection. Labels for surface grab samples are not marked differently than photic zone labels, so it is the responsibility of the sampler to know which samples are collected at the surface (hardness, PFAS, and 1-4 Dioxane).

3. CHAIN-OF-CUSTODY PROCEDURES

All samples submitted to the WSS Chemistry Laboratories must be accompanied by appropriate documentation. However, when samples are collected for legal or evidentiary purposes, a Chain of Custody (COC) must be established. This process requires that every stage of a sample's handling, including collection, storage, transport, and receipt by the laboratory—be recorded in writing or through approved procedures. The documentation must provide a continuous record of who possessed the sample at each step to ensure traceability, protect sample integrity, and prevent tampering. The ISB Supervisor will inform sampling staff when a COC is necessary, such as for special studies, legal cases, or projects involving external laboratories. Additional COC needs may be required at the discretion of the ISB Supervisor. For further details, refer to Appendix 9 and the WSS Chemistry Laboratory Sample Submission Guidance Document.

4. FIELD INSTRUMENTS

ISB uses a wide array of instrumentations for recording water quality parameters. Currently, In-Situ is the main manufacturer used. Instructions for use, calibration, and maintenance as written by the manufacturer should always be followed at a minimum, unless otherwise directed by the QAPP. Per the ISB QAPP, sampling meters are calibrated before each use, which differs from the manufacture's recommendation. DWR produced a guidance sheet that outlines basic calibration, maintenance, and acceptance criteria for meters commonly used by DWR (Appendices 1-5). All meter guidelines and guidance sheets found in this document are supplementary too and not a replacement for the manufacturer's directions. Consult the manufacturer's instructions for use and maintenance not outlined in this document. In-Situ manufacturer manuals are found online by selecting the model and then selecting "Manual". Water Quality Multiparameter Sondes | Multiprobes

In-Situ Field Meter Calibration Guidelines

The ISB Program has developed calibration steps and guidance that should be followed to keep calibration procedures, record keeping, and data consistent over time. Calibration procedures for In-Situ meters are found in Appendix 1.

- Calibrations shall be performed in a clean, stable environment. Field calibrations are not recommended and should be avoided if possible. If calibrations are performed in the field, note that on the calibration sheet. Field calibrations for DO may be required with large changes in atmospheric pressure associated with altitude changes.
- Always use fresh, commercially made standard solutions to calibrate field meters. Always check the expiration date of the standard solutions. Do not use expired standard solutions.
- Well or tap water should be used to store probes. Do not use distilled water or deionized water to store probes. pH 4 solution may be used for long-term storage.
- Never pour decanted or used standard solutions back into the original standards bottle.
- Standard solutions used to calibrate/check the meter should be poured into the used standard/rinse solution bottles for further rinsings.

- Used standard/rinse solutions used to rinse the probes during calibration/checks may be discarded down the drain. Most calibration standards kits have two bottles of each standard. One bottle of new fresh standard from the standards box used to calibrate/check the probe. The second bottle is the used standard/rinse solution used to rinse the probes before calibration/checks.
- Protocol is to rinse the probe three times with used standard/rinse solution before adding new standard solution to calibrate/check the probe. Used standard/rinse solutions are used to rinse the probe three times and reduce the amount of new standard used and save money.

Annual Thermistor Checks (Review in annual revision)

At a minimum, the thermistor check will be conducted once per year.

The annual In-Situ Factory Calibration in September through December of each year will be used for an additional check point but not used to qualify data.

The In-Situ Field Meter Temperature Probes are starting to fail 3 years after purchase, so it is imperative to perform more thermistor checks throughout the year in order to reduce the number of possible data qualifications based on thermistor check failures.

Thermistor Check Data Entry

The thermistor results should be recorded in the ArcGIS Survey 123 form called “AMS and RAMS Thermistor Check”.

Thermistor Check Performance Criteria.

The performance criteria for a thermistor check is ≤ 0.5 °C difference between the field meter temperature and the NIST-certified digital thermometer temperature for both the ambient and cool water temperature checks. A thermistor check failure results when the check does not meet the performance criteria (> 0.5 °C) at either one temperature (ambient or cool water) or both temperatures (ambient and cool water).

The performance criteria for thermistor checks are from the North Carolina Wastewater/Groundwater Laboratory Certification Branch Approved Procedure for the Analysis of Temperature.

“All compliance temperature measurements must be made with a National Institute of Standards and Technology (NIST) traceable temperature-measuring device that has a demonstrated accuracy of ± 0.5 °C and equilibrates rapidly.”

"To check a compliance temperature-measuring device, compare readings at two temperatures that bracket the range of compliance samples routinely analyzed against a Reference Temperature-Measuring Device and record all four readings. The difference between the readings from both devices must be ≤ 0.5 °C."

Field meter measurements that fail a thermistor check are qualified with the qualifier code J12.

Pre-sampling and Post-Sampling Meter Use Guidance

All field meters shall be calibrated before sampling (pre-sampling) and checked after sampling (post-sampling). Post-sampling checks (sometimes referred to as post-calibration or out cal) are completed after daily sampling activities on the day of sampling. Post-sampling checks are completed to determine if the meter drifted out of acceptable calibration ranges. Pre-sampling calibration is to make sure the field meter is calibrated correctly within acceptable calibration ranges before sampling.

Pre-Sampling Calibration QA/QC

Any meters that fail pre-sampling calibration should not be used in the field. Have the meter fixed and/or use another meter that passes pre-sampling calibration QA/QC criteria.

Failed Field Meter Post-Sampling Check Response Action

Anytime there are two field meter post-sampling check failures, or one pre-sampling calibration failure, field meters should not be used for sampling and fixed or replaced as soon as possible.

Field Meter and Probe Temperature Issues

When calibrating during pre-sampling calibration and checks during post-sampling checks, wait for the probe to reach room temperature. For instance, on hot summer days, let the probe cool down before starting the post-sampling check. During sampling trips, field meters and probes should be kept inside of vehicle cabs and out of the sun to avoid extreme temperatures.

Manufacturers' specifications for standards are usually stated at 25°C. Calibrating or checking standards at temps different than 25°C or at different temperatures than what the probe was calibrated earlier in the day will cause numbers to vary and may be out of range.

Probe manufacturers provide temperature compensation in the software. Even with the temperature compensation, the probe manufacturers recommend calibrating at temperatures close to the sample temperatures.

From In-Situ pH and ORP Sensor guidance document. *"Temperature compensation is provided in the software to account for measurements taken at temperatures different from the calibration temperature. For most accurate results, try to calibrate at the same temperature as the expected sample temperature."*

Calibration Sheets (Hard Copy and Digital ArcGIS Survey123 Forms).

The new hard copy field meter calibration sheet (Figure 11, used after June 2024) uses calibrated readings to compare to standards. The first page is the pre-sampling calibration form and instructions, while the second page is post-sampling checks and instructions.

The old hard copy calibration sheet had errors comparing initial readings to standards or table values and had initial readings. Initial readings (before calibration) should be observed, and the meter needs to be serviced if initial readings are erratic or if the calibrated meter reading is not within acceptable calibration ranges (not initial readings). Initial readings are indicators that things may be wrong, but initial readings are not quality control checks. The quality-control check is the calibrated meter reading.

Hard Copy Field Meter Calibration Sheet	**Dates of Use**
"Water Quality Monitoring Field Meter Calibration Sheet" in various formats and editions.	Prior to July 2024
ISB Field Meter Calibration Sheet	After June 2024

ArcGIS Survey123 Digital Field Meter Calibration Sheet.

The new digital field meter calibration sheet in ArcGIS Survey123 (after April 2024) is called "ISB AMS RAMS Field Meter Calibration Sheet" (Figure 10).

The new digital ArcGIS Survey123 field meter calibration sheet form and the new hard copy calibration sheet "ISB AMS RAMS Field Meter Calibration Sheet" are

exactly the same. Transferring data from the new hard copy calibration sheet to the ArcGIS Survey123 app should involve the same questions and order of the calibration steps.

Table 1. Dates and Versions of Field Meter Calibration Forms and Sheets.

Digital Field Meter Calibration Sheet Notes	Dates of Use
Hard Copy "Water Quality Monitoring Field Meter Calibration Sheet" in various formats and editions.	Prior to July 2024
Digital Hard Copy "Water Quality Monitoring Field Meter Calibration Sheet" saved in ArcGIS Survey123 as "Meter Calibration Sheet – Beta"	September 15, 2020, to July 2024 (Approximately)
Digital ISB Field Meter Calibration Sheet saved in ArcGIS Survey123	After April 2024 (Approximately)

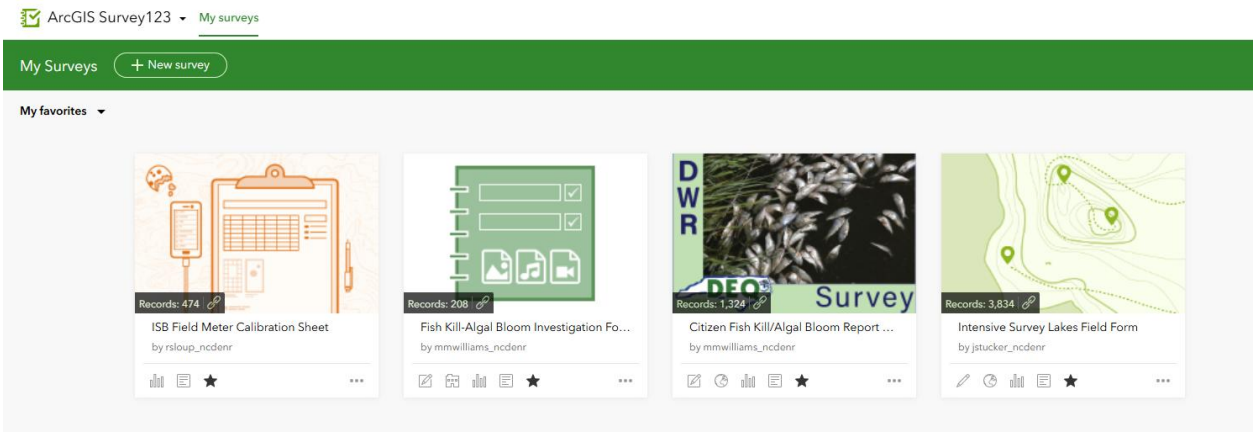


Figure 10. Select forms found in ArcGIS Survey123

"Meter Calibration Sheet-Beta" is the old ArcGIS Survey123 digital form. "ISB Field Meter Calibration Sheet" is the new ArcGIS Survey123 digital form.

<u>Field Meter Calibrations should be entered into ArcGIS Survey123.</u>

All field meter calibrations associated with ISB, AMS and RAMS sampling are expected to be added to the ArcGIS Online (AGOL) table (via the ArcGIS

Survey123 app or browser). The digital calibration sheets are completed in ArcGIS Survey123 and sent in ArcGIS Survey123 to an online database (ArcGIS Online table). Field meter calibrations should be entered in ArcGIS Survey123 "ISB AMS RAMS Field Meter Calibration Sheet" by the end of the following week following each sampling event.

The ArcGIS Survey123 form can be accessed by downloading on the tablet or accessing through another device as an app or ArcGIS Online Account. ArcGIS Online is a browser-based /cloud-based mapping, analysis, collaborative web GIS platform that allows you to use, create, share maps, scenes, apps, layers, analytics, and data.

Options for Entering Field Meter Calibration Data into ArcGIS Survey123.

There are two general ways to enter field meter calibration results into ArcGIS Survey123. The first is using a hard copy calibration sheet to record results from the VuSitu calibration app and then at the end of the day, transfer the hard copy calibration results to the digital ArcGIS Survey123 form. The second is completing the field meter calibration using the VuSitu app and entering calibration results simultaneously into ArcGIS Survey123.

First. Calibration data can be written on the hardcopy calibration sheet (Figure 11) and then manually entered in the ArcGIS Survey123 app at a later time. Field personnel can record all calibration data on the hardcopy calibration sheet for the pre-sampling calibration and post-sampling check sections. The calibration data then can be entered into the ArcGIS Survey123 app later in the day or the next day. Use field sampling dates if the data is entered into ArcGIS Survey123 app on a different day than when sampling occurred.

Second. Field personnel can conduct field meter calibration using the VuSitu app and enter field meter calibration data in ArcGIS Survey123 app simultaneously. There are two options to complete the calibration and ArcGIS Survey123 app simultaneously.

1. First, some field personnel enter field calibration data into ArcGIS Survey123 at the time of calibration by using the same device such as a state issued tablet and switch between the VuSitu field meter calibration app and ArcGIS Survey123 app.
2. Second, field personnel use another device (besides the state issued tablet) to enter the calibration data into the ArcGIS Survey123 app in real time as they calibrate the field meter using the VuSitu app on the state issued tablet.

Field personnel that enter the calibration data into ArcGIS Survey123 app simultaneously while calibrating the field meter using the VuSitu app have noted that they save the ArcGIS Survey123 digital form in their outbox after pre-sampling calibration is completed in the morning. After saving the digital form in the morning, they are then able to resume entering post-sampling calibration data into the ArcGIS Survey123 app in the afternoon after the trip is complete. They are then able to submit the form after completing the post-sampling checks at the end of the day.

Entering Calibration Data from In-Situ VuSitu App to ArcGIS Survey123 Digital Calibration Sheet Already on State Issued Tablet or Other Device.

To start entering the calibration data digitally, tap on the ArcGIS Survey123 app Icon. Select “ISB Field Meter Calibration Sheet”. Select + Collect.

Directions to Set Up ArcGIS Survey123 App If Not Already Installed on State Tablet or Other Device.

You will need access to the ArcGIS account from IT to view calibration data or fill out ArcGIS Survey123 on your computer.

Field personnel or field meter users must contact IT to set up an ArcGIS online account with a Basic User role through NC DEQ. Once you have an account, then login to ArcGIS Survey123 using Enterprise Account to make an ArcGIS URL connection to tablet.

Use these instructions “Login to ESRI’s Survey123 Using Enterprise Account” to make an ArcGIS URL connection to the tablet and signing in.

In-*situ* field parameter measurements

Parameters typically measured

- **Conductivity** (µS/cm @ 25 °C)
- **Dissolved Oxygen** (DO- mg/L and %)
- **pH** (Standard Units)
- **Temperature** (°C)

Additional Calibrations and Use of multiparameter Meters

Battery Voltage

- Use the correct battery source for the particular instrument in use.
- Battery voltage must be in an acceptable range before calibrating and using the meter (see respective manual).

Depth

- Depth is calibrated each day in the field at the first site prior to sampling. Depth should also be calibrated in between lakes, especially if there is a significant change in elevation.
- Record all field depth measurements to the nearest tenth of a meter (if needed).

Calibrated Backup Field Meters

Although meters are maintained, failure can occur at any time. Calibrated backup meters, meter manuals, batteries and calibration buffers/standards are required during sampling. Inability to collect data due to a meter failure is unacceptable. See Appendices 1–5 for detailed guidance on using, maintaining, and storing field meters commonly used by DWR. Field meters should be calibrated monthly regardless of use to maintain status.

ISB/AMS/RAMS Field Meter Calibration Sheet (06/30/2024)

Collector: _____ Agency/Study/Location: _____

Meter Model: _____ Meter Probe Serial #: (not TROLL COM Bluetooth #) _____

	Date mm/dd/yyyy	24 HR Time (hh:mm)	Initials
Pre-Sampling Calibration (on First Page)			
Post-Sampling Check (on Second Page)			

Weird non-calibrated (Initial) readings could be an indication that the meter needs maintenance or calibrations may not be within acceptable ranges. This form stopped tracking initial readings 4/2024.

IF THE ANSWER IS NO TO ANY PRE-SAMPLING CALIBRATION QC CHECK, THE FIELD METER IS NOT PROPERLY CALIBRATED AND SHOULD NOT BE USED IN THE FIELD. REPAIR FIELD METER OR USE ANOTHER CALIBRATED FIELD METER FOR SAMPLING.

Dissolved Oxygen (RDO 100% Saturation Calibration)	Temp °C	Barometric Pressure (mmHg)	Calibrated D.O. % Saturation Meter Reading	Calibrated D.O. Meter Reading (mg/L)	D.O. Table Value (mg/L) (corrected Solubility of O_2 based on pressure and temp.)	Is Calibrated D.O. Meter Reading (mg/L) within ±0.5 mg/L of Table Value?
Pre-Sampling Calibration						Yes or No

Conductivity Standards	Standard Value	Lot Number	Expiration Date
Specific Conductivity HIGH			
Specific Conductivity MID CHECK			

±10% Ranges for S.C.

Standard	Range
100	90-110
500	450-550
1,000	900-1,100
15,000	13,500-16,500
50,000	45,000-55,000

Be sure to change the default High S.C. Standard Value of 1431 µS/cm (In-Situ Brand) on the In-Situ screen to the S.C. High Standard Value reported on the Manufacturer's Certificate of Analysis sheet provided with each new standard. Actual S.C. standard values may be different than what is reported on the box. Verify and change the S.C. standard value on the box to the standard value reported on the Certificate of Analysis. Use Certificate of Analysis standard values when possible.

Specific Conductivity (S.C.) (µS/cm) 1 pt. Calibration	Dry Air Zero S.C. Non-Calibrated Meter Reading	Is Dry Air S.C. Non-Calibrated Meter Reading Within 2 (µS/cm) of 0?	Specific Conductivity HIGH Standard Calibrated Meter Reading	Is S.C. HIGH Standard Calibrated Meter Reading within 10% of Std. Value?	Specific Conductivity Mid Standard Check Meter Reading	Is S.C. MID Standard Check Meter Reading within 10% of Std. Value?
Pre-Sampling Calibration		Yes or No		Yes or No		Yes or No

pH Standards	Standard Value	Lot Number	Expiration Date
7.0 pH Standard (1st Buffer)	7.0		
4.0 or 10 pH Standard (2nd Buffer)	4.0 or 10.0 Circle		

Calibrate with pH 2 point calibration. First with pH standard 7.0 then pH standard 4 or 10. You will not be able to see the 7.0 calibrated reading until you are finished with the 2 point calibration and can get both the calibrated 7.0 and 4.0 or 10 readings from the Calibration Report.

pH (S.U.) 2 Point Calibration	pH 7.0 Calibrated Meter Reading	Is pH 7.0 Calibrated Meter Reading within ±0.2 S.U. of pH Standard 7.0?	Record the mV reading while performing the 7.0 Pre-Sampling Calibration.	Did you change the Electrolyte Filling Solution and/or Reference Junction?	pH 4.0 or 10.0 Calibrated Meter Reading	Is pH 4.0 or 10.0 Calibrated Meter Reading within ±0.2 S.U. of pH Standard Value?	Confirmation 7.0 Standard Check Meter Reading	Is Confirmation pH 7.0 Std. Check Meter Reading within ±0.2 S.U. of pH Standard 7.0? (old sheets is 0.1 S.U.)
Pre-Sampling Calibration		Yes or No		Yes or No		Yes or No		Yes or No

Figure 11. Meter Calibration Sheets

NFQA Participation Requirements to Complete ISB, AMS, and RAMS Program Field Meter Data Collection.

Annually, the ISB, AMS and RAMS field personnel, Regional Office staff, and DWR field personnel who use field meters will participate in United States Geological

Survey (USGS) National Field Quality Assurance (NFQA) program. Current ISB field personnel (and/or other personnel assisting the ALMP, AMS, and RAM Programs) must participate in the NFQA in order to collect field meter data.

The NFQA is a yearly proficiency test for pH and specific conductance that provides accuracy/precision data for field measurements and identifies water quality analysts who need additional training. Field personnel who do not receive satisfactory readings are provided with additional resources/guidance on protocols and equipment repairs to help them complete field meter calibrations and field meter measurements successfully within QA/QC criteria guidelines. All program NFQA participants receive a NFQA report called the “National Field Quality Assurance Project (NFQA) Results”. The QA Coordinator oversees the NFQA for the Water Sciences Section and others who participate in the program.

III. FIELD PARAMETER MEASUREMENTS

For field parameter measurements that are typically conducted using a multiparameter meter (water temperature, dissolved oxygen, pH, and specific conductivity), see Appendices 1–5 for detailed guidance on calibrating, using, maintaining, and storage of meters and probes commonly used by DWR. This SOP and the attached meter guidance sheets are supplementary too and not a replacement for the manufacturer's instructions manual. Consult the manufacturer's instructions for use and maintenance not outlined in this document. In-Situ (The meter ISB currently deploys) manufacturer manuals are found online by selecting the model and then selecting "Manual". Water Quality Multiparameter Sondes | Multiprobes

1. WATER TEMPERATURE

Water temperature measurements are taken by a multiparameter meter (ISB uses In-Situ meters as of the most recent revision), dial Celsius-thermometers, or a thermistor. Below are some general considerations while collecting water temperature data.

- The meter should have a scale marked for every 0.1°C.
- Make readings with the multiparameter meter or thermometer in water for a duration long enough to permit equilibrium.
- The temperature sensor on the multiparameter meters is factory set and does not require recalibration.
- At least once a year, check the meter thermometer against a precision thermometer certified by the National Institute of Standards and Technology (NIST). See Appendix 8 for instruction on this annual check.
- Temperature readings must be recorded as degrees Centigrade (°C) to the nearest tenth of a degree. During field use, the temperature readings should always be read when they are stable and before the other parameters are read to ensure stable readings for all parameters.

2. AIR TEMPERATURE

Refer to the previous procedure, but this time measuring the ambient air temperature above the water surface instead of the water itself. Do not use immersion thermometers to measure the air temperature.

3. DISSOLVED OXYGEN

Dissolved oxygen analysis measures the amount of gaseous oxygen dissolved in an aqueous solution. Dissolved oxygen is a parameter measured by electrometric methods (e.g. In-Situ, Hydrolab, and YSI meters) but can also be measured via chemical methods (Winkler Method). Testing must be done immediately at the sampling location, as a grab sample, which is why electrometric methods are favored.

Electrometric Method Calibration

All field meters should be calibrated before and checked after sampling activities (at least daily). The calibration data should be entered on a meter calibration sheet (Figure 10). Detailed guidance for calibrating dissolved oxygen is provided in Appendices 1–5.

Acceptance Criteria for DO calibration

- Calibrated meters should be compared to a DO table to ensure calibration was done correctly (Appendix 2). USGS has an online tool called DOTABLES that ISB currently uses to calculate solubility of oxygen based on temperature and barometric pressure (DOTABLES | U.S. Geological Survey (usgs.gov)).
- Appendix 2 describes the calculations needed to correct for elevation and a table used at sea-level.
- Dissolved oxygen concentrations need to be calibrated within 0.5mg/L of the elevation corrected table concentration for a given temperature.

Winkler Method - azide modification (Standard Methods, 18th edition)

The azide modification effectively removes interference caused by nitrite, which is the most common interference in biologically treated effluents and incubated

BOD samples. The azide modification is not applicable under the following conditions:

- Samples containing sulfite, thiosulfate, polythionate, appreciable quantities of free chlorine or hypochlorite.
- Samples high in suspended solids.
- Samples containing organic substances which are readily oxidized in a highly alkaline solution, or which are oxidized by free iodine in an acid solution.
- Untreated domestic sewage.
- Biological flocs.
- Where sample color interferes with endpoint detection.

In instances where the azide modification is not applicable, electrometric methods should be employed.

Below are some general considerations while collecting dissolved oxygen data using the Winkler Method:

- Collect surface water samples in narrow-mouth glass-stoppered BOD bottles of 300 ml capacity with tapered and pointed ground glass stoppers and flared mouths. Once analysis is complete and the information is transcribed to the lab sheet, it will be returned to the sampler.
- Avoid entrapping or dissolving atmospheric oxygen. Do not allow the sample to remain in contact with air or be agitated, because either condition may result in a change to its gaseous content.
- Where samples are collected from shallow depths (less than 5 feet) use of an APHA-type sampler is recommended. Use of a Kemmerer type sampler is recommended for samples from depths greater than 5 feet. Bleed sample from bottom of samplers through a tube extending to the bottom of a BOD bottle. Fill bottle to overflowing.
- Record sample temperature to nearest degree Celsius or more precisely.
- Reagents
    - Manganous sulfate solution
    - Alkaline iodide-sodium azide solution. – Sulfuric acid (H_2SO_4) concentration

- Sodium thiosulfate solution 0.025 N
- Starch solution

Analysis Steps:

1. Add 2 mL of manganous sulfate solution to sample container by holding the tip of the pipette below the surface of the liquid.
2. Add 2 mL of alkaline iodide-sodium azide solution by holding the tip of the pipette below the surface of the liquid.
3. Replace BOD bottle stopper, avoid trapping air bubbles, and shake well by inversion.
4. When the precipitate settles, leaving a clear supernatant above the manganese hydroxide floc, shake again.
5. Allow floc to settle again, at least 200 mL of clear supernatant should be above the floc.
6. Remove the stopper and add 2 mL of concentrated sulfuric acid by holding the pipette above the surface of the liquid and allowing the acid to run down the neck of the bottle. Reinsert the stopper and mix by inversion until no floc is visible.
7. Withdraw 203 mL of the solution into an Erlenmeyer flask.
8. Titrate with 0.025 N sodium thiosulfate solution to a pale straw color.
9. Add 1 mL of starch solution and continue titration to the first disappearance of blue color.
10. Record the # of mL of thiosulfate used, where 1 mL thiosulfate = 1 mg/L DO.

4. pH (ELECTROMETRIC METHOD)

Information on pH

Precision and accuracy

±0.2 pH unit represents the limit of accuracy under normal conditions for measurements of water and poorly buffered solutions. For this reason, report pH values to the nearest 0.1 pH unit. Calibrate instrument within 0.2 pH units of the standard pH buffer value.

Calibration Reagents

Calibrate the electrode system against standard buffer solutions of known pH. Always use fresh commercially made buffers to calibrate field meters. Buffer solutions and samples should be stored in polyethylene bottles. Never pour decanted or used buffer solution back into the original bottle.

Procedure

Always follow the manufacturer's instructions for pH meter storage and preparation of electrodes. Recommended short term storage of electrodes varies with type of electrode and manufacturer. See Appendices 1–5 for detailed guidance on calibrating, using, maintaining, and storage of pH meters and probes commonly used by ISB. Never store probes in DI water; tap water and/or pH buffer 4.0 is preferred.

Note: All field meters should be calibrated before and checked after sampling activities daily. The calibration data should be entered on a meter calibration sheet.

Multiparameter In-Situ Meters

A training outline for In-Situ meters used by ISB is listed in Appendix 1.

5. SPECIFIC CONDUCTIVITY/SALINITY

The specific conductance (conductivity) of a solution is a measure of its ability to carry an electrical current. This ability depends on the presence of ions, their total concentration, mobility, valence, relative concentration, and on the temperature of measurement. Specific conductance is the conductance afforded by 1 cc (ml) of a

solution of electrolyte and is reported in micromhos per centimeter (µmhos/cm). Specific conductance measurements are used in water analysis to obtain a rapid estimate of the dissolved solids content of a water sample.

Specific Conductivity Meter Calibration

Detailed meter guidelines for calibrations are listed in Appendices 1-5.

Note: All field meters should be calculated beforehand and checked after sampling activities daily. The calibration data should be entered on a meter calibration sheet.

Additional Calibration Information

- *Acceptance Criteria:* Calibrate instrument within ±10% of the calibration standard's true value.
- Always calibrate with fresh, certified conductivity standards.

6. SECCHI DISK TRANSPARENCY

This is a measurement of water transparency obtained by observing a specially marked, circular, black and white disk which is lowered through the water column until it is not visible. This measure of the point at which the disk is non-visible is considered the Secchi depth.

Secchi disk use (Figure 12)

Conditions for Secchi disk readings

- Measured from the shaded, protected side of boat.
- Minimal waves or ripples are desirable.
- Do not wear sunglasses or a hat while taking the Secchi depth reading.

NOTE: Any departure from these conditions should be specifically stated on the field sheet.

Method

1. Rope should be accurately graduated in meters, with 0.1 m graduations for the first meter, followed by 0.5 m graduations thereafter. Annual verification of correct graduation is necessary as rope may stretch with continued use.
2. Observer's eye should be 1 meter above the water surface.

3. Lower the disk into the water to the depth at which the disk disappears and record that depth at the water surface.
4. Lift the disk and record the depth at the water surface in which the disk first reappears.
5. Record the average reading from previous 2 steps on the field sheet as the Secchi depth reading to the nearest tenth of a meter.

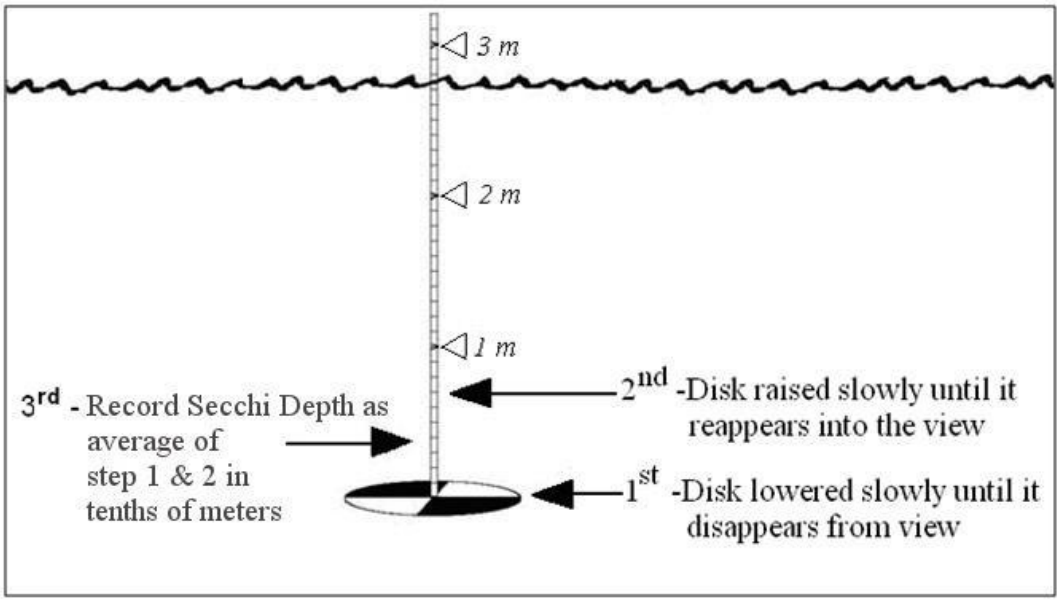


Figure 12. Secchi Disk

7. LIGHT ATTENUATION

Light attenuation is the measurement of the decrease in light intensity through the water column as depth increases due to absorption and scattering effects of water molecules. This is calculated by obtaining a vertical profile of light, using a PAR (photosynthetically active radiation) meter.

PAR Meter Preparation

1. Obtain an independent datalogger such as LI-COR LI-1400.
2. Connect a deck sensor (LI-190R Quantum Sensor) to the BNC connector labelled I1 and an underwater sensor (LI-192 Underwater Quantum Sensor) to the BNC connector labelled I2. Enter the calibration factors (supplied by the manufacturer) for each probe if not already entered.

3. Place the deck sensor in an unshaded location on the boat to record the available ambient light.
4. Turn on the LI-1400 meter and press the View key.
5. Using the left or right keys, navigate to “NEW DATA” and press Enter.
6. Using the left or right keys, navigate until channel I1I is displayed; this shows the instantaneous reading for that channel.
7. Scrolling down will move the cursor to the second row of data
8. Using the left or right keys, navigate until channel I2I is displayed; this shows the instantaneous reading for that channel allowing viewing of both channels of instantaneous data at once.

Methods

1. Lower the underwater sensor on the SUNNY, not shaded, side of the boat to a depth of about 10 cm to represent the surface.
2. Once readings stabilize, record the values from both sensors (μE/m^2/s), along with the water depth of the underwater sensor. Log the values in the datalogger.
3. Lower the underwater sensor to 0.5 m (6"), allow the values to stabilize and record the values from both sensors, along with the water depth of the underwater surface.
4. Repeat the following schedule:
    - Shallow Sites (≤ 2 m) – Every 0.5 m interval.
    - Nominal depths (>2 <10 m) – Every 0.5 m (near surface) and every 1 m interval to near bottom (0.5 m off-bottom)
    - Deep Sites (>10 m) – 0.5 m (near-surface) and every 1 m interval to 10 m, then at 5 m intervals, thereafter, to near-bottom (0.5 m off-bottom)

      NOTE: Follow schedule, unless specified differently for the individual sampling project.
5. If the meter impacts the bottom, allow 2-3 minutes for the disturbed conditions to settle before taking the reading.
6. If the light measurements become negative before reaching the bottom, terminate the profile readings at that depth.

8. REFERENCE POINT-TAPE-DOWN MEASUREMENT

The reference point-tape-down method is a procedure for determining relative vertical distance between fixed bridge points and stage of a water body below the bridge structure.

Procedure for Reference Point-Tape-Down

1. Use a weight-tape gage consisting of a graduated (0.1 ft) steel tape to which is fastened a small cylindrical weight (dimwap) of known length.
2. Locate reference point (RP) as documented on the station location sheet. They are often located on the outer edge of bridge railings.
3. Measure by suspending the weight-tape from the reference point (measuring) to the water surface.
4. The reference point value is indicated by direct reading of the suspended tape where it intercepts the fixed reference point. Read from the top of the bevel if the reference point is beveled.
5. Record measurement and add on the length of the weight.

9. STAGE MEASUREMENTS

These procedures apply to U.S. Geological Survey (USGS) permanent stream gauging stations to support streamflow and water quality assessments.

Obtaining Stage Measurements

Follow Techniques and Methods 3-A7 (Sauer & Turnipseed, 2010).

Prior to Sampling

- Contact the USGS North Carolina Water Science Center at least two weeks prior to sampling to obtain permission from the district chief for access to instrument shelters.

- Complete USGS-provided training on reading stage measuring devices, covering device operation, calibration, and troubleshooting. Document training completion in field logs.

Stage Measuring Devices

Use USGS-approved devices, selecting the primary device based on site conditions. Cross-check with a secondary device at 10% of sites to ensure accuracy within 0.02 ft.

Staff Gage: Read water level to nearest 0.01 ft, noting time and flow conditions.

Wire Weight Gage: Lower cable until weight touches water surface; record level relative to datum.

Electric-Tape Instrument: Measure depth electronically, ensuring sensor is clean.

Automatic Digital Recorder: Download continuous data, verifying timestamp alignment.

Graphic Recorder (Bubble Meter): Check gas-purge system for obstructions; record stage.

QA/QC Procedures

Verify device calibration before sampling (e.g., confirm staff gage alignment with datum).

Flag readings deviating >0.05 ft from expected values for review; check for debris or malfunctions.

Export digital data to a secure server daily and verify against field notes.

Reporting

Submit stage data to NC DEQ's water quality database and USGS NWIS within 30 days, formatted per EPA WQX standards. Share trends via AccessDEQ for stakeholder use.

IV. WATER SAMPLE COLLECTION AND PRESERVATION

1. BOTTLES AND PRESERVATION

Surface water, soil or sludge samples for submittal to the DWR Chemistry Laboratory (the Lab) must be collected using Lab and EPA approved containers and in accordance with approved collection, preservation and holding times. The Lab maintains a website with links to the approved preservation and holding times for all parameters for which the laboratory analyzes.

Field staff are responsible for being familiar with the Lab's procedures and following them accordingly. Preservatives can be added by pipette or pre-measured vials depending on the sensitivity of parameter being measured. If a parameter is not on the Lab's website, speak with the appropriate lab staff to determine how to proceed. Any samples submitted to the Lab must be accompanied by a Lab Sheet (Figure 5). Immediately after sampling, labeling, and chemical preservation, samples are placed in coolers on ice, along with a temperature blank. Once samples arrive at the laboratory, support staff check the temperature blank (included in each cooler) to ensure that they are in appropriate temperature range (4 – 2°C), assign lab tracking numbers, and distribute them to the appropriate analytical units. Any samples not meeting temperature, holding time, or preservation requirements or are otherwise not submitted in accordance with the SOP are subject to rejection as per Section 13: *Corrective Actions* of the Laboratory Section QAM. Laboratory staff will attempt to contact the collector by phone or email before rejecting. If conditionally accepted, the laboratory will document the anomaly with a Sample Condition Upon Receipt (SCUR) and/or Sample Anomaly Report (SAR) form and include copies with the final analytical report. Results from anomalous samples will be reported using the appropriate qualification code(s).

2. COLLECTION METHODS FOR CONVENTIONAL PARAMETERS

Collection for majority of the conventional parameters can be done by the multiple methods introduced in Section I, “Sample Collection Types”. The following section is an overview of the types of parameters collected by DWR along with the required sample size, bottle type, preservative method and holding time.

All samples should be taken within 100m of the fixed site location.

Note: There are some parameters that can ONLY be collected as a surface grab at 0.15 m below the surface and will be stated in the collection method statement. Although, holding times vary from hours to days, **all samples collected should be submitted to the laboratory as soon as possible.**

BOD 5 – Day (Biochemical Oxygen Demand)

This test determines the amount of organic material in wastewater and surface waters by measuring the oxygen consumed by microorganisms in decomposing organic constituents. The test consists of the determination of dissolved oxygen prior to and following 5-day incubation of the sample at 20°C, thus establishing the amount of oxygen used. Coordinate collection dates and sample delivery with the lab to allow for 5 consecutive incubations days.

Collection method:

- Collect surface grab sample in a 1-liter plastic bottle.
- Deliver within ≤48 hours
- Cool to ≤ 6.0° C; keep the sample out of light during transportation
- For WWTP effluents, collect the sample ahead of disinfection when possible.

COD (Chemical Oxygen Demand)

COD measures pollution strength (Sawyer & McCarty, 1967). It is a measure of the amount of oxygen required to oxidize organic and oxidizable inorganic compounds in wastewater and surface waters.

Collection method:

- Collect sample in a 200 ml plastic bottle
- Acidify the sample with H_2SO_4 to pH <2.
- Cool the sample to ≤ 6.0°C
- There is a 28-day holding period.

Coliform

Fecal coliform bacteria are superior to total coliforms as indicators of possible pathogenic contamination of water. The total coliform group includes organisms, principally of the aerogenes group, that are not necessarily of fecal origin. The aerogenes may be a considerable portion of the total coliforms on occasion. They may have no sanitary significance since they can come from soil and vegetation, especially grains. Essentially all fecal coliforms, on the other hand, are of fecal origin and therefore potentially are accompanied by pathogens.

General Collection Methods

- The WSS Chemistry Laboratory provides 250 mL sterile plastic bottles pre-preserved with sodium thiosulfate (Na2S2O3) (0.1mL 10% Na2S2O3/125 mL) to de-chlorinate sample water. Fecal coliform bottles must be ordered from the WSS Chemistry Laboratory.
- Coliform samples are always collected as a surface grab sample. In no case should composite samples be collected for microbiological examination.
- Do not field rinse the bottle with sample. Fill it directly to one half inch from the top to allow mixing of the sample before analysis.
- Use caution to avoid contaminating the sample with fingers, gloves, or other materials.
- Cool to ≤ 6.0°C and return to lab **in less than 6 hours** from time of collection. The DWR Lab will analyze any coliform samples that are received in less than 24 hours; however, the data may not be acceptable for some uses due to extended holding time.
- Fecal coliform bottles received from the laboratory should have lot numbers written on the box or package. The lot number of the fecal coliform bottles should be written on the field sheet in the Lots/Comments section.

Surface Sampling By-Hand

- Grab samples should be collected directly into the sample bottle.
- Remove the bottle top to protect bottle and cap from contamination; avoid touching the inside of the bottle and cap.
- Grasp the bottle securely near the base with one hand and plunge the bottle mouth down into the water to avoid surface scum. Position the bottle towards the current flow and away from the hand of the collector, the shore, the side of the sampling platform, or boat. The sampling depth should be 0.15m below the water surface.
- If the water body is static, create an artificial current by moving the bottle away from the sampler while tipping the bottle slightly to allow water to enter.
- Tip the bottle slightly upwards to allow air to exit and the bottle. Fill the bottle directly one-half inch from the top.
- After removing the bottle from the stream, close and label the bottle.

Surface Sampling by Weighted/Cage Bottle Frame (Figure 4)

- Remove the cover and lower the device to the water.
- It is preferable to use nylon rope which does not absorb water and will not rot.
- Swing the sampling device downstream and then allow it to drop into the water while pulling on the rope so as to direct the bottle upstream.
- Pull the sample device rapidly upstream and out of the water, simulating the scooping motion of grab sampling.
- Take care not to dislodge dirt or other material from the sampling platform.

Residue (Solids)

Residue refers to solid matter suspended or dissolved in water or wastewater.

Residue Types

- Total Residue - is the term applied to the material left after evaporation of a water sample, and its subsequent drying in an oven at a defined

temperature. Total residue includes nonfilterable residue and filterable residue. Also known as Total Solids.

- Nonfilterable Residue (Suspended) - the portion of total residue retained by a filter. The concentration of other water quality parameters is related to suspended solids since the solid structure may contain biochemical and chemical oxygen demand materials, trace metals, nutrients, pesticides, and toxic or hazardous materials absorbed on the surface. Also, known as Total Suspended Solids.
- Filterable Residue (Dissolved) - the portion of total residue that passes through the filter. Dissolved solids consist mainly of inorganic salts, small amounts of organic matter, and dissolved gases. Also called Total Dissolved Solids.
- Volatile and Fixed Residue - the residue remaining after ignition for 1 hour at 550°C represents the ash or fixed solids, and the weight loss incurred is a reasonably accurate measure of organic matter or volatile solids.

Collection method:

- Use a 500 ml plastic bottle to collect **each** type of residue sample. Note: non-filterable residue (total suspended solids) requires the use of a ½ gallon plastic bottle.
- Cool to ≤ 6.0°C.
- The sample has a holding time of 7 days.

Alkalinity/Acidity

Alkalinity is a measure of the buffering capacity of water - the power of the water to neutralize hydrogen ions - and it is expressed in terms of an equivalent amount of calcium carbonate. Alkalinity is caused by the presence of carbonates, bicarbonates, and hydroxides. Acidity is the power of the water to neutralize hydroxy ions - and it is expressed in terms of an equivalent amount of calcium carbonate. Acidity is a result of the presence of free carbon dioxide, strong mineral acids, weakly dissociated acids, salts of strong acids, and weak bases.

Collection method:

- Collect sample with a 500 ml (for each parameter) plastic bottle,
- cool to ≤ 6.0° C.

- Holding time is 14 days.

TOC (Total Organic Carbon)

TOC measures the organic carbon present in water. When an empirical relationship can be established between TOC, BOD, and COD, the TOC provides a quick and convenient way of estimating the other parameters that express the degree of organic contamination.

Collection method:

- Collect sample with a 200 ml plastic bottle,
- Add H_3PO_4 to pH <2 and cool to ≤ 6.0°C.
- Holding time is 28 days.

Turbidity (Clarity of Water)

Turbidity is measured in Nephelometric Turbidity Units (NTU). It is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in straight lines. Turbidity in water is a result of suspended matter such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, plankton and other microscopic organisms.

Collection method:

- ALMP samples - collect turbidity as a composite photic sample by lowering an integrated sampler to twice the Secchi disk reading and slowly raising the sample back up to the surface.
- Collect the sample in a 500 ml plastic bottle
- Cool to ≤ 6.0°C. The sample should be protected from light (store in closed cooler).
- The sample must be received by the lab in **less than 48 hours**.

Chloride

Chlorides are found in most natural waters. They may be of natural mineral origin or artificially introduced. Chloride concentrations are higher in wastewater than in raw water because sodium chloride (NaCl) is a common article of diet and

passes unchanged through the digestive system (American Public Health Association, 1992). Industrial processes also increase chlorides in wastewater effluents.

Collection method:

- Collect the sample in a 500 ml plastic bottle, typically collected directly from the water body as a surface grab (0.15 M deep).
- Cool to ≤ 6.0°C.
- Holding time is 28 days.

Chlorophyll *a* and Algal Biomass

Chlorophyll a - Chlorophyll *a* is the photosynthetic green pigment contained in plants. The measurement of this pigment provides an estimate of algal biomass.

Collection method:

- Samples for phytoplankton should be taken with an integrated depth sampling device (Labline).
- This device should be lowered to twice the Secchi depth (approximately 1% light penetration) and slowly raised to the surface.
- Pour sample into a 500 ml wide-mouth opaque plastic bottle to collect the sample.
- Cool to ≤ 6.0°C.
- Filtration must occur within 48 hours of collection (see *Surface Water Samples: Containers, Preservation and Hold Times* document on the Water Sciences Section Lab's website).

Algae - Algae are used as biological indicators of water quality. By determining the types and quantity of algae present in a water body and utilizing physical and chemical data collected at the same time, inferences can be made concerning the trophic state of a water body. Algae are sampled from the water column (phytoplankton), attached to rocks and debris (periphyton), and from floating mats (filamentous/nuisance growths). The primary type sampled by DWR is phytoplankton, although all forms of algae can be sent to the Ecosystems Branch (EB) Laboratory of the Environmental Sciences Section for analyses.

Collection method:

- Samples for phytoplankton should be taken with an integrated depth sampling device (Labline).
- This device should be lowered to twice the Secchi depth (approximately 1% light penetration) and slowly raised to the surface.
- Pour sample into a 500 mL plastic disposable bottle and preserve with approximately 2.5 mL of modified Lugol's solution or until a dark straw color (tea) is reached.
- If a Labline is unavailable, a surface grab sample can be taken.
- Scoop samples are taken only when no quantitative methods are possible or as an additional sample for ease in identification.
- Live samples are taken as above (Labline preferred) but are not preserved. Cool to ≤ 6.0 C. Send to the Lab or EB lab in **less than 24 hours**.

Chlorophyll a and Algal Sample Submittal Procedure

- Samples should then be sent to the Central Laboratory along with nutrient and chemical samples.
- Bloom samples should include one preserved and one unpreserved (live) phytoplankton sample along with chlorophyll *a* and nutrient samples and a completed bloom form. Bloom forms and modified Lugol's solution (for preservation) are obtained from the Ecosystems Branch in Raleigh.
- After samples are logged in at the Central Lab and with the Ecosystems Group, they are analyzed per the Ecosystems Branch's SOP manual.

Parameters collected in conjunction with phytoplankton samples.

- Physical Parameters- Are measured at the surface and at every meter or half meter from the surface to bottom according to depth. Parameters include:
  - Temperature
  - Dissolved Oxygen
  - pH
  - Secchi Depth
  - Conductivity
  - Salinity should be taken where appropriate.

- Chemical samples- May include ammonia/ammonium, nitrate/nitrite, total Kjeldahl nitrogen, orthophosphate, total phosphorous, and chlorophyll *a* are required to accompany phytoplankton samples.

  NOTE: Check with the lab prior to sampling for orthophosphate to ensure analysis capabilities.

Record Management

- Public Version (Citizen Fish Kill/Algal Bloom Report Form) - The public version is the one listed on the DWR website at this address: Citizen Fish Kill/Algal Bloom Report Form (arcgis.com). When the public version is submitted, an email goes out to all staff in the appropriate regional office. This reporting form will populate to the dashboard.
- Staff (DEQ employee version) (Internal Reporting Fish Kill and Algal Bloom) - The Public reporting form has the exact same information, but the Staff form is intended for State of NC staff to use for a citizen report since it won't alert other staff at the appropriate regional office. The address for this form is: DWR Citizen Report (arcgis.com). This reporting form will populate to the Fish Kill and Algal Bloom Report Dashboard.
- Fish Kill – Algal Bloom Investigation Form. There is a digital version of the fish kill and algal bloom field investigation form available (we prefer if folks use this digital version rather than the paper/PDF version, but we'll accept both!). All three of these forms (public and staff reporting forms and field investigation form) are also available to download on the Survey123 app, which may be easier for staff in the field to use.

There are previous versions of some of these forms on the app, so staff should search for and use the forms with the exact titles below:

  - Citizen Fish Kill/Algal Bloom Report Form (Public)
  - Internal Reporting Fish Kill and Algal Bloom (Staff)
  - Fish Kill-Algal Bloom Investigation Form (Field Investigation Form)

Color

Color in water may result from the presence of natural metallic ions (iron and manganese), humus and peat materials, plankton, weeds, and industrial wastes. True color is the color of water from which turbidity has been removed by

filtration or centrifugation. The term apparent color includes not only color due to substances in solution, but also due to suspended matter. Apparent color is determined on the original sample without filtration or centrifugation. In stream samples, unaffected by industrial wastes, usually only true color is analyzed. In some highly colored industrial wastewater, color is contributed principally by colloidal or suspended material. Therefore, apparent color may be a more appropriate measure for samples related to industrial wastewater. The color value of water is extremely pH dependent and increases as the pH of the water is raised. Therefore, always measure in-*situ* pH and specify the pH at which the color is determined.

Accepted Methods to Determine Color

There are three accepted methods to determine color (USEPA, 1994): Platinum-cobalt, spectrophotometric, and ADMI. Each of these methods yields different information. Their proper uses and interpretations must be reviewed to determine the appropriate test based on the purpose of the sampling.

Collection method:

- Use a 500 ml plastic bottle to collect a surface grab sample.
- Cool sample to ≤ 6.0°C.
- Sample must be submitted to the lab in **less than 48 hours**.

Chromium, Hexavalent [Cr^{+6}]

The principal chromium emissions into surface waters are from metal-finishing processes such as electroplating, pickling, and bright dipping. Uncontrolled emissions have great potential for contaminating the fresh waters with the relatively toxic form, Cr (+6). Other smaller discharges of Cr^{+6} are from the additive in circulating cooling waters, laundry chemicals, and animal glue manufacture.

Collection method:

- Collect sample in a 400 mL plastic disposable bottle
- Field filter
- Ammonium sulfate to pH 9.3-9.7 within 15 minutes
- Cool sample to ≤ 6.0°C

- Hold time is 28 days.

NOTE : Lab should be notified that this sample will be submitted for analysis prior to sample collection.

Cyanide (CN^-)

Cyanides occur in the effluents from gas works and coke ovens, from the scrubbing of gases at steel plants, from metal cleaning and electroplating processes, and from chemical industries. Most of the cyanide in water is in the form of HCN (hydrogen cyanide). Toxicities may vary markedly with pH and a given concentration that is innocuous at pH 8 may become detrimental if the pH is lowered to 6 or less. In natural streams, cyanides deteriorate or are decomposed by bacterial action, so that excessive concentrations may be expected to diminish with time.

Collection method:

- Use a 250 mL reusable, dark bottle supplied by the WSS Chemistry Laboratory to collect a surface grab sample directly from the water body. Cyanide samples require a tag (no adhesive labels). Do not field rinse prior to sampling.
- Add 6N sodium hydroxide (NaOH) to pH>10 but <11. Recommended 0.75 to 1.0 droppers full for the new 250ml bottles. The final pH after preserving should be verified with pH test strips. Pour a sample aliquot onto pH strips.
- If chlorine is present, see Appendix 5.
- Cool sample to ≤ 6.0°C.
- Sample has a holding time of 14 days.

Fluoride (F^-)

Fluoride at 0.8 to 1.5 mg/L in drinking water aids in the reduction of dental decay, especially among children. Fluorides in high concentrations are not a common constituent of natural surface waters, but they may occur in detrimental concentrations in ground water. Fluorides are used as insecticides, for disinfecting brewery apparatus, as a flux in the manufacture of steel, for preserving wood and mucilage, for the manufacture of glass and enamels, in

chemical industries, and water treatment. While not normally found in industrial waste, they may be present in traces, or in higher concentrations resulting from spillage. Sampling should occur as far from the engine as possible for inorganic anions.

Collection method:

- Use a 500 ml plastic bottle to collect a surface grab sample directly from the water body.
- Sample must be cooled to ≤ 6.0°C.
- Holding time is 28 days.

Formaldehyde (HCHO)

Formaldehyde is a colorless gas with a pungent odor. It is usually stored and transported as an aqueous solution containing 37-50% formaldehyde by weight and 1-15% methanol. Formaldehyde is used in the production of urea-formaldehyde and phenol formaldehyde resins. These resins are used in the production of plywood, particleboard, foam insulation, and a wide variety of molded or extruded plastic items. Formaldehyde is intensely irritating to mucous membranes, and the National Institute for Occupational Safety and Health recommends that formaldehyde be handled as a potential occupational carcinogen. Formaldehyde is used for preserving biological specimens.

Collection method:

- Ask lab for guidance if needed for special study.

HEM: Grease and Oil

For the grease and oil analysis, groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in trichlorotrifluoroethane (Freon). Grease and oils, either vegetable oil, animal fats, or mineral hydrocarbons, when introduced to surface waters, are found floating on the surface, emulsified or solubilized in the water column, or settled on the bottom as a sludge. Potential contributors to oil pollution are all agencies engaged in production, transportation, handling, and use of oil. Also, ships, railroads, civic dumps, salvage dumps, machining operations, and the most notable - garages and filling stations. Grease from animal and vegetable oils enters waterways from food processors and restaurants. Surface waters are

at all times to be kept virtually free from oil or grease, not only for esthetic reasons and taste and odor problems for domestic water supply, but evidence has demonstrated both acute lethal toxicity and long term sublethal toxicity of oils to aquatic organisms.

Collection method:

- Collect 2 liters (two 1-liter glass wide mouth mason jars, Teflon-lined caps) of sample. Do not field rinse prior to sample collection.
- A surface grab sample at 0.15 m deep is the only collection method.
- Acidify the sample with HCL or H_2SO_4 to pH <2.
- Sample must be cooled to ≤ 6.0°C Holding time for this sample is 28 days.

Total Hardness

Hard waters are generally considered to be those waters that require considerable amounts of soap to produce a foam or lather and that also produce scale in hot water pipes, heaters, boilers, and other units in which the temperature of water is increased materially. In general, surface waters are softer than ground waters. The hardness of water reflects the nature of the geological formations with which it has been in contact. Natural sources of hardness principally are limestone that are dissolved by percolating rainwater made acid by dissolved carbon dioxide. Industrial and industrially related sources include the inorganic chemical industry and discharges from operating and abandoned mines.

Classification of water by hardness content (Conc., mg/l $CaCO_3$) (USEPA, 1976)

Hardness Classification	**$CaCO_3$Concentration (mg/l)**
Soft	0 - 75
Moderately Hard	75 - 150
Hard	150 - 300
Very Hard	300+

The constituents that impart hardness to water are polyvalent cations, chiefly calcium (Ca^{++}) and magnesium (Mg++). These form insoluble complexes with a variety of anions ($HCO3^-$, $SO4^-$, Cl^-, NO_3^-, SiO_3^-). By convention, hardness is reported on the basis of equivalence as mg/l calcium carbonate ($CaCO_3$).

The WSS Chemistry Laboratory analyzes hardness by titration if total hardness as $CaCO_3$ by titration (mg/L) is checked on the field sheet.

Collection method:

- Sample must be collected as a surface grab (0.15 m from the surface) in a 500 ml plastic bottle.
- Acidify the sample with HNO3 to pH <2 and cool to ≤ 6.0°C
- Holding time is 6 months.

Specific conductance (Specific Electrical Conductance)

The specific conductance (conductivity) of a solution is a measure of its ability to carry an electrical current. This ability depends on the presence of ions, their total concentration, mobility, valence, relative concentration, and on the temperature of measurement. Specific conductance is the conductance afforded by 1 cc (ml) of a solution of electrolyte and is reported in micromhos per centimeter (µmhos/cm). Specific conductance measurements are used in water analysis to obtain a rapid estimate of the dissolved solids content of a water sample (see section III subsection 5.0). This measurement is normally made using a field meter; however, the following procedure can be used if necessary.

Collection method:

- Use a 200 ml plastic bottle collected as a surface grab (0.15 m from the surface).
- Sample should be cooled to ≤ 6.0°C.
- Holding time is 28 days.

MBAS - Methylene-Blue-Active Substances

This test determines surfactants with no specificity, so the materials determined are designated as MBAS. This method depends on the formation of a blue salt or ion pair when methylene blue, a cationic dye, reacts with anionic surfactants. Surfactants are organic materials, which have the property of being surface

active in aqueous solution. All surfactants have rather large polar molecules. One end of the molecule is particularly soluble in water, and the other is readily soluble in oils. The surfactants include soaps, detergents, emulsifiers, wetting agents, and penetrants. Of these substances, the synthetic detergents are most important and are used in the greatest amounts. Presently, about 80 percent of all synthetic detergents are of the anionic type, and the MBAS method determines the presence of these surfactants. The most widely used anionic surfactant is linear alkylbenzene sulfonate (LAS). The detergent manufacturing industry changed to the production of LAS because it is more readily biodegradable than the older ABS (alkyl benzene sulfonates).

Collection method:

- **The lab must be notified that this sample will be collected and submitted for analysis.**
- Use a 500 mLl plastic bottle to collect a surface grab sample (0.15 m from the surface) and cool to ≤ 6.0°C.
- Sample must be returned to the lab in **less than 48 hours**.

Phenols (C_6H_5OH)

An aromatic compound known as carbolic acid. In concentrated solutions, phenol is quite toxic to bacteria and is widely used as a germicide. Phenol is obtained from coal tar and manufactured synthetically. It is used extensively in the synthesis of organic products, particularly phenolic-type resins. Phenolic waste arises from the distillation of wood, from gas works, coke ovens, oil refineries, chemical plants, and from human and animal refuse.

Collection method:

- Use two- 1 liter glass (phenol bottles) bottles to collect a surface grab (0.15 m from the surface)
- Check for chlorine, if it is detected add Ferrous Ammonium Sulfate (Appendix 5).
- Acidified the sample with H2SO4 to pH <2.
- Cool the sample to ≤ 6.0°C
- There is a 28-day holding period.

Sulfate (SO_4)

The sulfate ion is one of the major anions occurring in natural waters. Sulfates occur as the final oxidized state of sulfides, sulfites, and thiosulfates. Sulfates may also occur as the oxidized stage of organic matter in the sulfur cycle. Sulfates may be discharged into numerous industrial wastes, tanneries, sulfate-pulp mills, textile mills, and other plants that use sulfates or sulfuric acid. Sulfate is important to public water supplies because of its cathartic effect upon humans when it is present in excessive amounts (upper limit-250 mg/l U.S.P.H.S.). Sulfates are of considerable concern to wastewater treatment plants because of odor and sewer corrosion problems resulting from the reduction of sulfates to hydrogen sulfide (H_2S or hydro sulfuric acid in an aqueous solution).

Collection method:

- Sample should only be collected as a surface grab sample.
- Collect a surface grab (0.15 m from the surface) in a 500 mL plastic bottle
- Cool the sample to ≤ 6.0°C.
- Sample has a hold time of 28 days.

Sulfide (S^-)

Sulfides are constituents of many industrial wastes, tanneries, paper mills, chemical plants, and gas works. Sulfides are also generated in sewage and some natural waters by the anaerobic decomposition of organic matter. Sulfides react with hydrogen ions to form HS^- or H_2S. The toxicity of sulfides derives primarily from H_2S rather than from the hydrosulfide (HS^-) or sulfide (S^{-2}) ions. H_2S is very toxic and has claimed the lives of numerous workers in sewers but owing to the unpleasant taste and odor (rotten eggs), most people or animals avoid consuming a harmful dose.

Collection method:

- Samples should only be collected as a surface grab.
- Collect three 40 mL glass VOA vials with Teflon-lined septum (pre-preserved with 0.1 mL of zinc acetate) directly as a surface grab (0.15 m from the surface).
- Allow the sample to overflow the vial.
- Add 3-4 drops of 6N NaOH to pH >9.

- Cap the vial when sample is overflowing, leaving no air space.
- Cool the sample to ≤ 6.0 °C.
- Holding time is 7 days.

Phosphorous and Nitrogen (Nutrients)

Phosphorus occurs in natural waters and in wastewater almost solely as phosphates. Evidence indicates that high phosphorus concentrations are associated with accelerated eutrophication of waters when other growth promoting factors are present, and aquatic plant problems develop in reservoirs, and other standing waters at phosphorus values lower than those critical in flowing streams.

Nitrogen is one of the fertilizing elements essential to the growth of algae. Such growth is often stimulated to an undesirable extent in bodies of water that receive excess inputs of nitrogen from either point or nonpoint sources.

Nutrient Types:

- NH_3 (Ammonia) - In surface or ground waters, ammonia results from the decomposition of nitrogenous organic matter. It may also result from the discharge of industrial waste from chemical or gas plants, from ice plants, or from scouring and cleaning operations where ammonia water is used. The conversion of ammonia to nitrites and nitrates by bacteria requires oxygen, and so the discharge of ammonia nitrogen and its subsequent oxidation can seriously reduce the dissolved oxygen levels in rivers and estuaries.
- TKN (Total Kjeldahl Nitrogen) - Analytically, organic nitrogen and ammonia can be determined together and are referred to as Kjeldahl nitrogen, a term that reflects the technique used in their determination.
- $NO_2 + NO_3$ (Nitrites + Nitrates) - Nitrites are quickly oxidized to nitrates. Nitrates are the end product of the aerobic stabilization of organic nitrogen. Nitrates also occur in percolating ground waters as a result of excessive application of fertilizer or leaching from septic tanks. Nitrates are seldom abundant in natural surface waters because of uptake by plants.
- Total P (Phosphorus) - Phosphorus occurs in natural waters and in wastewater almost solely as phosphates. High phosphorus concentrations

are associated with accelerated eutrophication of waters when other growth promoting factors are present.

- PO_4 (Orthophosphate) - Orthophosphate is used as fertilizer and is applied to agricultural and residential cultivated land where it is carried into surface waters with storm runoff.

Collection Methods for Unfiltered Nutrients (NH_3, TKN, NO_2+NO_3, and Total P)

- Use a 500 ml plastic disposable bottle for sample collected.
- Acidify the sample with H_2SO_4 to pH<2. **Note**: Addition of an excessive amount of acid will interfere with the sample analysis
- Cool the sample to ≤ 6.0 °C.
- Holding time is 28 days.

Collection Method for Orthophosphate (PO4-P) and Dissolved P (Filtered Nutrients)

This water sample must be filtered in the field. A detailed Standard Operating Procedure for field filtering using a vacuum pump can be found in Appendix 3. Be careful- **do not** allow filter residue to touch filter apparatus or forceps.

- Use a 200 ml plastic bottle for each sample.
- Dissolved P sample is acidized to pH <2 by adding H_2SO_4.
- Dissolved P and PO_4-P samples must be cooled to ≤ 6.0 °C.
- Holding time for PO_4-P is less than 48 hours.
- The holding time for Dissolved P is 28 days.

NOTE: For Turbid Samples - change filters during process.

Metals

The following metal parameters are collected in one bottle: Cd, Cr, Cu, Ni, Pb, Zn, Ag, Al, Be, Ca, Co, Fe, Li, Mg, Mn, Na, K, Ba, As, Se, Hg.

Whenever metal samples are collected the collection of field pH is essential. Metals are always collected as a surface grab

Collection method – all metals with the exception of Hg:

- Total metals: Collect one 500 ml of sample in a plastic disposable bottle directly from the water body as a surface grab (0.15 from the surface). Add HNO_3 to pH <2. Cool the sample to ≤ 6.0 °C.
- Dissolved metals: Using the provided 1 L plastic bottle filled with DI water, condition the filter. Once the filter is conditioned this bottle can then be used to collect a surface grab sample. This sample is then filtered utilizing a peristaltic pump and two separate dissolved metals duplicate samples are filled. Add HNO3 to pH<2. Cool the samples to ≤ 6.0 °C.
- Metals have a 6-month holding time.
- Please see detailed instructions in Appendix 6.

Collection method –Hg alone:

- Follow Mercury – EPA1631 Trace-Level Mercury Sampling Standard Operating Procedure detailed in Appendix 7.

Cadmium (Cd)

In the elemental form, cadmium is insoluble in water. It occurs in nature largely as the sulfide salt, greenockite or cadmium blend, often as an impurity in zinc-lead ores. Cadmium is used in metallurgy to alloy with copper, lead, silver, aluminum, and nickel. It is also used in electroplating, ceramics, pigmentation, photography, and nuclear reactors. Cadmium salts are sometimes employed as insecticides and anthelminthics. Cadmium salts may be found in wastes from electroplating plants, pigment works, textile printing, lead mines, and chemical industries. Cadmium has been shown to be toxic to man when ingested or inhaled.

Chromium (Total Cr)

The principal chromium emissions into surface waters are from metal finishing processes such as electroplating, pickling, and bright dipping. Other smaller discharges of chromium are from the additive in circulating cooling waters, laundry chemicals and animal glue manufacture, leather tanning, and textile dyeing. Chromium is one of the least toxics of the trace elements. Chromium is not acutely toxic to humans.

Copper (Cu)

Copper salts in natural waters are generally the result of pollution attributable to the corrosive action of water on copper and brass tubing, to industrial effluents, and algaecide. Copper salts are used in textile processes, pigmentation, tanning, photography, engraving, electroplating, insecticides, and fungicides. Because copper in concentrations high enough to be dangerous to human beings renders water disagreeable to taste, it is believed that copper is probably not a hazard in domestic water supplies. However, copper in water may be disadvantageous or detrimental for certain industrial uses. In trace amounts, copper may be beneficial or even essential for the growth of living organisms. In excessive quantities it has been found toxic to a wide variety of aquatic forms, from bacteria to fish.

Nickel (Ni)

Nickel toxicity to man is believed to be very low. Systemic poisoning of human beings by nickel or nickel salts is almost unknown. Nickel does not merit serious consideration as a water pollutant, but nickel ions may be detrimental to beneficial uses. Nickel is toxic to some plants. Nickel is used in metal plating, batteries, as a catalyst in the preparation of edible oils, and in solar energy equipment.

Lead (Pb)

Lead is a cumulative poison. The poisoning usually results from the cumulative toxic effects of lead after continuous, long-term consumption rather than from occasional small doses. Lead exists in nature mainly as the sulfide (galena). Some natural waters contain lead in solution where mountain limestone and galena are found. Lead may also be introduced into water as a constituent of various industrial and mining effluents or as a result of the action of the water on lead in pipes. Atmospheric fallout and rainout of particulate lead are considered the most significant sources of lead input into natural surface waters, especially in urban areas. Storm runoff originating in urban areas will tend to be high in lead concentration. The low solubility of lead in the aqueous phase of natural systems and the formation of stable complexes with organic matter are manifested in the low uptake by some plants and animals. There are extremely low concentrations of lead in natural bodies of water in proportion to the concentration in the beds of lakes and streams. The net effect of these sluggish dynamics is a high degree of accumulation with prolonged exposure.

Zinc (Zn)

Zinc is used extensively for galvanizing, in alloys, for electrical purposes, in printing plates, for dye manufacture, and dyeing processes. Zinc salts are used in paint pigments, cosmetics, pharmaceutics, dyes, and insecticides. Zinc is found in high concentrations in natural waters in zinc mining areas and in effluents from metal plating works. In most surface and ground waters it is present only in trace amounts. There is some evidence that zinc ions are absorbed strongly and permanently on silt with a resultant inactivation of the zinc. Zinc has no known adverse physiological effects upon man except at very high concentrations. For esthetic considerations, high concentrations of zinc in domestic water are undesirable. At 30 mg/l, zinc gives water a milky appearance and causes a greasy film on boiling. It is toward fish and aquatic organisms that zinc exhibits its greatest toxicity at much lower concentrations.

Silver (Ag)

Silver metal is used in jewelry and silverware, in alloys, for electroplating, and in the processing of food and beverages. Silver nitrate is used in photography, ink manufacture, electroplating, coloring porcelain, and as an antiseptic. Traces of silver can be expected to reach natural waters from such sources. Silver is bactericidal and toxic at low concentrations.

Aluminum (Al)

Aluminum is the third most abundant element of the earth's crust. Aluminum occurs in many rocks, ores, and clays, but never as a pure metal in nature. The metal itself is insoluble, but many of its salts are readily soluble. Aluminum is not likely to occur for long in surface waters because it precipitates and settles or is absorbed as aluminum hydroxide or aluminum carbonate. In streams the presence of aluminum ions may result from industrial waste or more likely from wash water containing aluminum from water treatment plants.

Beryllium (Be)

A relatively rare element found chiefly in the mineral beryl, this substance is not likely to occur in natural waters. Although the chloride and nitrate forms are very soluble in water and the sulfate form moderately so, the carbonate and hydroxide forms are almost insoluble in cold water. Beryllium is used primarily in metallurgy to produce special alloys, in the manufacture of X-ray diffraction tubes and electrodes for neon signs, and in nuclear reactors. Beryllium is not harmful when taken internally through the digestive tract but

has been incriminated in pulmonary ailments of workers exposed to beryllium dust.

Calcium (Ca)

Calcium is the most abundant dissolved cationic constituent of natural fresh waters. This element is widely distributed in the minerals of rocks and soils. Calcium carbonate is frequently found as a cementing agent between mineral particles of sandstone and other detrital rocks. Calcium is one of the constituents of hard water and is a scale former in hot water systems. Prevention of corrosion of cast iron water distribution systems may be obtained through controlled precipitation of calcium carbonate. Lime ($CaOH_2$), and dolomite [$CaMg(CO_3)_2$] are frequently employed as neutralizing agents in water and wastewater treatment.

Cobalt (Co)

Cobalt and its salts are used for making alloys, in nuclear technology, as pigment in the china and glass industry, and as binders in the tungsten-carbide tool industry. Cobalt has a relatively low toxicity to man, and traces of cobalt are essential to nutrition.

Iron (Fe)

Iron interferes with laundering operations, imparts objectionable stains to porcelain fixtures, and causes difficulties in distribution systems by supporting growths of iron bacteria. Iron also imparts a taste to water, which is detectable at very low concentrations. In addition to corrosion products, natural waters may be polluted by iron-bearing ground water.

Lithium (Li)

An alkali metal, it is not widely distributed in nature, being found in a few minerals and in certain spring waters. Lithium is used in metallurgy, medicinal waters, some types of glass, and as lithium hydroxide in storage batteries. Lithium is toxic at high concentrations.

Magnesium (Mg)

Magnesium ions are of particular importance in that they occur in significant concentration in natural waters, and along with calcium, form the bulk of the hardness reaction. Magnesium is considered relatively non-toxic to man and not a public health hazard because, before toxic concentrations are reached in water, the taste becomes quite unpleasant. At high concentrations,

magnesium salts have a laxative effect, particularly upon new users, although the human body can develop a tolerance to magnesium over a period of time.

Manganese (Mn)

Manganese is essential for the nutrition of both plants and animals. Manganese is undesirable in domestic water supplies because it causes an unpleasant taste, deposits on food during cooking, stains, and discolors laundry and plumbing fixtures, and fosters the growth of some microorganisms in reservoirs, filters, and distribution systems. Manganese frequently appears in surface waters as the result of decaying vegetation, in waters with acidic pH values, and acidic waters from coal mine drainage. In ground water subject to reducing conditions, manganese can leach from the soil and occur in high concentrations.

Sodium (Na)

Sodium salts are extremely soluble in water; any sodium that is leached from soil or discharged to streams by industrial wastes will remain in solution. Sodium is the cation of many salts used in industry and as such is one of the most common ions in process waste. Sodium in drinking water may be harmful to people suffering from cardiac, renal, and circulatory diseases.

Potassium (K)

One of the more common elements, potassium is one of the most active metals, and for that reason it is not found free in nature but only in the ionized or molecular form. Potassium is used for fertilizers and some varieties of glass. It is an essential nutritional element, but in excessive quantities it acts as a cathartic.

Barium (Ba)

Barium ions are not normally present in natural surface or ground waters in measurable concentrations although they have been detected in a few springs and in effluents from areas where barytes, $BaSO_4$, or witherite, $BaCO_3$, are mined. Barium and its salts are used in the metallurgical industry for special alloys, in the paint industry, in cements designed to withstand salt water, and in the ceramic and glass industries. Because of possible toxic effects on the heart, blood vessels and nerves, a surface water supply standard of 1.0 mg/l was established.

Arsenic (As)

Arsenic may occur in water as a result of mineral dissolution, industrial discharges, or the application of insecticides. Arsenic is toxic to humans and accumulates in the body.

Selenium (Se)

Elemental selenium is practically nontoxic, but hydrogen selenide and other selenium compounds are extremely toxic and resemble arsenic in their physiological reactions. Selenium poisoning occurs mostly among livestock, and the toxic effects appear to be associated with the consumption of high concentrations of selenium in food, such as locoweed or grains grown in soils with high concentrations of selenium, rather than from water consumption. Selenium occurs in sulfur deposits, sulfides of metals, volcanic emissions, sedimentary rocks, rich organic soils, and coal. Selenium is used in the electronics industry, xerographic copying machines, photoelectric cells, glass and ceramics, pigment manufacture to color plastics, paints, enamels, inks, and rubber. It is also used as a component of plating solutions. It can also be found in discharges from coal-fired power plants.

Mercury (Hg)

Mercury and mercuric salts are considered to be highly toxic to humans and aquatic life. Elemental mercury is inert chemically and insoluble in water and is not likely to occur as a water pollutant. Mercuric salts occur in nature chiefly as the sulfide HgS, known as cinnabar, but numerous synthetic organic and inorganic salts of mercury are used commercially and industrially. They are used in medicinal products, disinfectants, detonators, pigments, and photoengraving. Many of the mercuric and mercurous salts are highly soluble in water.

3. PESTICIDES AND ORGANICS

This section outlines procedures for collecting and analyzing pesticides and organic pollutants, including pesticides, semi-volatile organic compounds (SVOCs), acid herbicides, and volatile organic compounds (VOCs). Best practice is to collect these analytes first at your sample station to minimize contamination.

Pesticides

Pesticides are any substance or mixture of substances intended for preventing, destroying, repelling, or mitigating insects, rodents, fungi, viruses, or weeds, and other forms of plant or animal life considered to be pests.

Pesticides are categorized into three groups:

- Inorganic - arsenicals, mercurials, borates, and fluorides
- Synthetic organic - chlorinated hydrocarbons, organic phosphates, and thiocarbamates
- Natural organic - rotenone, pyrethrum, and nicotine

Pesticides may also be classified by their biological usefulness as algaecides, acaricides, fungicides, and herbicides.

Organics

Organics are carbon-containing pollutants, including VOCs (e.g., benzene), SVOCs (e.g., PAHs), 1,4-Dioxane, per- and polyfluoroalkyl substances (PFAS) and pesticides, monitored for environmental and health impacts.

Collection Methods:

Pesticides, SVOCs, and Acid Herbicides

- Collect samples in a 1-gallon amber glass jug with a Teflon-lined cap, per EPA Methods 525 (pesticides), 625 (SVOCs), and 515 (herbicides).
- Perform a surface grab at 0.15 m depth to capture runoff-related pollutants.
- Add sodium thiosulfate (pre-measured packet) to neutralize chlorine, then cool to 4°C.
- Complete a chain-of-custody form, detailing collection time and preservatives.
- Holding time is 7 days, discard samples exceeding this limit.

Purgeable Organics (VOA)

- Use three 40-mL Teflon vials per EPA Method 524. For non-chlorinated waters, select vials pre-preserved with sodium bisulfate (NaHSO4).

- Submerge vial and cap at 0.15 m depth, fill with no water turbulence, and cap underwater with no air space to prevent volatilization.
- For chlorinated waters: Add 0.6 g ascorbic acid to an empty vial, fill/cap underwater, then uncap above water to add 0.25 g sodium bisulfate, recapping with no air space.
- Place vials in a Ziploc bag in a cooler at 4°C.
- Include a trip blank (lab-filled vial with deionized water) in a separate Ziploc bag in the same cooler. Analyze to detect contamination (e.g., VOCs > detection limit).
- Holding time is 14 days with preservation.
- Collect field blanks and duplicates at 10% of sites to assess contamination or variability.

1,4-Dioxane

- Use four 40-mL glass vials with Teflon-lined screw caps per EPA Method 522.
- Submerge vial and cap at 0.15 m depth, fill with no water turbulence, and cap underwater with no air space to prevent volatilization.
- Place vials in a Ziploc bag in a cooler at 4°C.
- Include a trip blank (three lab-filled vials with deionized water) in a separate Ziploc bag in the same cooler. Analyze to detect for contamination.
- Holding time is seven days with preservation.

PFAS

- While wearing powder-free nitrile gloves, fill one 250-mL polypropylene bottle with PFAS-free blank water for your sampling site field blank as per EPA method 1633. Double bag this bottle and store in a cooler at 4°C.
- Fill three 250-mL polypropylene bottles per EPA method 1633 with source water to halfway up bottle neck threads, headspace is needed for lab to run analysis. Never store cap on boat surface. These bottles are then double-bagged and stored in cooler with blanks at 4°C.
- Include a separate double-bagged trip blank (lab-filled bottle with PFAS-free blank water) in the same cooler.
- Samples should be taken upstream of the boat to avoid contamination.
- Only PFAS samples should be stored in the cooler together.

- Holding time is 14 days until extraction.

Analysis Requests

List suspected pesticides/organics (e.g., atrazine, benzene) on the laboratory form, referencing the Central Laboratory's analyte list or email edith.henderson@deq.nc.gov. This enables targeted analyses, reducing turnaround time.

V. SEDIMENT COLLECTION AND PRESERVATION

1. COLLECTING SUSPENDED SEDIMENT

Samplers and Applications

For more descriptive information about these samplers see references (Inter-Agency Committee on Water Resources 1965).

U.S. DH-48 – When in wadable streams

U.S. DH-59 [Equal Width Increment Method (E.W.I.)]

Used when:

- Too deep for wading but less than 15 feet deep.
- From low bridges.
- Velocities less than approximately 5 ft/ sec.

Sampling Tips

- Set out safety equipment (cones, high visibility vests, etc.) as necessary and assemble sampling equipment. Note: Prior to using any sampler, it should be thoroughly cleaned and inspected.
- Rinse sampler with distilled water before the first station and between stations to wash away any contaminants.
- Use the upstream side of bridges if possible.
- Go to midstream or the area where most of the flow is occurring. First sampling point must be made where the flow is greatest.

- Lower and raise the sampler at a consistent rate with the nozzle oriented upstream to the bottom, immediately reverse it and raise to above the water surface. Repeat until jar is filled within approximately 3 inches of the top of the jar (350-440 cc). Rate must not exceed 0.4 times the mean velocity and must be fast enough to keep from overfilling.
- If bottle overfills - discard sample, rinse bottle, and collect again. Use a smaller nozzle or a faster transit rate.
- Raise the sampler and pour contents into a clean sample splitter. For cleaning instructions see USGS references. The sample splitter should be rinsed with distilled water before the first station and between stations.
- Sample at the next sampling point and place contents into a mixing churn.
- Ideally try for 3 sampling points, midstream and quarter points, but the situation might indicate otherwise (if maximum flow is not midstream). Sampling points should be equally spaced.
- If more sample is needed for the churn, take a second set of samples at the same transit rate at all verticals.
- Churn sample at a uniform rate of about nine inches per second. Disc should touch the bottom of the tank on every stroke, and the stroke length should be as long as possible without breaking the water surface.
- After churning for about l0 strokes, withdraw sub-samples and place in ½ liter bottles. As sub-samples are withdrawn, maintain churning rate. If there is a break in withdrawals, the stirring rate must be re-established before withdrawals can continue.

Variations on Suspended Sediment Sampling

When suspended materials in the stream are uniformly distributed, a representative sample can be obtained by sampling vertically at one location near the center of the flow.

Use surface or dip sampling instead of depth integrated sampling when:

- Stream velocity is too high.
- Large floating and moving submerged debris is in the stream.
- A depth-integrated sampler is not available.
- The depth of the stream is very shallow.

2. COLLECTING BOTTOM SEDIMENT

Containers and Volumes

Use certified jars for sediment samples as indicated and provided by Chemistry Laboratory.
Use 4 oz or 8 oz glass jars with Teflon lined lids as indicated by the "Soil & Sediment Samples - Containers, Preservation, Hold Time Table"

3. BOTTOM SEDIMENT SAMPLERS, APPLICATIONS, AND PROCEDURES

For more descriptive information about these samplers see references.

Ekman grab (Figure 13)

Locations Suitable for Use:

- Use in soft finely divided littoral bottoms of lakes, ponds, and streams that are free from vegetation (sticks, partially decayed leaves, etc.) as well as intermixtures of sand, stones, and other coarse debris.
- Calm water.
- Low velocity streams.
- Low bridges (messenger can damage spring mechanism if used from high bridges).

Sampling Tips:

- Make sure grab is operating correctly. The grab can cause severe injury. Do not activate unit while holding.
- If sampling from a low bridge, it may be advisable on wide streams to take 3 samples (midstream and quarter sections) and composite them to form one sample in a Nalgene mixing tub.
- Set in open position by locking open the spring-operated jaws.
- Operating procedures are similar to those of the Petersen grab.

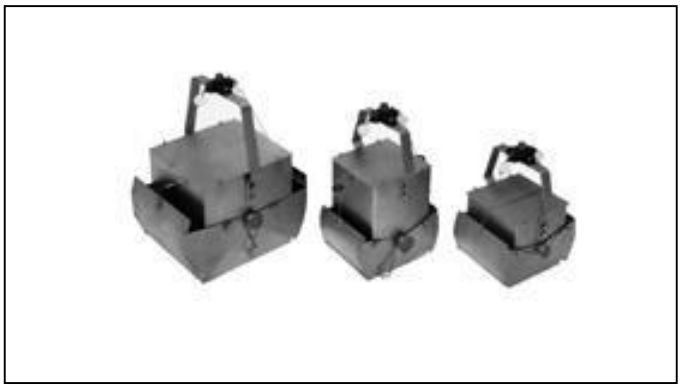


Figure 13. Ekman Grab Samplers

Petersen grab (Figure 14)

Locations Suitable for Use:

- Hard bottoms (sand, gravel, marl, clay, etc.).
- Strong velocities.
- Very deep water.

Sampling Method and Tips

- Use with hoist because of its weight.
- Make sure grab is operating correctly and rinse in water at first station and between stations.
- Move jaws to open position, bring free end of horizontal locking bar into position in the locking notch on upper bar, insert safety pin lock.
- Swing grab over side, remove safety pin lock, and lower slowly to bottom.
- When grab is at the bottom, allow a moment for it to sink into the bottom then slack off on the line.
- Resume tension on the line to close grab.
- Pull grab to surface, swing inboard over a tub and discharge sample.
- Place sample in jar. Approximately one pint of sample is needed.
- If jaws of grab are jammed due to a stick, rock, or other hard object, discard sample, clean grab and sample again.

Figure 14. Peterson Grab Sampler

Ponar grab (Figure 15)

Locations Suitable for Use:

- All types of bottoms except the hardest clays.
- Strong velocities.
- Very deep water

Sampling Method and Tips

- Use with hoist because of its weight.
- Make sure grab is operating correctly and rinse in water at first station and between stations.
- Move jaws to open position, bring free end of the horizontal locking bar into position in locking notch on upper bar and insert safety pin lock.
- Remove safety pin lock and lower sampler slowly.
- When the grab is at the bottom, wait a minute to allow it to sink, and then slack off the cable.
- Lift the sample maintaining tension and raise steadily and slowly to surface.
- Swing inboard and open sampler over a tub to discharge sample.
- Place sample in jar. Approximately one pint of sample is needed.
- If an object is wedged between the jaws, discard sample, clean sampler, and sample again.
- At the conclusion of sampling, replace the safety pin lock.

Figure 15. Ponar Grab Sampler

4. BOTTOM CORE SAMPLERS, APPLICATIONS, AND PROCEDURES

Phleger core sampler (Figure 16.)

Locations Suitable for Use:

- Use with hoist because of its weight.
- Use where water is too deep to use hand coring devices.
- Sampling soft, sandy or semi-compacted sediments.

Phleger Core Sampler Methods

- Make sure sampler and core tubes are clean and operating properly, rinse corer at first station and between stations.
- Lower sampler to bottom, then raise off the bottom approximately one to two meters.
- Drop sampler again to collect core.
- Swing sampler inboard over a Nalgene tub.
- Remove tube and core, measure out top two inches of core.
- Place this portion of core into jar.
- Repeat sampling until approximately one pint of sample is obtained.

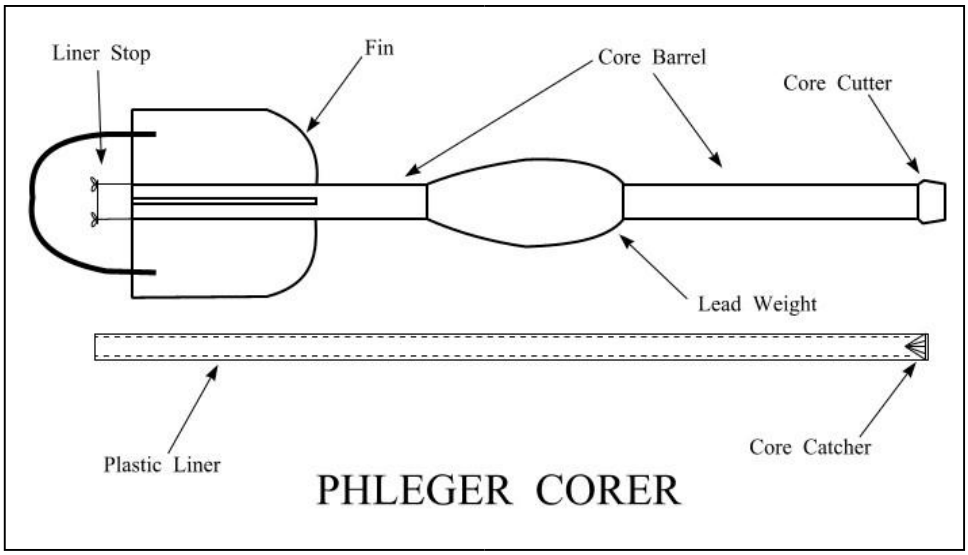


Figure 16. Phleger Corer Diagram

Wildco Light Duty Model 2414 Core Sampler

Locations Suitable for Use:

- Use by hand or at the end of a line.
- Where sediment is relatively soft.

Wildco Light Duty Model 2414 Core Sampler Methods

- Make sure sampler and core tubes are clean and operating properly, rinse corer at first station and between stations.
- Lower sampler to bottom, raise again and drop if necessary to take sample.
- Remove plastic core, measure out top two inches of core.
- Place this portion of core into jar.
- Repeat sampling until approximately one pint of sample is obtained.

<u>Hand coring device– for shallow water use.</u>

Procedure for hand coring device:

- Make sure sampler is clean before using. Rinse before first station and between stations.
- Take sample by turning sampler into sediment.

- Remove sampler and core.
- Measure out top two inches of core.
- Place this into jar.
- Repeat sampling until approximately one pint of sample is obtained.

Hand Sampling

Method

- Face upstream in shallow, wadable streams.
- Make sure that spoon or scoop has been thoroughly cleaned.
- Scoop the sample directly into the jar and get a representative sample. It may be advisable to take several samples and consolidate (midstream and quarter points).

VI. STANDARD CLEANING PROCEDURES

1. GENERAL

The procedures outlined in this section are to be used by all personnel to clean sampling equipment and sample containers that aren't already clean prior to field use. Any deviation from them may result in the contamination of samples. Please remember to use any potentially harmful chemicals in a fume hood. For ISB purposes, these include hexane, acetone, or alcohol such as methanol or ethanol. Use these chemicals under a fume hood with proper ventilation. If in doubt, please refer to the compound's Safety Data Sheet (SDS) for best practices.

All equipment and reusable sample containers used to collect samples will be identified before leaving for the sampling event. During the sampling event, any equipment or sample container malfunctions should be noted. Equipment or reusable sample containers needing cleaning or repairs should not be stored with clean equipment, sample tubing or sample containers. Equipment, reusable sample containers, disposable sample containers, and sample tubing that are not used during a sampling event may not be replaced in storage without being re-cleaned if these materials are transported to a facility or study site where herbicides, pesticides, organic or other toxic materials are present or suspected of being present. All portions of unused coils of tubing that are returned shall be re-cleaned before being re-stocked. If these materials are transported to a facility in connection with a routine inspection or study where toxic or organic materials are known or suspected of being present, they need to be cleaned before being placed back in storage.

Sufficiently clean equipment and sample containers should be transported to the field so that an entire study can be conducted without the need for cleaning equipment in the field. However, this will not always be possible when using coring equipment, dredges, buckets, water samplers, pumps and other such equipment. Field cleaning procedures are included to cover these special cases in part 7 of this section.

2. AUTOMATIC SAMPLING EQUIPMENT

General Cleaning - *For All Automatic Samplers*

- The exterior and accessible interior (excluding the waterproof timing mechanism) portions of automatic samplers will be washed with phosphate free laboratory detergent and rinsed with tap water.
- The face of the timing case mechanism will be cleaned with a damp cloth.
- All sample intake tubing will be discarded after use. Pump tubing should be cleaned with pesticide grade solvents.
- New pre-cleaned, silicone pump tubing (see section on cleaning tubing) will be installed with the aluminum or Teflon tubing caps intact.
- When using the samplers for collecting samples for metals and/or organic samples, the metal distributor tubes should not be used for this purpose.
- The automatic samplers should not be used for collecting samples for organic analyses in the individual bottle mode since there is no way to properly clean the distributor plate to remove any residual organic compounds. The sample tubing headers may not be used to collect samples for organic analyses for the same reason.

ISCO Specific Cleaning Procedures

The following cleaning procedures are provided as guidelines. Whenever possible please follow the manufacturers' cleaning instructions

Automatic sampler rotary funnel, distributor and metal tube.

- Use only for non-organic sample collection using individual sequential bottles.
- Clean with hot water, phosphate free laboratory detergent and a brush.
- Rinse thoroughly with hot tap water.
- Rinse thoroughly with distilled water.
- Replace in sampler.

Automatic sampler headers

- Rinse entire header with hot water, a bottle brush, and phosphate free laboratory detergent.
- Disassemble header and rinse thoroughly with hot tap water, using a brush to remove particulate matter and surface films.
- Rinse plastic portion of the header with 20 percent nitric acid. Do not use acid on metal parts.
- Rinse thoroughly with tap water.
- Reassemble header and rinse with distilled water.
- Let dry thoroughly and wrap with aluminum foil.
- Headers may not be used when collecting samples for organic analyses.

Glass reusable composite containers (2 ½, 3- and 5-gallon capacities)

- After using, rinse with water in the field, seal with aluminum foil to keep the interior of the container wet and return to the laboratory.
- Wash thoroughly with hot tap water and phosphate free laboratory detergent, using a bottle brush to remove particulate matter and surface film.
- Rinse thoroughly with hot tap water.
- Wash with 10 percent nitric acid.
- Rinse thoroughly with tap water (at least 3 times).
- Rinse thoroughly with distilled water (at least 3 times).
- Rinse thoroughly with acetone (pesticide grade). Caution: Acetone must be removed before using. Residual acetone will interfere with certain analyses.
- Rinse twice with DI water. Allow to air dry.
- Cap with aluminum foil or Teflon film.
- Do not use composite containers used to collect samples at facilities manufacturing pesticides, herbicides or other toxic or noxious compounds. These are to be properly disposed of at the DWR Chemistry Laboratory.
- Glass composite containers used to collect in-process wastewater samples at industrial facilities will be discarded after sampling.

- Any bottles that have a visible film scale or discoloration remaining after this cleaning procedure are to be discarded.

Glass sequential sample bottles (automatic sampler base for sequential mode)

- Rinse bottles in the field after using with tap water and seal with aluminum foil or cap for return to laboratory.
- Rinse thoroughly with hot tap water.
- Wash with 20 percent nitric acid.
- Rinse thoroughly with tap water.
- Place in dishwasher - phosphate free detergent cycle followed by tap and distilled water rinse cycles.
- Replace in covered, automatic sampler base, cover with aluminum foil for storage.

Bottle siphons

- Use a new siphon for each sampling location.
- Pre-rinse the 3/8-inch Teflon tubing (used to make siphons for organic analyses) as in Teflon tubing cleaning instructions.
- Flush the PVC 3/8-inch tubing used for samples other than those collected for organic analyses with sample before use.

Teflon composite mixer rods

Use the sample cleaning procedure outlined for glass reusable composite containers above.

Automatic sampler rubber pump tubing

- Only new pre-rinsed tubing should be used for each automatic sampler set up.
- Rinse tubing with hot tap water for five minutes.
- Working in a fume hood, rinse outside of tubing with hexane.
- Install in automatic sampler.
- Cap both ends of tubing with aluminum foil or Teflon film.

Teflon sampler tubing (pure Teflon or Teflon lined)

- If required length is known pre-cut Teflon tubing or clean l00 feet coil intact.

- Working in a fume hood, rinse outside of tubing with hexane.
- Working in a fume hood, flush interior of tubing with hexane.
- Air dry.
- Cap each end of tubing with aluminum foil or Teflon tape and completely wrap the coil of Teflon tubing with aluminum foil to prevent contamination.

Polyvinyl chloride sample (PVC) tubing (1/18, l/l4, or 3/8 Inch)

- Use only new tubing.
- Use in selective sampling where organics are not of concern.
- Flush the tube with sample immediately after the sampler is set up at the sampling site to remove any residues from the manufacturing or extruding process.
- Store tubing in original container and do not remove from this container until needed.

Stainless steel tubing

- Tubing will be flushed in the field with tap water after use and cleaned as follows upon return to the laboratory:
- Wash with phosphate free laboratory detergent and a long bottle brush.
- Rinse with hot water for 5 minutes.
- Working in a fume hood, rinse with acetone.
- Rinse with distilled water for one minute.
- Air dry.
- Working in a fume hood, rinse with hexane.
- Completely wrap tubing, including ends, with aluminum foil to prevent contamination during storage.

3. MISCELLANEOUS SAMPLING AND FLOW MEASURING EQUIPMENT

Miscellaneous flow measuring and sampling equipment should be washed with phosphate free laboratory detergent and rinsed with hot tap water before being stored. For Lablines, rinse at least three times with distilled deionized water.

A different procedure is used for any equipment utilized in organic or toxics sampling.

4. STAINLESS STEEL SAMPLING EQUIPMENT

For collecting samples for organic analyses:

- Follow the procedures given in the Automatic Sampler Section, Glass Reusable Composite Containers, but omit acid rinse.
- Wrap equipment completely in aluminum foil to prevent contamination during storage.

5. OTHER FIELD INSTRUMENTATION

NOTE: Where available, always follow the manufacturer's recommendations for cleaning the device (see Appendices 1-5).

The interior and exterior of Labline samplers should be washed with a mild phosphate-free detergent (liquid dishwashing detergent, for example: Liquinox) and rinsed with tap water before storage. Other field instrumentation should be wiped with a damp cloth. The desiccant in flow meters and other equipment should be checked and replaced, if necessary, each time the equipment is cleaned.

Keep meters clean and in good operating condition. Probes should be rinsed with deionized water at the end of each sampling day, properly stored and cleaned on a regular basis.

6. ICE CHESTS AND SHIPPING CONTAINERS

All ice chests and reusable shipping containers will be air dried before storage. Ice chests and shipping containers should be washed with a mild phosphate free detergent (ex: Liquinox) at the beginning of each sampling season and after there is a sample spill or other suspected contamination event.

7. FIELD CLEANING PROCEDURES

For routine operations involving classic parameter analyses, water quality sampling equipment such as Kemmerers, buckets, dredges, and etc. may be cleaned with sample or tap water between sampling locations. A brush may be used to remove deposits of material or sediment if necessary. Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream gauging equipment should be cleaned with tap water after use and between measuring locations. When sampling equipment (not tubing) is to be utilized for collecting organic or toxic samples, the following cleaning procedure is to be used between sampling locations:

- Clean with tap water and brush if necessary.
- Rinse with pesticide grade acetone.
- Rinse thoroughly with tap water (if available).
- Rinse with distilled or deionized water.

It must be emphasized that these procedures are only to be used in the field. All equipment will be cleaned before storage at the laboratory utilizing the procedures previously outlined.

8. VEHICLES

All vehicles used by staff should be washed on a routine basis. This routine maintenance should minimize any chance of contamination of equipment or samples due to contamination of vehicles. When vehicles are used in conjunction with hazardous waste site inspections, or on studies where pesticides, herbicides, organic materials or other toxic matter are known or suspected to be present, a thorough interior and exterior cleaning should be performed at the conclusion of such investigations. All trash should be discarded at the end of each day to ensure

vehicle cleanliness. Excessive sand, mud, and debris from field sites will be swept out to facilitate equipment cleanliness.

9. DISPOSABLE SAMPLE CONTAINERS

All disposable sample containers will be stored in their original packing containers in a clean, dust free environment. When any packing container is opened, all disposable sample containers inside should be immediately capped if they are found uncapped.

VII. TIME-OF-TRAVEL & DYE TRACING

ISB no longer conducts dye tracing studies, nor do we have the meters or correct FWT dye. If a study is needed, ISB will need to purchase the new equipment and develop a method based on current practices and manufacturer SOPs. The previous SOP can be found below in Appendix 10.

VIII. FLOW MEASUREMENTS

ISB does not conduct routine flow measurements. The previous SOP can be found below in Appendix 11. For special studies, refer to USGS protocol found on their website.

- Discharge Measurements
- Stage Measurements

IX. BATHYMETRY FOR NON-TIDAL WATERS

1. EQUIPMENT

The following equipment is available for bathymetric surveys:

- Water level recorder and/or referenced gauge stations(s).
- Calibrated sounding line(s).
- Lowrance Sonar Unit with GPS and Sonar Logging enabled.
- SD Card (32GB).
- Boat or watercraft with sonar transducer properly installed.
- Computer with SD card reader and internet access.
- CiBioBase account
- ArcGIS software.

2. PRE-SURVEY PREPARATION

Lowrance Unit Setup:

- Calibrate and maintain according to the manufacturer's instructions before use. Check daily in the field against a field measurement of water depth and using a calibrated sounding line, as needed.
- Ensure your transducer is properly installed and submerged. Note the depth below the water's surface that the transducer sits to use later as an offset in bathymetric calculations.
- Set the correct sonar settings:
    - Frequency: 200 kHz.
    - Ping Speed: Set to maximum for dense data points.
    - Chart Speed: Match to boat speed (typically "Normal").
    - Depth Range: Set to "Auto" or manually adjust based on estimated depth.
- Set GPS recording to 1-second intervals for high spatial accuracy.
- Insert SD Card into the unit.

- Sounding lines are to be calibrated against steel surveyor's chain and shall be accurate to 0.1 foot.

3. DATA COLLECTION

Survey Design:

- Record the water level daily (for non-tidal waters) using a referenced gauge station.
- Use a zig-zag or parallel track pattern to ensure coverage. Some surveyors prefer to create a grid pattern in ArcGIS and upload these points to the Lowrance prior to the survey. This can help to keep parallel tracks or zig-zags evenly spaced.
- Maintain a consistent speed (2-5 mph ideally).
- Avoid sharp turns or sudden speed changes.
- Ensure full waterbody or area coverage, including shoreline contours and deeper zones. Note that you generally do not want to operate in less than 1 meter of water due to the fact that at these low depths propwash can impact the readings of the sonar unit. The shapefile of the shoreline can be used at a later date to clip the resulting raster file.

Sonar Logging:

- Begin sonar logging before entering the survey area, if possible.
- Navigate to the “Record Sonar” or “Log Sonar” menu.
- Ensure that files are being recorded to the correct memory card location and that the files are being saved in .SL2 format.
- Name each file including the waterbody name, date, collector’s initials and a unique identifier.
- Stop sonar logging and begin a new recording every hour. This ensures that if a file is corrupted all of the data for that day is not lost.
- Once the survey is complete, stop sonar logging for the current file. Power down the unit and remove and label the SD card.

4. DATA PROCESSING

Uploading, Interpolating and Exporting data using CiBioBase:

- Insert SD card into a computer and copy the .SL2 files into a backup folder.
- Login to your CiBioBase account.
- Click “Upload Trip”.
- Fill out metadata: waterbody name, date, GPS zone, surveyor, etc.
- Upload sonar log files.
- Assign correct survey type (bathymetry).
- CiBioBase automatically processes uploaded sonar logs through their proprietary algorithm.
- Wait for email confirmation that maps are ready.
- View the maps via “Trip Viewer”.
- Use the “Export” function to download.
    - Bathymetric contours (shapefile or KML).
    - Interpolated raster surfaces (GeoTiff, ASCII Grid).

Data Post Processing in ArcGIS:

Following processing by the CiBioBase algorithm, hydroacoustic data are exported as ASCII-grid records using the export function discussed above. These files can then be edited to include an offset for the location of the transducer and other needed offsets (ex: offset based on references depth gauge readings to represent the waterbody at full pool). Files are then imported into ESRI ArcGIS software (coordinate system: WGS84) and converted to raster files using spatial analyst. These files can then be clipped to a given shoreline shapefile.

X. WATER QUALITY VESSEL OPERATION

Water quality investigations frequently require DWR personnel to work in locations that are accessible only by boat. This necessitates that field staff be thoroughly trained in the safe operation of those boats and become familiar with the general maintenance and the particular operation of each vessel. This boating SOP provides a general operating guide to ensure that all boating and trailering activities are carried out in a safe manner and that all boats and motors are operated in a manner that reduces the frequency of repair. All field personnel should have a valid boating safety certificate and should read and thoroughly understand this SOP prior to operating any DWR boat.

1. BOAT SAFETY

Supplies Needed On-Board

Fire Extinguishers - Before operating boat, familiarize yourself with where the fire extinguisher is located. Check to make sure that it is fully charged.

Sound Producing Devices - Boats should be equipped with a can type air horn or a manually operated whistle.

Paddles or Oars - All boats should be equipped with oars or paddles.

Visual Distress Signals - When operating boats in coastal waters, the boat must be equipped with a flare kit. The kit should include handheld flares and a flare gun for aerial type flares.

PFD's (Personal Floatation Devices) - **All DWR employees are required to wear life preservers while under power on all DWR boats.** Boats will be equipped with a type 1, 2, or 3 PFD of suitable size for each person on board and a throw-able floatation device (throw cushion, flotation ring).

Lights - When operating a boat at night, the boat must display the front green and red navigational light and the rear beacon light. If planning to operate at night, the lights should be checked before leaving the loading area.

Safety Check

Weather - Check weather reports before leaving shore and remain watchful for signs of bad weather. Tune into the National Weather Service Report, on a

Marine radio or mobile device, periodically to check weather conditions, small craft advisories, gale warnings, etc. Do not go out on the water during lightning storms.

Care and Maintenance - All equipment and supplies should be properly secured. Keep decks and other spaces clean, free of clutter and trash. The vessel should be free of fire hazards with clean bilges and in good condition. Inspection and required maintenance on a regular schedule will ensure the hull and superstructure remain sound.

Ensure all repairs are made properly and with marine rated parts. Always carry a toolbox and know how to make minor repairs.

Communications - When operating in remote areas it is always a good idea to bring along a cellular phone for cases in which assistance may be needed. Two-way radios or cellular phones should be used when operating with two or more boats. When operating in coastal waters always bring along either a hand-held portable marine radio or a fixed mounted marine radio.

2. FIXED MOUNT/CONSOLE TYPE BOATS

Trailering

Pre-Trip Check and Preparation

- Install 1 ⅞ or 2" trailer ball to trailer hitch depending on the trailer.
- Disengage clamping mechanism on boat trailer tongue.
- Back vehicle up to boat trailer, with trailer tongue directly over the center of the trailer ball.
- Lower trailer jack so that the trailer tongue fits over the trailer ball.
- Engage the clamping mechanism (all the way) onto the trailer ball. Lock with a 2640 Master Lock.
- Hook up both safety chains by crossing the chains and hooking to holes on the trailer hitch. Do not tow trailer without safety chains.
- Hook up brake line cable to eye bolt attached to the vehicle.
- Plug in trailer lights and check the lights for proper operation.
- Secure gunwale boat strap.

- Check the bow eye to make sure safety chain is hooked up and winch is locked down securely.
- Periodically check the clamping mechanism on the trailer tongue to assure that it is screwed down all the way.
- Conduct an inspection, walk around the boat and trailer:
  - Test to see if the boat motor starts before traveling.
  - Check level of the engine oil on 4-cycle boat motors.
  - Check the trailer tire pressure and adjust if necessary.
  - Check the condition of the axel grease. Add grease as needed.
- When traveling, stop and check the trailer and boat; retighten boat straps as needed. Feel the trailer bearings to see if they are hot. If hot, they probably need to be greased or replaced.
- Trailer slowly over speed bumps and holes.
- When backing into parking areas, do not let back of trailer come into contact with curb to avoid damaging license plate bracket or trailer lights.

Boat Launching

Unloading

- Upon arrival at the boat ramp check the ramp to make sure it is suitable for launching, including checking that the water level is high enough for proper launching.
- **Install all the boat plugs**; check inside the bilge to make sure that the plug is installed.
- Remove the boat strap.
- Load the boat with equipment.
- Unplug trailer lights.
- Make sure the motor is in the "up" position before launching the boat.
- Keep winch "locked" until boat is in the water.
- When the boat is in the water lower the motor and then start the motor (see motor operating instructions).

- While one person is operating the boat another person should be manning the trailer winch.
- Unhook winch cable from bow eye, but do not remove safety chain until the boat is running and idling.
- If needed, back the vehicle up slightly and press the brake to bump the boat off the trailer.
- When boat is clear from the trailer, pull the vehicle out of the ramp **slowly** and park it.

Loading

- Slowly back the trailer into the water so that the center "guide roller" is visible above water.
- Line up the boat with the trailer and **very slowly** ease the bow of the boat onto the center roller. If boat is off center of the trailer, back up and try again.
- **Do not** approach trailer at a speed that will damage the boat hull or trailer if the center roller is missed!
- The person manning the trailer winch should signal the boat operator to go left or right, or to tell the boat driver to back off if they are going to miss the center roller.
- Once the bow is on the center roller, slowly advance the boat up onto the trailer as far as it will go. If it does not reach the stanchion, then hold the boat in position until the person manning the winch can get out enough cable to hook to the bow eye.
- Once the cable is hooked and tension is maintained then power down the motor and cut it off.
- Winch the boat onto the trailer until the bow is snug against the stanchion.
- Lock down the winch gear.
- Raise the motor to the "up" position and flip down the tilt lock bar, then lower the motor until it presses against the tilt lock bar.
- **Slowly** drive out of the boat ramp to the parking lot.
- Remove the boat plugs.
- Unload the boat.

- Hook up boat strap.
- Plug in trailer lights.
- Make sure that all aerials and/or Bimini tops are down.
- Walk around trailer and **double check everything!**

Boat Operation

Fueling

- Fill oil reservoir to required fill level with appropriate motor oil.
- When fueling boats with 2 cycle outboard motors without an oil reservoir add one pint of 2 cycle outboard motor oil to the gas tank for every six gallons of gasoline. Use marine fuel stabilizer in all fuel tanks.
- 4 cycle outboards do not require oil/fuel mixing.
- Fill tank to the maximum fill level.

Motor Operation

- Switch the battery "PERKO" switch to the "ALL" position.
- Pump the gas primer ball until it is tight.
- Lower motor into the water using hydraulic trim switch on the throttle lever.
- For motors with a choke, when starting "cold" the choke must be engaged. To engage the choke, push the ignition key in as far as it will go, then turn the key clockwise until the motor starts. If motor does not start within five seconds **do not** continue to engage the starter. Repump the primer ball and try again.
- Once the motor starts, disengage the choke and let the motor idle.
- Once the motor has been given ample time to warm up, back the boat off the trailer.
- Let motor idle down before changing from reverse to forward. Between forward and reverse, make a brief stop in neutral.
- If working in open water with ample depth for boat running, advance the throttle to plane out the boat, adjusting the trim if necessary.

- Once proper plane is achieved, throttle the boat back to 3/4 (approximately 4200 rpm) throttle. **Do not run the boat at full throttle.**

3. SMALL BOATS WITH PORTABLE MOTORS

Trailering

For the most part the same rules apply that were covered in the previous section with a few exceptions:

- Install appropriately sized trailer ball to trailer hitch.
- Flip down locking switch on trailer tongue and lock with a 2640 Master padlock.

Boat Operation

Fueling

- Obtain gas tank from storage cabinet.
- Make sure the tank selected is equipped with the proper fuel line connections for the motor you will be using.
- Most of the fuel tanks have a capacity of 6.6 gallons. Leave some head space in the fuel tanks - **do not overfill**.
- If a 2 cycle motor, add one pint of 2 cycle outboard motor oil for every six gallons of gasoline unless motor requires different oil and ratio.

Motor Operation

- Select the proper motor for the boat that is to be used.
  - 5 hp for Jon boat and 12' Alumacraft.
  - 15 or 25 hp for 14' Alumacraft.
- Place outboard motor in the **center of the transom** and completely tighten the clamping screws on the outboard motor.
- Place tank in boat and connect to motor to assure proper fitting.

- Run a chain through the gas tank handle, then through the handle on the outboard motor, and then through the hole in the boat and lock with a pad lock.
- Secure the motor to one side with a bungee cord to keep motor from swaying back and forth while going down the road.
- To run: connect fuel line to motor, and pump primer ball.
- Pull choke knob if motor is "cold".
- Turn tiller throttle lever to "start" position.
- Pull starter cord. If motor does not start after one or two pulls then pump the primer ball and try again.
- Once motor starts push choke lever in and let it run for about a minute then idle down with throttle lever.
- To put motor in gear, make sure motor is idling low and pull the gear lever forward to go in a forward direction. To go in reverse, push gear lever backward to reverse position with a brief stop in neutral.
- Once motor is in gear then throttle up the motor, once the boat is planned out back off the throttle about 1/4 turn.
- **Do not run the motor at full throttle, run at 3/4 throttle.**
- To turn the motor off, press the red kill button located next to the choke lever. On some motors the kill button is located on the end of the tiller.

4. TROUBLESHOOTING: FOR ALL BOATS

Problem - No power to starter

Check to see if "PERKO" switch is on the "ALL" position.

Check to see if throttle lever is in the neutral position.

Check battery terminal connections.

Check main fuse in outboard motor.

Check fuses inside console.

Problem - Motor is Turning Over but Will Not "Fire" or Start

Check to see if gas line is connected to motor.

Check to see that primer ball has been pumped until tight.

Check to make sure "deadman's" or kill button is clipped.

Make sure air vent screw is open on gas can.

Check spark plug wires, replace spark plugs (they may be fouled).

Problem - No Water is Coming Out of Flow Hole

Do not continue running motor. Shut down and attempt to unclog flow hole with a coat hanger or similar object.

If problem persists, do not use the boat. If out on the water when this occurs, get towed back to boat ramp.

Problem - Extreme Cavitations or "Porpoising"

Adjust trim with up and down button on throttle lever.

Make sure there is not an excessive amount of water in bilge.

Adjust the weight distribution of equipment and personnel in the boat (trim the boat up and shift weight).

XI. LAKES SAMPLING

Field data collection procedures for reservoirs and lakes differ from that of streams and rivers due to the differences in water depth and hydrology. This section focuses on procedures specific to physical and chemical water quality sampling of lakes.

1. FIELD PREPARATION

Pre-Sample Preparation

- Preparation of the lake sampling packet:
  - Sample tags and lab sheets must be legibly handwritten with permanent black ink. Adhesive labels for the sample tags may be prepared on a laser printer.

    - Include maps showing the locations of the sampling stations. Electronic copies of lake maps are on the ISB shared drive (Lake Maps folder).
    - Include special instructions and point-of-contact information as needed.
- Contact responsible parties at all publicly owned lakes several days in advance of sampling. Contact names are in the lakes file, Point of Contact Excel form, or in the Lakes Database. Changes in contact information will be noted and provided to the Lakes Database Administrator so that the database can be updated.
- Confirm availability and working condition of boats, motors, and vehicles on the group calendar.
- Verify the lake stations on the map with the station numbers on the lab sheets and tags.
- Check sampling kit for gloves, preservatives, gear, pens, and bottle kits.
- Always include extra bottles in case of accidents, defective bottles, and/or discovery of algal blooms or other environmental conditions that justify additional samples.
- Ensure that In-Situ meter, tablet, and any batteries needed for the sampling event are charging appropriately.
- Download Google Maps directions if expecting areas with low/ no cell service.

<u>Field Equipment Needed</u>

- Aquatic Plant & Algal Bloom Report Forms (Now on Survey 123).
- Cooler(s) with ice.
- Field Observation & Stratified Data Forms (Now on Survey 123).
- Lab Sheets and Tags in sealed bag.
- Preservatives- Lugols solution, H_2SO_4, HNO_3.
- Calibrated Water Quality Meter.
- Labline with Rope.
- Camera.
- Sample Bottles.
- Nitrile Glove
- Calibrated backup meters.
- Pens and Pencils.
- Maps.
- PDF.
- Boat Oars.
- Gas Tank for boat.
- Boat Plug & Anchor.

- Winter- cold weather suit (as needed).
- First aid/safety box/PPE.
- Electric motor & 2 fully charged batteries (if needed).
- Secchi Disc with line marked in 1-centimeter increments.

Calibration Materials-(*e.g. Calibration solutions, calibration sheets, tools as needed, charging cables).*

Field Sheets

Make sure that the field sheets (Figure 5) are carried to the lake stations along with pens or any non-erasable ink for writing and a clipboard. Clearly write the station number, date, time, depth, dissolved oxygen, temperature, pH, conductivity, and Secchi are recorded on the field sheet. At the top of the field sheet record, the name of the water body, which meter is used, and the names of the field samplers are also recorded.

All field observations including weather conditions and watershed characteristics/anomalies are recorded in Survey 123 at time of sampling.

2. LAKE DATA COLLECTION

Lake Physical Data Collection Methods

- All parameters should be taken within 100m of the fixed site location
- Secchi depth measurement is taken as described in Section III of this document.
- Dissolved oxygen, water temperature, conductivity and pH are measured with a multiparameter meter beginning at the surface of the lake (0.15 meters from the surface).
- From the surface to either the bottom of the lake or to a depth of 10 meters, physical measurements are recorded at 1- meter increments.
- Below ten meters, physical measurements are recorded at 5-meter increments until the bottom of the lake is reached.

Lake Water Sample Collection

Description

- All parameters should be taken within 100m of the fixed site location
- Samples will be collected at the surface, photic zone, or at the bottom, which are described below and in detail in Section I.
- If "SUR" is part of the station number, the sample is to be collected at the surface. If 'BOT" is part of the station number, the sample is collected one foot above the bottom. If "SUR" or "BOT" doesn't accompany the station number, the sample is collected in the photic zone.
- As with all samples for laboratory analysis, a lab sheet must be completed as described in Section II of this SOP.
- Detailed definitions of these parameters and methods of collection are found in Section III.

Types of Typical Lake Samples

a. **Surface Grab** – Surface grab samples (1,4-dioxane, PFAS, chloride, hardness, fecal coliform bacteria, and metals) are collected 0.15 meters below the water's surface and this can be done by hand dipping the bottle. The bottle top and bottle opening should be protected from contamination. Grasp the bottle near the base and plunge its mouth down into the water, avoiding surface scum. Position the bottle away from the hand of the collector, the shore, the side of the sampling platform, or boat.

b. **Photic Zone** – the photic zone is defined as the column of water in the lake from the surface down to a depth equal to twice the Secchi depth measurement. Photic zone samples (residue, turbidity, chlorophyll *a*, nutrients, and phytoplankton) are collected by raising and lowering the Labline at a steady speed within the photic zone until it is full. A description of this procedure is given in this SOP in Section I.

c. **Bottom sampling** - Use of a discrete depth water sampler (Van Dorn, Niskin, or Labline) is accomplished by inserting the two plugs in the top of the Labline and lowering it just above the bottom of the lake. This needs to be done gently so as not to stir up sediments on the bottom. The plugs can be released by firmly jerking the rope on the Labline. Wait until the Labline is full before bringing it back to the surface. This can be

determined by observing air bubbles from the sampler rising to the lake surface, the stopping or feeling the weight of the Labline at the end of the rope.

Field Records and Information

- Photographs are to be taken of various locations on each sampled lake to record the shoreline and lake characteristics. In particular, an unusual shoreline/ watershed activity, aquatic plants, algal blooms, or other water quality issues are to be photographed with a camera or phone.
- Comments and questions from citizens, lake managers, water treatment plant supervisors, etc. are to be recorded (written) along with contact information and individual's name and title (if any). This information will be submitted to the Lakes Database Administrator upon return to the ISB office.

Typical Lake Sampling Parameters

Below is a list of typical lake water sample types. Descriptions of how samples are preserved and collected can be found in Section I and Section IV in this document.

Physical Parameters

Conductivity.	Temperature (°C).
Dissolved Oxygen(mg/L).	Secchi Depth.
	pH.

Chemical Parameters

Nutrients.	Hardness.
Residue.	Metals.
Turbidity.	Chlorophyll *a.*

*Additional parameters may be collected based on specific lake conditions and/or requests.

Biological Parameters

- **Fecal coliform bacteria:** Water samples are collected at the surface of the lake.
- **Phytoplankton:** Water samples are generally collected as a photic zone sample. Bloom samples may be collected at the surface of the lake, as needed.
- **Aquatic Plants:** Record information using Survey 123 along with a specimen if there appears to be problematic aquatic plants or for identification. Include a map of the location showing where the plant specimen(s) were collected.
- **AGPT (Algal Growth Potential Test):** These samples are collected annually after consultation with EPA. All lab analyses are performed by the EPA Region IV laboratory in Athens, GA. In general, the EPA laboratory prefers to have a list of lakes, sample stations, and number of samples to be collected by the end of August for the next sample season. Intercommunication with ISB and Basin Planning should be utilized for site selection. Samples should be collected during the growing season, typically in July and/or August. The EPA furnishes the bottles (1 liter, plastic), sample labels, COC, prepaid shipping labels, and coolers. Samples are collected in the photic zone with an intergraded sampler (Labline), and no preservative is used. If the sample station is too shallow to utilize the intergraded sampler, then a surface grab is acceptable. Samples for AGPT should be collected with minimal headspace and stored in the dark on ice or in the refrigerator at 4 °C. Samples may also be frozen if the laboratory will not receive them within 5 days of collection, or to facilitate storage and/or transport. ISB typically freezes these samples and ships them in groups. If samples will be frozen, headspace of approximately 1-2” should be left in the container to allow expansion of ice. Electrical tape (or similar) should be used around the cap to prevent sample loss or contamination. AGPT samples can be stored at 4 °C for up to 5 days prior to sample processing or may be frozen at −20 ˚C for up to 180 days. The samples are shipped overnight back to the EPA Athens, GA Region IV laboratory for analysis.

<u>Lab and Field Sheets</u>

All lab sheets should be **legibly** filled out with applicable dates, times, depths, etc.

a. The same time is recorded on lab sheets for the same station. A field observation sheet should also be filled out and any other notable features recorded in Survey 123.
b. Any notes of unusual observations of lake water quality or shoreline activities that could impact water quality should also be submitted.

3. LAKE DATA MANAGEMENT

Data specific to the Intensive Survey Branch Lake Monitoring Program collected prior to 2006 are warehoused in the Lakes Database. This historical database is maintained but not updated with new data. Chemical and physical data collected from 2006 to current are housed in the Labworks Database.

- Physical field data are currently collected using the VuSitu application to record data continuously with a recording rate of every 2 seconds. The field operator of the InSitu probe watches the live readings and marks the datapoint at which the readings stabilize for the surface, each subsequent subsurface meter up to 10 meters and then every 5 meters thereafter ending with a final reading approximately 0.25 meters above the waterbody sediment interface. The recording is then saved locally to the tablet and uploaded to an FTP site.
- Additional field data and observations are recorded in the Survey123 form “Intensive Survey Lakes Field Form” and uploaded to the Survey123 cloud database.
- VuSitu field csv files are downloaded and processed by the Ambient Lakes Monitoring Coordinator. Processing includes checking files for accuracy, removing extraneous recording such that only one record per depth (marked by the field operator) is included in the file, file naming (naming convention: LabNumber_YYYY-MM-DD_StationCode), ensuring that the data start and stop in the correct locations in the csv and saving as an xlsx file.
- Once processed the Database Administrator is responsible for merging the files, processing them through the use of the “PhystoLabworks” database in order to format them for LabWorks upload, checking the data for outliers and deviations from historical data and uploading verified data to the LabWorks database to the fields associated with the assigned lab

number. Additionally, the Database Administrator manually enters the Secchi disk readings recorded in the survey123 form “Intensive Survey Lakes Field Form” into the LabWorks database.

- Chemical water quality results are uploaded to the LabWorks database to the appropriate fields associated with the assigned lab number by the Central Chemistry Lab of DWR independently of the physical data upload.

XII. SEDIMENT OXYGEN DEMAND

ISB no longer conducts sediment oxygen demand studies. If a study is needed, ISB will update their SOP based on current EPA methods found on their website. The previous SOP can be found below in Appendix 12.

XIII. REFERENCES

American Public Health Association. 1992. Standard Methods for the Examination of Water and Wastewater, 18th edition. Washington, D.C.

Buchanan, T.J., and W.P. Somers. 1969. Stage Measurement at Gauging Stations. Techniques of Water-Resources Investigations of the United States Geological Survey, Book 3, Chapter A7. United States Geological Survey.

------. 1973. Discharge Measurements at Gauging Stations. Techniques of WaterResources Investigations of the United States Geological Survey, Book 3, Chapter A8. United States Geological Survey.

Howard, H., 1988. U.S. Environmental Protection Agency, Region IV, Athens Ga. personal communication.

Hutchinson, G.E. 1975. A Treatise on Limnology. John Wiley and Sons, Inc., New York.

Instrument Specialties Company. 1988. Instruction Manual Model 2700 Sampler. Lincoln, Nebraska.

Instrument Specialties Company. 1991. 3700 Portable Sampler. Instruction Manual. Lincoln, Nebraska.

Inter-Agency Committee on Water Resources. 1965. Instructions for sampling with depth integrating suspended-sediment samplers, US DH-48 and DH-59. Measurement and Analysis of Sediment Loads in Streams, Report J. St. Anthony Falls Hydraulic Laboratory, Minneapolis, Minnesota.

Kittrell, F.W. 1969. A Practical Guide to Water Quality Studies of Streams. United States Department of the Interior. Federal Water Pollution Control Administration. 135 pp.

Lawhorn, D., 1988. U.S. Environmental Protection Agency, Region IV, Athens Ga. personal communication.

Murphy, P. J. , Hicks D. B., 1986. In-Situ Method for Measuring Sediment Oxygen Demand. Pages 307-322. Sediment Oxygen Demand. Processes, Modeling and Measurement. Institute of Natural Resources, University of Georgia.

Sawyer, Clair N. and Perry L. McCarty. 1967. Chemistry for Sanitary Engineers. McGraw Hill, New York. 518 pp.

Smoot, G. F., and C. E. Novak. 1968. Calibration and maintenance of vertical-axis type current meters. Techniques of Water-Resources Investigations of the United States Geological Survey, Book 8, Chapter B2. United States Geological Survey.

United States Environmental Protection Agency. 1976. Quality Criteria for Water. Washington, D.C. 256 pp.

------. 1980. Standard Operating Procedures and Quality Assurance Manual. Athens, Georgia.

------. 1994. Federal Register. Volume 59, No. 20. 40 CFR Part 136. Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Technical Amendments.

United States Geological Survey. 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Reston, Virginia.

------. 1992. Selected Water Quality and Biological Characteristics of Streams in Some Forested Basins of North Carolina, 1985-88, United States Geological Survey WaterResources Investigations Report 92-4129. Raleigh, N.C.

Wilson, Jr., James F. 1968. Fluorometric Procedures for Dye Tracing. Techniques of Water-Resources Investigations of the United States Geological Survey, Book 3, Chapter A12. United States Geological Survey.

XIV. ADDITIONAL RESOURCES

American Fisheries Society, Water Quality Section. 1979. A Review of the E.P.A. Red Book: Quality Criteria for Water. Bethesda, Maryland. 313 pp.

Arthur H. Thomas Co. 1981. Scientific Apparatus Catalog. Philadelphia, Pennsylvania. 1544 pp.

Babbitt, Harold E., James J. Doland and John L. Cleasby. 1967. Water Supply Engineering. McGraw Hill, New York. 672 pp.

California State University. 1991. Operation of Wastewater Treatment Plants - A Field Study Training Program, 3rd Edition. Sacramento, CA. 666 pp.

Grant, Douglas M. 1978. Isco Open Channel Flow Measurement Handbook, 1st edition. Instrument Specialties Company. Lincoln, Nebraska. 221 pp.

Guy, Harold P. and Vernon W. Norman. 1970. Field Methods for Measurement of Fluvial Sediment. Techniques of Water-Resources Investigations of the United States Geological Survey, Book 3, Chapter C2. United States Geological Survey.

Hydrolab Corporation. 1984. Operation and Maintenance Manual for Hydrolab Surveyor II. Austin Texas.

------. 1991. H20 Multiparameter Water Quality Data System Operating Manual. Austin, Texas.

------. 1991. Scout 2 Multiparameter Water Quality Data Transmitter Operating Manual. Austin, Texas.

Kahl Scientific Instrument Corporation. 1980. Catalog. El Cajon, California.

Leupold & Stevens, Inc. 1974. Stevens Water Resources Data Book, 2nd Edition. Beaverton, Oregon. 200 pp.

McKee, Jack E. and Harold W. Wolf, eds. 1963. Water Quality Criteria. California State Water Resources Control Board, Publication 3-A. 548 pp.

New York State Department of Health. Manual of Instruction for Sewage Treatment Plant Operators. Health Education Service, Albany, New York. 243 pp.

North Carolina Division of Environmental Management. 1986. Administrative Code

Section: 15NCAC2B .0100-Procedures for Assignment of Water Quality Standards, 15NCAC 2B:0200-Classifications and Water Quality Standards Applicable to Surface Waters of North Carolina. Raleigh, N.C.

Simmons, Clyde E. 1981. Quality Assurance Plan for Water Quality Activities of the North Carolina District. United States Geological Survey. Reston, Virginia. 53 pp.

Simmons, Clyde E. and Ralph C. Heath. 1979. Water Quality Characteristics of Streams in Forested and Rural Areas of North Carolina. U.S. Geological Survey Water Resources Investigations 79-108. United States Geological Survey.

Smoot, George F. and Charles E. Novak. 1968. Calibration and Maintenance of Vertical-Axis Type Current Meters. Techniques of Water-Resources Investigations of the United States Geological Survey, Book 8, Chapter B2. United States Geological Survey.

United States Environmental Protection Agency. 1974. Methods for Chemical Analysis of Water and Wastes. Cincinatti, Ohio. 312 pp.

------. 1989. Short Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Second Edition. EPA/600/4-89/001. 249 pp.

------. 1991. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Fourth Edition. EPA/600/4-90/027. 216pp.

United States Geological Survey. 1982. Water Quality of North Carolina Streams, United States Geological Survey Water-Supply Paper 2185 A-D. Washington, D.C.

Water Pollution Control Federation. 1970. Operation of Wastewater Treatment Plants. Lancaster Press, Inc., Lancaster, PA. 193 pp.

------. 1971. Simplified Laboratory Procedures for Wastewater Examination. WPCF Publication No. 18.

Welch, P.S. 1948. Limnological Methods. McGraw Hill, New York. 381 pp.

Wildco. Petersen and Ponar Bottom Samplers. Trippensee Publishing Co., Saginaw, Michigan. 19 pp.

------. Wildco-Eckman Bottom Samplers. Trippensee Publishing Co., Saginaw, Michigan. 15 pp.

------. 1974. Wildco Instruments and Aquatic Sampling Supplies, Catalog No. 77. Saginaw, Michigan. 110 pp.

Wilson, James F., Ernest D. Cobb, and Frederick A, Kilpatrick . 1986. Fluorometric

Procedures for Dye Tracing. Techniques of Water-Resources Investigations of the United States Geological Survey, Book 3, Chapter A12. United States Geological Survey.

Yellow Springs Instrument Co., Inc. 1982. Instruction Manual YSI Model 58 Dissolved Oxygen Meter. Yellow Springs, Ohio. 28 pp.

------. 1983. Instructions for YSI Model 33 and 33M S-C-T Meters. Yellow Springs, Ohio. 16 pp.

APPENDICES

APPENDIX 1: DWR's In-Situ Mulitparameter Guidance Sheet

In-Situ Field Meter Pre-Sampling Calibration Instructions

To start the calibration process with the In-Situ field meter and VuSitu app, tap the VuSitu app Ico begin calibration.

- Let the probe reach room temperature. This is especially important for post-sampling checks if the probe has been out in the cold or heat. Field meters and probes should not be exposed to hot/cold temperatures or sun in vehicle trunks and truck beds, if possible. Keeping the probes and meters close to room temperature will allow for more consistent readings and less time waiting for the meter to reach room temperature.
- Press  on the Wireless TROLL Com. Allow the Wireless TROLL Com to connect via Bluetooth to a Bluetooth-enabled mobile device and the VuSitu mobile app. The Wireless TROLL Com doesn't have internal data storage. It allows data transfer between an instrument and VuSitu app.
- Tap the VuSitu menu icon on the tablet and select the connected instrument from the list. Verify that the Wireless TROLL Com serial number that you are connecting to the VuSitu app matches the Wireless Troll Com serial number being selected by the VuSitu app. The VuSitu app will search for Wireless TROLL Coms that are in close proximity to the VuSitu app via Bluetooth.
- The Wireless TROLL Com serial number can be found underneath the yellow protective flap covering the charging port. The Wireless TROLL Com serial number is the top number and can be hard to read (Figure 17).
- The probe and Wireless TROLL Com selected by the tablet may not be the equipment you are trying to calibrate. To change the Wireless TROLL Com serial number selected, select "Access Menu", "Connect", and select

appropriate Wireless TROLL Com serial number. Be sure to turn the Wireless TROLL Com on.

- The Vu-Situ app searches for the Wireless TROLL Com serial number and then once detected, it switches to the multimeter probe serial number during calibration/viewing.
- The multimeter probe serial number is located on the side of the multimeter probe (Figure 18).
- To add a device (Wireless TROLL Com serial number not listed), turn the Wireless TROLL Com on, in VuSitu app select choose or add new device, under Bluetooth see which Wireless TROLL Com serial numbers the app detects (Wireless TROLL Com should be turned on). Select the correct Wireless TROLL Com serial number/power pack.
- On the hardcopy calibration sheet or Survey123 app, add collector's name as first initial and last name, study/agency is ALMP and sampling location is the appropriate location, meter model (example In-Situ Aqua TROLL 400), and the meter serial number is the number on the multimeter probe (Figure 18), not the Wireless TROLL Com serial number (Figure 17). The multiparameter probe meter serial number is located on the side of the probe (Figure 18) and should be the serial number written on either the hard copy calibration sheet or digital ArcGIS Survy123 form. Recording the probe serial number on the calibration sheet allows tracing and checking probes if calibrations/readings are in error.
- Tap "Calibrations" to view a menu of the calibrations and settings. Select the appropriate calibration menu items as needed (RDO Calibration, SC Calibration, etc.).

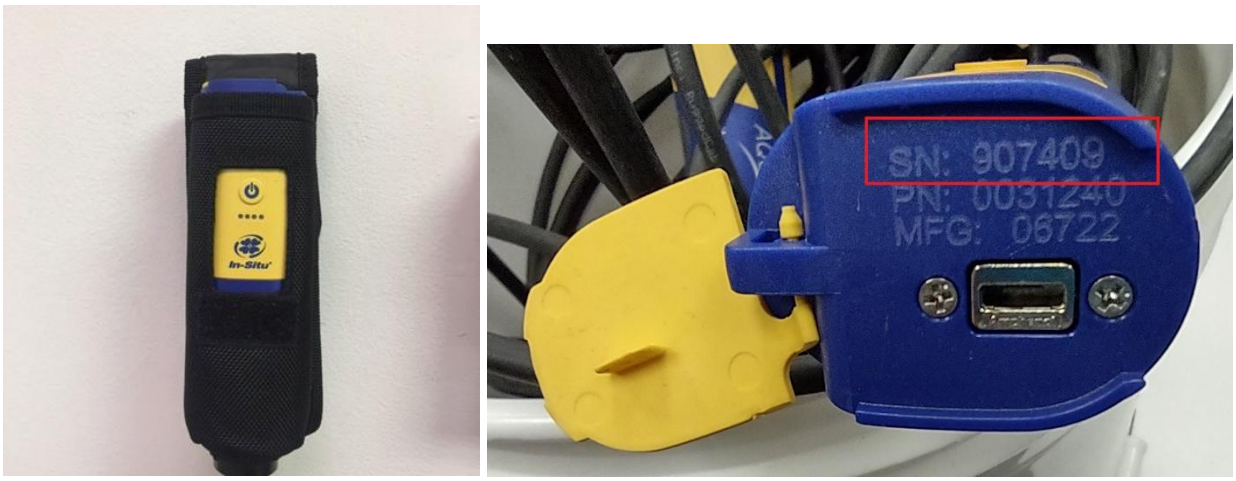


Figure 17. The Wireless TROLL Com Serial Number Location.

The Wireless TROLL Com serial number (SN) is the top number above the charging port underneath the yellow protective flap covering the charging port, boxed in red. The Wireless TROLL Com serial number (SN) is the top number and is hard to read. The Wireless TROLL Com serial number is the number that links to the tablet.

Figure 18. The In-Situ Field Meter Multiparameter Probe Serial Number Location.

The In-Situ field meter multiparameter probe serial number is located on the side of the probe, circled in red, and should be the serial number written on the field meter calibration sheet.

Dissolved Oxygen Calibration

Rinse the calibration cup with DI water. Then, fill the cup with DI water and screw on to probe. Gently invert the probe 3 times to rinse the probes with DI water. Remove and empty the cup.

Dry off probe with a Kimwipe. Either use the provided sponge soaked in water or add a small amount of tap water into the calibration cup. Thread the cup a couple of notches onto the probe leaving the cup loose enough for air exchange.

In the Vu-Situ app tap “Calibrations”. From the calibrations menu, select “RDO Saturation”. Then select “100% Saturation”. Let values stabilize. The screen will change colors (red to yellow to green) and will turn green and inform you when the values are stabilized. Verify the values are stabilized by the message as the colors are often hard to distinguish between each other.

“Accept” calibration. Calibration is complete, select “Next”. From either the calibration report or live readings screen, find the calibrated dissolved oxygen (D.O.) mg/L reading, the dissolved oxygen (D.O.) % saturation, temperature, and barometric pressure.

To get to the live readings screen from the calibration screen, tap the back arrow at the top of the screen. Select “Live Readings”. If using the live readings screen, water must still be in the cup.

The calibration report is the best number to use since it is the number saved as the calibration value at the instant the calibration is accepted. Historically, live readings have been used.

Using the temperature and barometric pressure, find the dissolved oxygen (D.O.) table value from the Solubility of Oxygen table (Figure 19). Record the solubility of oxygen table value (mg/L) based on temperature and barometric pressure.

USGS also has an online tool called DOTABLES to calculate solubility of oxygen based on temperature and barometric pressure (DOTABLES | U.S. Geological Survey (usgs.gov)).

Is the calibrated dissolved oxygen (D.O.) mg/L reading within ±0.5 mg/L of the table value reading? Circle Y/N for pre-sampling. If the answer is no, wait awhile; the temperature of the probe may need to equilibrate with the room temperature.

If the answer is still no, use another calibrated field meter or attempt to fix the probe. Contact the ALMP Coordinator or QA Coordinator for assistance if the meter continues to fail calibrations and do not use that meter during sampling.

Table 6.2–2. Solubility of oxygen in freshwater at various temperatures and pressures.—Continued

[Solubility shown in milligrams per liter; values are based on equations published by Benson and Krause (1980, 1984); Temp. (°C), temperature in degrees Celsius; solubility values for atmospheric pressures from 600 to 695 millimeters of mercury are after the values for 0-40 °C]

Temp. (deg C)	Atmospheric pressure, in millimeters of mercury																		
	700	705	710	715	720	725	730	735	740	745	750	755	760	765	770	775	780	785	790	795
15.0	9.27	9.34	9.41	9.48	9.54	9.61	9.68	9.75	9.81	9.88	9.95	10.02	10.08	10.15	10.22	10.29	10.35	10.42	10.49	10.56
15.5	9.18	9.24	9.31	9.38	9.44	9.51	9.58	9.64	9.71	9.78	9.84	9.91	9.98	10.04	10.11	10.18	10.24	10.31	10.38	10.44
16.0	9.08	9.14	9.21	9.28	9.34	9.41	9.47	9.54	9.61	9.67	9.74	9.80	9.87	9.94	10.00	10.07	10.13	10.20	10.27	10.33
16.5	8.98	9.05	9.11	9.18	9.24	9.31	9.37	9.44	9.50	9.57	9.64	9.70	9.77	9.83	9.90	9.96	10.03	10.09	10.16	10.22
17.0	8.89	8.95	9.02	9.08	9.15	9.21	9.28	9.34	9.41	9.47	9.54	9.60	9.66	9.73	9.79	9.86	9.92	9.99	10.05	10.12
17.5	8.80	8.86	8.92	8.99	9.05	9.12	9.18	9.24	9.31	9.37	9.44	9.50	9.57	9.63	9.69	9.76	9.82	9.89	9.95	10.01
18.0	8.70	8.77	8.83	8.90	8.96	9.02	9.09	9.15	9.21	9.28	9.34	9.40	9.47	9.53	9.59	9.66	9.72	9.78	9.85	9.91
18.5	8.62	8.68	8.74	8.80	8.87	8.93	8.99	9.06	9.12	9.18	9.24	9.31	9.37	9.43	9.50	9.56	9.62	9.69	9.75	9.81
19.0	8.53	8.59	8.65	8.72	8.78	8.84	8.90	8.96	9.03	9.09	9.15	9.21	9.28	9.34	9.40	9.46	9.53	9.59	9.65	9.71
19.5	8.44	8.50	8.57	8.63	8.69	8.75	8.81	8.87	8.94	9.00	9.06	9.12	9.18	9.25	9.31	9.37	9.43	9.49	9.55	9.62
20.0	8.36	8.42	8.48	8.54	8.60	8.66	8.73	8.79	8.85	8.91	8.97	9.03	9.09	9.15	9.21	9.28	9.34	9.40	9.46	9.52
20.5	8.28	8.34	8.40	8.46	8.52	8.58	8.64	8.70	8.76	8.82	8.88	8.94	9.00	9.06	9.12	9.18	9.25	9.31	9.37	9.43
21.0	8.19	8.25	8.31	8.37	8.43	8.49	8.55	8.61	8.67	8.73	8.79	8.85	8.92	8.98	9.04	9.10	9.16	9.22	9.28	9.34
21.5	8.11	8.17	8.23	8.29	8.35	8.41	8.47	8.53	8.59	8.65	8.71	8.77	8.83	8.89	8.95	9.01	9.07	9.13	9.19	9.25
22.0	8.04	8.09	8.15	8.21	8.27	8.33	8.39	8.45	8.51	8.57	8.63	8.68	8.74	8.80	8.86	8.92	8.98	9.04	9.10	9.16
22.5	7.96	8.02	8.08	8.13	8.19	8.25	8.31	8.37	8.43	8.48	8.54	8.60	8.66	8.72	8.78	8.84	8.89	8.95	9.01	9.07
23.0	7.88	7.94	8.00	8.06	8.11	8.17	8.23	8.29	8.35	8.40	8.46	8.52	8.58	8.64	8.69	8.75	8.81	8.87	8.93	8.98
23.5	7.81	7.86	7.92	7.98	8.04	8.09	8.15	8.21	8.27	8.33	8.38	8.44	8.50	8.56	8.61	8.67	8.73	8.79	8.84	8.90
24.0	7.73	7.79	7.85	7.90	7.96	8.02	8.08	8.13	8.19	8.25	8.30	8.36	8.42	8.48	8.53	8.59	8.65	8.70	8.76	8.82
24.5	7.66	7.72	7.77	7.83	7.89	7.94	8.00	8.06	8.11	8.17	8.23	8.28	8.34	8.40	8.45	8.51	8.57	8.62	8.68	8.74
25.0	7.59	7.65	7.70	7.76	7.81	7.87	7.93	7.98	8.04	8.10	8.15	8.21	8.26	8.32	8.38	8.43	8.49	8.54	8.60	8.66
25.5	7.52	7.58	7.63	7.69	7.74	7.80	7.85	7.91	7.97	8.02	8.08	8.13	8.19	8.24	8.30	8.35	8.41	8.47	8.52	8.58
26.0	7.45	7.51	7.56	7.62	7.67	7.73	7.78	7.84	7.89	7.95	8.00	8.06	8.11	8.17	8.22	8.28	8.33	8.39	8.44	8.50
26.5	7.38	7.44	7.49	7.55	7.60	7.66	7.71	7.77	7.82	7.88	7.93	7.99	8.04	8.10	8.15	8.20	8.26	8.31	8.37	8.42
27.0	7.32	7.37	7.43	7.48	7.53	7.59	7.64	7.70	7.75	7.81	7.86	7.91	7.97	8.02	8.08	8.13	8.19	8.24	8.29	8.35
27.5	7.25	7.30	7.36	7.41	7.47	7.52	7.57	7.63	7.68	7.74	7.79	7.84	7.90	7.95	8.01	8.06	8.11	8.17	8.22	8.27
28.0	7.19	7.24	7.29	7.35	7.40	7.45	7.51	7.56	7.61	7.67	7.72	7.77	7.83	7.88	7.93	7.99	8.04	8.10	8.15	8.20
28.5	7.12	7.18	7.23	7.28	7.33	7.39	7.44	7.49	7.55	7.60	7.65	7.71	7.76	7.81	7.87	7.92	7.97	8.02	8.08	8.13
29.0	7.06	7.11	7.16	7.22	7.27	7.32	7.38	7.43	7.48	7.53	7.59	7.64	7.69	7.74	7.80	7.85	7.90	7.95	8.01	8.06
29.5	7.00	7.05	7.10	7.15	7.21	7.26	7.31	7.36	7.42	7.47	7.52	7.57	7.62	7.68	7.73	7.78	7.83	7.89	7.94	7.99

Figure 19. An Example of a Solubility of Oxygen Chart at Various Temperatures and Pressures

In-Situ Calibration Report

In-Situ's VuSitu app produces a calibration report as the meter is calibrated (Figure 20). A calibration report can be produced after calibrating as well. For calibration values, the calibration report is the best place to get the calibrated values for the calibration sheet. It is the only place to get the actual pH 7.0 calibration value recorded during the 2-point calibration. The confirmation pH 7.0 check was a way to check the pH 7.0 calibration without using the calibration report.

The calibration report only reports pre-sampling calibrated values. Note all post-sampling values will be from the live readings screen like you are measuring a field sample (no calibration process).

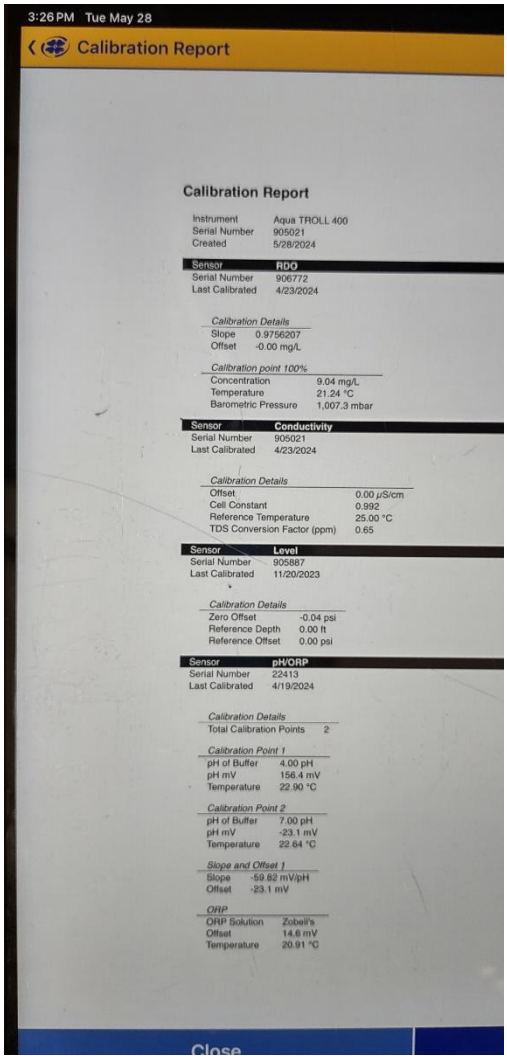


Figure 20. In-Situ Field Meter VuSitu Calibration Report.

<u>Specific Conductance (Spec Cond.) (S.C.) (µS/cm) Calibration.</u>

Zero Dry Air Check. Rinse the probe with DI water. Rinsing with tap water may lead to higher than zero readings. Dry probe with Kimwipe. In dry air without a cup attached, record the dry air specific conductance QC Zero check from the live reading screen. Is this reading ±2 of zero? Circle Y/N. If the answer is no, use another calibrated field meter or attempt to fix the probe.

Conductivity High Calibration. Next check or use professional judgement to estimate the highest specific conductivity (S.C.) that you may encounter in the field. The high and low conductivity standards should bracket the conductivity readings field personnel will encounter in the field. For the high conductivity value choose a standard with a value higher than the highest reading you may encounter in the field. Most freshwater sites would require a high standard of 1,000 µS/cm. Saltwater sites may use a standard of 10,000 µS/cm. The mid check specific conductivity standard is usually ½ of the high standard.

Record the lot number, expiration date, and standard value of the high S.C. standard (1000 µS/cm most likely).

Rinse the probe with deionized water then rinse with used standard/rinse high S.C. standard solution. Discard rinses with used standard/rinse solutions down the drain. Then fill cup with new high S.C. standard at least 2/3 full and screw cup onto probe body.

Move to calibration screen. Select “Conductivity”. Select “1-point Calibration”. Check the reference temperature listed on the standards box or bottle. Most conductivity standards are normalized to 25˚C. Some standards are normalized to 20˚C. You may need to change the reference temperature to either 20˚C or 25˚C depending on the standards used. ALMP does not alter specific conductivity readings based on temperature.

Allow probe to equalize. **In-Situ will try to autodetect the conductivity standard. In-Situ VuSitu app may have 1431 µS/cm as the default value, so you must go to the cog wheel and set the value to 1000 µS/cm or the appropriate standard value being used. Key in the correct conductivity standard value and tap “Set Custom”**. This step is very important since accepting a calibration of 1431 µS/cm when the standard is 1000 µS/cm will result in improper calibration.

On some of the screens both actual conductivity and specific conductivity will be listed. Always use specific conductivity.

Tap “Accept” and allow meter to calibrate. Calibrated high S.C. meter readings can either be viewed from the calibration report or the live readings screen.

For the live readings screen, close calibration report. At the top of the screen tap the back arrow (<) beside the In-Situ logo and go to the live readings screen. Record calibrated reading from the live readings screen.

Check to make sure the recorded high S.C. calibrated reading value is within ±10% of the standard selected (see ±10% ranges on the sheet). Is the high S.C. calibrated

reading within ±10% of the standard selected? Circle Y/N. If the answer is no, use another calibrated field meter or attempt to fix the probe.

After calibrated readings are measured/recorded, pour the new standard used to calibrate the probe into the used standard/rinse high S.C. solution bottle for further rinsings.

Conductivity Mid Check. Record lot number of the mid check standard, standard value, and expiration date of mid S.C. check standard. The mid S.C. check standard should be ½ of the high S.C. standard. Rinse probe with DI water then rinse with some of the used standard/rinse mid S.C. check standard solution. Discard all use standard/rinse solutions down the drain. Fill cup with new mid S.C. standard solution. Record mid S.C. check standard reading from live readings screen. This step does not require calibration; it is only a mid-check using the live readings screen. There is no calibration or calibration report. Is the mid S.C. check ±10% of the standard selected? Circle Y/N. If the answer is no, use another calibrated field meter or attempt to fix the probe.

If specific conductivity readings are not within 10% of the standard value, then the multiparameter probe should not be used until repaired. Use a different calibrated multiparameter probe for sampling and have the failed probe repaired.

Pour the newly used mid S.C. check solution into the used standard/rinse mid S.C. standard solution bottle to use for further rinses.

Specific Conductivity Standard Value on Box vs. Certificate of Analysis. The specific conductivity reported on the outside of the box is sometimes not the actual specific conductivity of the standard solution contained within the box. Each box of specific conductivity standard solution may have a different specific conductivity value contained within the box due to variations in manufacturing. The correct specific conductivity value is the value printed on the Certificate of Analysis sheet included with the box. It is advisable to check to see if the actual specific conductivity value on the sheet and the box match or change the value on the box to conform to the Certificate of Analysis value. Use the Certificate of Analysis standard value whenever possible.

<u>pH (S.U.) Calibration.</u>

pH 7.0 and 4.0 or 10.0 Two-Point Calibration. Record the lot number and expiration date of the 7.0 calibration buffer solution. Record the second calibration

standard buffer chosen, either 4.0 or 10.0. Circle pH 4.0 or 10.0. Record the second calibration standard buffer lot number and expiration date.

pH Selection of 4 or 10. Select either pH of 4.0 or 10.0 to do the second point calibration. Choose a standard closest to the pH of the sample site(s) in question or at least encompasses the greatest number of sites within the range of either pH 4.0 to 7.0 or pH 7.0 to 10.0. Typically, the pH 4.0 standard is best for streams and rivers while the pH 10.0 standard is best for lakes. Check the historical data from the sites or make best professional judgement to ensure you are including the second pH calibration point that is closest to the majority of your sites, whether it is between pH 1.0 to 7.0 (pH=4.0) or 7.0 to 14.0 (pH=10).

Rinse probe with DI water. Then rinse the probe with some of the used standard/rinse 7.0 pH buffer solution. Discard all rinses. Fill calibration cup with new pH 7.0 standard buffer.

Within the calibration menu select pH, then select 2-point calibration, then hit next. Once the calibration screens turn colors and it says the calibration is complete check that the mV reading is between -30 and 30 mV. If not, do not tap “Accept”. If the mV reading is between -30 and 30 mV, the screen is green, and it says calibration is complete, then tap “Accept”.

Do not tap “Next” until after you pour the pH 7.0 buffer into the pH 7.0 used standard/rinse solution bottle, rinse the probe with DI water, choose pH 4.0 or 10.0, rinse the probe with your second pH used standard/rinse solution (pH 4.0 or 10.0), fill the cup with new pH 4.0 or 10.0 standard solution, and lightly screw the cup back on. The second pH standard buffer should be in the cap ready to calibrate before tapping “Next”.

Once you tap “Next”, the meter immediately starts calibrating the second pH solution. Once the screen turns colors and the calibration is complete, tap “Accept”. Tap “Next”. From the calibration report you can find the first calibration reading around 7.0 and the second calibration reading around either 4.0 or 10.0. Using the calibration report is the only way to get the 7.0 calibrated reading since the first calibrated reading of pH 7.0 solution is not available until after the 2-point calibration is completed.

You can record the second calibrated standard reading either from the calibration report or the live readings screen. It is probably better to record the readings from the calibration report.

With the second calibration standard solution still in the cup, you can go to the live readings screen to get the second (4.0 or 10.0) calibrated meter reading if not

recorded from the calibration report. The first calibrated meter reading of pH 7.0 solution is not available at the live readings screen. With all data recorded, pour the second new standard solution into the used standard/rinse bottle and rinse the probe with DI water.

Are the pre-sampling first pH solution calibration pH meter readings (7.0) and the second calibration pH meter readings (4.0 or 10.0) within ±0.2 S.U. of the standard values? Circle Y/N for each calibration point. If no, the meter is not calibrated correctly and should not be used. If the answer is no, use another calibrated field meter or attempt to fix the probe.

Confirmation Buffer 7.0 check. Rinse probe with DI water. Then rinse the probe with used standard/rinse 7.0 pH buffer solution. Discard all rinses. Go to the live readings screen. Fill calibration cup with some new 7.0 standard pH buffer, wait for the readings to stabilize, and record pH value under confirmation buffer 7.0. Is the confirmation buffer 7.0 within ±0.2 S.U. of the 7.0 buffer standard? Circle Y/N. If no, use a different calibrated meter or repair. Pour pH 7 standard solution into the used standard/rinse bottle. Rinse the probe with DI water. Store the probe in the cup with tap water.

If the meter passes all pre-sampling QC checks, then the meter has been properly calibrated and is ready for field measurements and sampling.

Post-Sampling Checks.

To complete post-sampling checks, follow post-sampling check instructions on either the hard copy calibration sheet or the ArcGIS Survey123 form, except there will be no calibrating. Conduct rinses and use of new standards in the same manner as the pre-sampling process. Obtain your meter readings from the live readings screen.

All standards used in the pre-sampling calibration process will be used in the post-sampling checks to check for drift. For instance, if pH standards of 7.0 and 10.0 were used to calibrate the probe, then use pH 7.0 and 10.0 to conduct a post-sampling check. Use the same specific conductivity standards in the post-sampling check as was used in the pre-sampling calibration.

Make sure the probe has reached room temperature before beginning the post-sampling check. Often, it is helpful to put fresh tap water in the cup as soon as you return to the office after sampling, so the probe can reach room temperature while you put away equipment or do other tasks.

Failed Calibration Response Actions.

Failed Pre-Sampling Calibration Field Meter Response Action. Any field meters that fail pre-sampling calibration should not be used in the field. Have the meter fixed and/or use another meter that passes pre-sampling calibration according to calibration QA/QC criteria.

Failed Post-Sampling Check Field Meter Response Action. Anytime there are two consecutive field meter post-sampling check failures, field meters should not be used for sampling and fixed or replaced as soon as possible.

APPENDIX 2: Uncorrected DO Table and Correction Chart

Sea Level *(Uncorrected D.O. Values)*

Dissolved Oxygen (D.O.) TABLE

Corrected D.O. Table for a specific locatio or DWR office are available upon request from the ES

Altitude at Sea Level = 0 feet **Barometric Pressure (BP) at Sea Level = 760 mm Hg**

Temp (°C)	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9	Temp (°C)
0	14.6	14.6	14.5	14.5	14.5	14.4	14.4	14.3	14.3	14.3	0
1	14.2	14.2	14.1	14.1	14.1	14.0	14.0	13.9	13.9	13.9	1
2	13.8	13.8	13.8	13.7	13.7	13.6	13.6	13.6	13.5	13.5	2
3	13.5	13.4	13.4	13.4	13.3	13.3	13.2	13.2	13.2	13.1	3
4	13.1	13.1	13.0	13.0	13.0	12.9	12.9	12.9	12.8	12.8	4
5	12.8	12.7	12.7	12.7	12.6	12.6	12.6	12.5	12.5	12.5	5
6	12.4	12.4	12.4	12.4	12.3	12.3	12.3	12.2	12.2	12.2	6
7	12.1	12.1	12.1	12.0	12.0	12.0	12.0	11.9	11.9	11.9	7
8	11.8	11.8	11.8	11.8	11.7	11.7	11.7	11.6	11.6	11.6	8
9	11.6	11.5	11.5	11.5	11.4	11.4	11.4	11.4	11.3	11.3	9
10	11.3	11.3	11.2	11.2	11.2	11.2	11.1	11.1	11.1	11.1	10
11	11.0	11.0	11.0	11.0	10.9	10.9	10.9	10.9	10.8	10.8	11
12	10.8	10.8	10.7	10.7	10.7	10.7	10.6	10.6	10.6	10.6	12
13	10.5	10.5	10.5	10.5	10.4	10.4	10.4	10.4	10.4	10.3	13
14	10.3	10.3	10.3	10.2	10.2	10.2	10.2	10.1	10.1	10.1	14
15	10.1	10.1	10.0	10.0	10.0	10.0	10.0	9.9	9.9	9.9	15
16	9.9	9.8	9.8	9.8	9.8	9.8	9.7	9.7	9.7	9.7	16
17	9.7	9.6	9.6	9.6	9.6	9.6	9.5	9.5	9.5	9.5	17
18	9.5	9.4	9.4	9.4	9.4	9.4	9.4	9.3	9.3	9.3	18
19	9.3	9.3	9.2	9.2	9.2	9.2	9.2	9.1	9.1	9.1	19
20	9.1	9.1	9.1	9.0	9.0	9.0	9.0	8.97	8.9	8.9	20
21	8.9	8.9	8.9	8.9	8.8	8.8	8.8	8.8	8.8	8.8	21
22	8.7	8.7	8.7	8.7	8.7	8.7	8.6	8.6	8.6	8.6	22
23	8.6	8.6	8.5	8.5	8.5	8.5	8.5	8.5	8.4	8.4	23
24	8.4	8.4	8.4	8.4	8.4	8.3	8.3	8.3	8.3	8.3	24
25	8.3	8.2	8.2	8.2	8.2	8.2	8.2	8.2	8.1	8.1	25
26	8.1	8.1	8.1	8.1	8.1	8.0	8.0	8.0	8.0	8.0	26
27	8.0	8.0	7.9	7.9	7.9	7.9	7.9	7.9	7.9	7.8	27
28	7.8	7.8	7.8	7.8	7.8	7.8	7.7	7.7	7.7	7.7	28
29	7.7	7.7	7.7	7.7	7.6	7.6	7.6	7.6	7.6	7.6	29
30	7.6	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.4	30
31	7.4	7.4	7.4	7.4	7.4	7.4	7.4	7.3	7.3	7.3	31
32	7.3	7.3	7.3	7.3	7.3	7.2	7.2	7.2	7.2	7.2	32
33	7.2	7.2	7.2	7.1	7.1	7.1	7.1	7.1	7.1	7.1	33
34	7.1	7.1	7.0	7.0	7.0	7.0	7.0	7.0	7.0	7.0	34
35	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.9	6.8	35

* All D.O. values are in mg/L

D.O. Correction Chart

Altitude (ft)	Barometric Pressure (mmHg)	Correction Factor	Altitude (ft)	Barometric Pressure (mmHg)	Correction Factor
100	757	0.996	2400	695	0.914
200	755	0.993	2500	692	0.910
300	752	0.989	2600	689	0.907
400	749	0.985	2700	686	0.903
500	746	0.981	2800	684	0.900
600	743	0.978	2900	682	0.897
700	740	0.974	3000	679	0.893
800	737	0.970	3100	676	0.890
900	735	0.967	3200	673	0.886
1000	732	0.963	3300	671	0.883
1100	729	0.959	3400	669	0.880
1200	727	0.956	3500	666	0.876
1300	724	0.952	3600	663	0.873
1400	721	0.949	3700	661	0.870
1500	718	0.945	3800	658	0.866
1600	715	0.941	3900	656	0.863
1700	713	0.938	4000	654	0.860
1800	710	0.934	4100	651	0.857
1900	708	0.931	4200	648	0.853
2000	705	0.927	4300	646	0.850
2100	702	0.924	4400	644	0.847
2200	699	0.920	4500	641	0.844
2300	697	0.917			

How to Correct D.O. Table Values:

Corrected D.O. Value = Value from Sea Level Table x Correction Factor

1) Use the temperature displayed on your meter and the "Sea Level Table" (on the back of this page) to find the *Uncorrected D.O. Value*.

2) Use your location's altitude and the "D.O. Correction Chart" (on this page) to find the corresponding *Correction Factor*.

3) Multiply the *Uncorrected D.O. Value* (from step 1) by the *Correction Factor* (from step 2) to get the *Corrected D.O. Value*.

4) The value calculated in step 3 (*Corrected D.O. Value*) and the value displayed on the meter should be within $\pm$ 0.5 mg/L of each other.

Corrected D.O. Tables for a specific location orDWR office are available upon request from the ESS QA Coordinator.

D.O. Correction Factors

08/24/2007

APPENDIX 3: Filtering in the Field

Filtering Chlorophyll a in the field

Apparatus and Equipment

- Whatman™ GF/F filters - glass fiber, 47 mm, with nominal pore size of 0.7 µm. purchase or get from wet chem.
- Centrifuge tubes, 50 ml plastic, graduated - used to store filters in after filtration.
- Aluminum foil, to protect filtered samples and extracts from sunlight.
- Tweezers or flat-tipped forceps, to handle filters.
- Vacuum source, capable of maintaining a vacuum up to 6 inches' mercury (20kPa). Do not vacuum the filter to dryness, try to keep it moist.
- Vacuum Filtration apparatus, consisting of Millipore 47 mm hydrosol stainless-steel vacuum filter holder and vacuum manifold.
- Ice maintained > 6ºC - used to store and transfer samples. If you are filtering in a hotel room, keep samples in a freezer if possible; then transfer into a cooler with ice, by placing samples under the ice.
- 250 mL class A Graduated cylinders.
- Polyethylene squirts bottles.
- Deionized water.
- Disposal pH paper.

Sample Collection, Preservation, Shipment and Storage

- Due to the limitations of filtering samples in the field, it is the DWR Laboratory Section's policy to filter chlorophyll samples the day that the samples are Collected, not to exceed 48 hours from collection.
- Store samples in a freezer if possible until shipment.
- Shipment of the sample must be in a cooler filled with ice.
- The pH of each sample is checked on the day of collection prior to filtration in the field Samples from acidic water analyzed after filtration to prevent possible chlorophyll *a* degradation from residual acidic water on the filter. Samples on filters from water having pH 7 or higher may be frozen and analyzed within 21 days of collection.

- For filed collections and filtering please record the pH for all the samples.
- Acidic samples please notify the wet Chem lab and ship as soon as possible, if not samples results considered estimated with the assigned qualifier.
- All apparatus should be clean and acid free.
- No preservations required.
- Filtration should be performed in subdued light as soon as possible after sampling.

Sample filtration procedure:

1. Place numbered chlorophyll bottles in numeric order and check field sheets to make sure all samples are present.
2. Record lab numbers on the Chlorophyll *a* Benchsheet allowing for a duplicate after every tenth sample.
3. Assemble the filtration apparatus and attach the vacuum source.
4. Place a glass-fiber filter on the filter base, and attach top portion of funnel.
5. Prior to filtering samples, verify vacuum filtration does not exceed 6 in. Hg (20kPa) there is a vacuum control device and meter to control the filtration vacuum if possible.
6. Gently agitate the water sample to suspend particulates (i.e., stir or invert several times) and transfer 150 ml or an appropriately measured volume into a polyethylene graduated cylinder. Pour the subsample into the filter funnel. Lower volumes maybe use deepening on the turbidity and suspended matters in the sample.
7. Turn the vacuum source on and allow sample to filter. Do not suck the filter dry with the vacuum; instead slowly release the vacuum as the final volume approaches the level of the filter and completely release the vacuum as the last bit of water is pulled through the filter. Some samples may require more filtration time than others. Excessively long filtration times (i.e., >10 minutes) may damage cells and result in loss of chlorophyll. If filtration times exceed 10 minutes, filter smaller sample volumes. A sufficient volume has been filtered when a visible green or brown color is apparent on the filter. Please record if > 10 minutes or repeat with lower volume.
8. Using tweezers or forceps, remove the filter from the base, fold once with particulate matter inside and place in a labeled polyethylene centrifuge tube to be stored in the freezer until analysis.
9. Wrap the tubes containing the samples and duplicates in aluminum foil, label (by writing on masking tape) with the date, time, and chemist's initials when

arrived at the lab hand to wet Chem unit personals after dropping off collections from at receiving. Or store immediately in the Wet Chemistry freezer at –20°C or –70°C in the dark until extraction. If samples are delivered after hours, send an email to inform supervisor and lead chemist.

Chlorophyll *a* Bench sheet
EPA Method 445.0 Rev. 1.2-1997 modified option (In Vitro Determination of Chlorophyll a)
Instrument: Turner Designs Trilogy Laboratory Fluorometer Version 1.2 P/N 998-7210 S/N: 720000968

Lab #		**to**		**Steep information**		**Ambient Temperature**	
Analyst:				**Date**		**Start temperature °C**	
Analysis Date:				**Time In:**		**End temperature °C**	
Checked by:				**Date:**		**Analysis Times**	
Checked by Date				**Time Out:**		**Start Time**	
Page		**of**		**Total Time:**		**End time**	

Lab #	Date Filtered	Kpa <20	pH >6	Sample Vol. Filtered (mL)	Vol. Of 90% Acetone used (mL)	Sample Comments	Sequence Number
							1
							2
							3
							4
							5
							6
							7
							8
							9
							10
							11
							12
							13
							14
							15
							16
							17
							18
							19
							20

Samples with pH< 6.0 please note it in the comment section and confirm with pH meter then inform the analyst.
Samples must be filtered within 24 hours from the time of collection. If not, please note it in the comments section.

LRBs must be filtered at the beginning and the end of each filtration set (20 samples or less), duplicates every 10 samples. And so, on if more than 20 samples.
Filtration times must be < 10 minutes with vacuum < 20 Kpa which equal to < -5.0 PSI. if filtrations take longer use a smaller volume (100, 50, 25 mL).

STANDARD OPERATING PROCEDURES FOR FIELD FILTERING USING THE VACUUM PUMP PROCEDURE.

Field Procedure:

- Obtain filtering equipment including sterile 0.45 µm 47 mm diameter Millipore filters, glass fiber filters, nitrile gloves, forceps, and a supply of deionized (DI) water. An example of an appropriate filtering kit is Nalgene - filter holder with receiver 500 mL (Nalgene #300-4050).
- After donning gloves, thoroughly rinse the field filtering equipment with deionized water on the day of sampling at the first sampling station.
- Remove 0.45 um filter from package with clean forceps and place on the filter platform, grid side up.
- Inspect filter for proper placement-centered, no wrinkles, bends, cracks, holes, or gaps.
- Reassemble the apparatus.
- Attach hand pump to outlet of bottom chamber with tubing.
- For the first sample of the day, do field blank for quality control first using DI water
- Pour required volume of sample water in the top chamber (example-volume for orthophosphorus and dissolved phosphorus is at least 200 mL for each).
- Use hand pump to create vacuum.
- Continue adding sample and pumping until required filtered volume (based on parameter) is obtained and top chamber is empty. Note: It may be necessary to change filters several times or use a glass fiber pre-filter (see turbid samples options below) to obtain enough filtrate.
- Samples for all dissolved parameters can be filtered at once.
- Before disassembling, make sure that no sample remains in the top chamber and no pressure in the bottom chamber: remove tubing, or press release on pump.
- Disassemble apparatus.
- Decant filtrate into sample bottles, preserve and handle as per laboratory guidance.
- Remove filter with forceps and dispose of filter.
- Rinse filtering apparatus with DI water. This rinse must be repeated before field filtering at any additional locations (i.e. between stations).
- After last sample of the day is completed, do terminal field blank sample.

Turbid Samples Options:

When the filter becomes clogged:

Option 1: Change filters

- Finish filtering any sample left in top chamber.
- Ensure zero pressure in bottom chamber.
- Disassemble apparatus.
- Using forceps, remove clogged filter and replace with new filter. Caution: Don't let residue on filter contact any part of the interior of the apparatus or tips of forceps.
- Re-assemble apparatus and continue filtering.

Option 2: Pre-filter

The sample can be taken through a preliminary step using a filter with a larger pore size, such as a glass fiber filter.

This can be accomplished by placing the glass fiber filter on top of the 0.45 um filter on the filter platform. A small amount of DI water can be squirted on top of the combined filters to prevent vapor lock. It may be necessary to change these filters as they get clogged to obtain enough filtrate but this procedure should minimize the number of times the filters must be changed.

Quality Control Procedures

- The filtering apparatus and DI wash bottle should be regularly cleaned with phosphate-free detergent and completely rinsed with DI water, as is done with all other sampling equipment.
- Initial and terminal quality control samples (blanks) of filtered deionized water must be taken for each day's sampling for each parameter and submitted to the laboratory.
- Blanks must be filtered in the field: one at the beginning of the day before the first water sample is processed, and one at the end of the day after the last water sample is processed.
- Sources of contamination include:
    - air/environment
    - field staff
    - sampling equipment and bottle

    - filtration equipment (filter holder, filter, tubing)
    - DI water
    - chemical preservatives.
- Station location on the lab sheet should indicate QC sample type.
- Blanks should come back as non-detects.
- If blanks show detectable levels of analytes:
    - results from associated samples must be flagged, and flags reported to data users.
    - perform rigorous data review to see if contamination concerns are severe enough to warrant discarding the data.
    - patterns of dirty blanks should be reviewed and a plan for contamination source identification, corrective actions, and re-evaluations should be developed.

APPENDIX 4: Parameter Sample Container Guidance Table

Parameter	Sample Container	Sample Container	Label or Tag
1,4-Dioxane	40 mL glass vial with Teflon-lined cap (unpreserved). 4 vials needed.		Tag placed in bubble wrap bag or tag with rubber band around all vials.
1,4-Dioxane Trip Blank	40 mL glass vial with Teflon-lined cap (unpreserved). 3 vials needed.		Tag placed in bubble wrap bag or tag with rubber band around all vials.
Alkalinity	500 mL plastic disposable bottle.		Adhesive label on bottle.

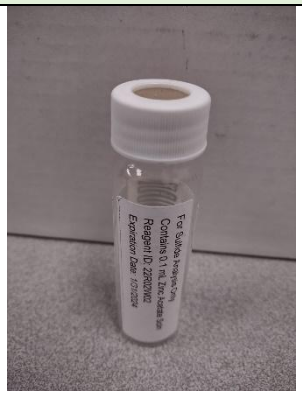
Parameter	Sample Container	Sample Container	Label or Tag
Chlorophyll-a	500 mL plastic brown wide mouth bottle, 17 cm x 7 cm bottle, reuseable.		Tag attached with rubber band. No adhesive label on bottle.
Color	500 mL plastic disposable bottle.		Adhesive label on bottle.
Cyanide	Plastic 250 ml, dark bottle, reuseable. **New** Bottle 09/2024 due to new Laboratory Cyanide Analysis machine.		Tag attached with rubber band. No adhesive label on bottle.

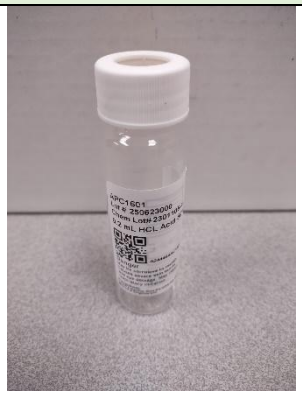
Parameter	Sample Container	Sample Container	Label or Tag
Fecal Coliform	250 mL Sterile plastic wide-mouth bottle pre-preserved with sodium thiosulfate preservative.		Tag attached with rubber band. No adhesive label on bottle.
Fluoride, Chloride, Bromide, Sulfate	500 mL plastic disposable bottle.		Adhesive label on bottle.
Hardness	500 mL plastic disposable bottle.		Adhesive label on bottle.

Parameter	Sample Container	Sample Container	Label or Tag
Mercury - Trace Level Mercury (HG1631) (Jar with the Star (*).	500 mL glass (borosilicate) with Teflon-lined cap. Cap marked with a star (*). WSS Chemistry Lab supplies this jar with Hg-free blank water.		WSS Chemistry Laboratory Supplied Sample Sheet.
Mercury Field Blank (FB) Jar without the Star (*).	500 mL glass (borosilicate) with Teflon-lined cap. Cap is not marked with a star (*).		WSS Chemistry Laboratory Supplied Sample Sheet.
Metals - Dissolved Metals Sample #1 (filtered)	500 mL plastic disposable bottle with M marked on it supplied by the WSS Chemistry Lab in a metals kit.		Adhesive label on bottle.

Parameter	Sample Container	Sample Container	Label or Tag
Metals - Dissolved Metals Sample #2 (filtered)	500 mL plastic disposable bottle with M marked on it supplied by the WSS Chemistry Lab in a metals kit.		Adhesive label on bottle.
Metals - Total Metals (Not filtered)	500 mL plastic disposable bottle with M marked on it supplied by the WSS Chemistry Lab in a metals kit.		Adhesive label on bottle.
Nutrients	500 mL plastic disposable bottle.		Adhesive label on bottle.
Oil and Grease	**(2)** 1 L glass wide-mouth jars with Teflon cap.		Adhesive label on jar.

Parameter	Sample Container	Sample Container	Label or Tag
Pesticides	4 L glass amber jug with Teflon-lined cap, one time use.		Adhesive label on jug.
Phytoplankton	500 mL plastic disposable bottle.		Adhesive label on bottle.
Residue, Non-Filterable -Total Suspended Solids	½ gallon plastic disposable bottle per site.		Adhesive label on bottle.
Semi-volatiles	4 L glass amber jug with Teflon-lined cap, one time use.		Adhesive label on jug.

Parameter	Sample Container	Sample Container	Label or Tag
Sulfides	40 mL glass vial with Teflon-lined cap (pre-preserved with 0.1 mL zinc acetate solution). <u>3 vials needed.</u> There are no sulfide trip blank vials.		Tag placed in bubble wrap bag or tag with rubber band around all vials.
Turbidity	500 mL plastic disposable bottle.		Adhesive label on bottle.
PFAS (Sample, Field Blank, and Trip Blank)	250 mL oblong plastic bottle Six bottles total: Three for Sample Two for Field Blank One for Trip Blank		Tag attached with rubber band. No adhesive label on bottle.

Parameter	Sample Container	Sample Container	Label or Tag
VOAs	40 mL glass vial with Teflon-lined cap (pre-preserved with HCl). <u>4 vials needed.</u>		Tag placed in bubble wrap bag or tag with rubber band around all vials.
VOA Trip Blank	40 mL glass vial with Teflon-lined cap (pre-preserved with HCl). <u>3 vials needed.</u>		Tag placed in bubble wrap bag or tag with rubber band around all vials.

APPENDIX 5: Standard Operating Procedures for Parameters if Chlorine is Present

Chlorine Testing.

From Section 4.3 *Situation-Based Preservation* in the DWR WSS Chemistry Laboratory Sample Submittal Guidelines. "*Chemical preservation can also be required if chlorine is detected in a sample. For these analytical parameters, chlorine interferes by binding with the target analyte and generally causing negative interference during analysis. When there is the possibility of chlorine being present in a collected sample (e.g., WWTP effluents) or if there is any doubt, the collected water sample must be checked in the field with a chlorine test strip. If chlorine is detected, then a specified chemical preservative is added to the sample bottle in sufficient amount to effectively de-chlorinate the sample (dechlorination procedures are included in the guidance tables).*"

Parameters where chlorine affects the laboratory analysis include 1,4-dioxane, cyanide, nutrients, pesticides, semi-volatiles, and VOAs. Chlorine test strips and preservatives can be ordered from the WSS Chemistry Laboratory.

Chlorine Test Strips.

There are three methods for testing with chlorine test strips. The first involves sampling the site water with a clean plastic disposable 500 mL bottle and use that water to test by pouring water over the test strip for 10 seconds. The second is to dip the test-strip directly into the water at the site (not the sample) and move back and forth for 30 seconds. The third is to use a second sample bottle to sample water just for pH or chlorine testing. With this method, the test strip can be dipped into the bottle for 10 seconds as it is not the actual sample. Dipping the test strip into the actual sample container is prohibited. It is generally recommended to use the second or third method.

Compare the test strip to the total chlorine color key on the test kit. If the sample was tested for chlorine, write on the field sheet that the site was tested for chlorine and the result of the test. If chlorine is detected, follow the standard operating procedure for dechlorination listed below and write on the field sheets that the samples were dechlorinated with the specific dechlorination additive required. There is a box on the field sheet to check whether samples are

chlorinated or dechlorinated in the field. Also contact the AMS Coordinator, WSS Chemistry Laboratory, and QA Coordinator if chlorine is found.

If the site has chlorine detected more than once or twice, AMS will address the situation and determine a course of action.

Dechlorination Reagents

Ascorbic Acid.

The recommended reagent for dechlorinating the cyanide sample is ascorbic acid. The Laboratory supplies ascorbic acid as a pack of 10 vials with 0.6 g ascorbic acid per vial.

Sodium Thiosulfate.

The recommended de-chlorination reagent for 1,4-dioxane, nutrients, pesticides, semi-volatiles, and VOAs is sodium thiosulfate (dissolve 3.5 grams in deionized water, then dilute to 1 liter). One mL of this sodium thiosulfate solution will remove 1 mg/L of residual chlorine in a 500 mL sample (prior to acid preservation).

The WSS Chemistry Laboratory SOP's state sodium thiosulfate ($Na_2S_2O_3$) (0.1 mL 10% $Na_2S_2O_3$/125 mL) should be added to dechlorinate these parameters. Chlorine test strips and sodium thiosulfate can be ordered from the WSS Water Chemistry Laboratory.

The Laboratory supplies sodium thiosulfate as 10% sodium thiosulfate in 30 mL eye dropper bottles. A standard eye dropper volume is said to be 1 mL or 20 drops.

Wear gloves and eye protection while handling preservatives. Consult the ascorbic acid and sodium thiosulfate SDS for further safety information.

Parameter Specific SOP in Presence of Chlorine

1,4-Dioxane with Chlorine present.

Site water for 1,4-dioxane should be tested for the presence of chlorine. Chlorine test strips provided by the WSS Chemistry Laboratory can be used to test the sample water for presence of chlorine. If chlorine is present, then the samples should be preserved with sodium thiosulfate ($Na_2S_2O_3$) (0.1 mL 10% $Na_2S_2O_3$/125 mL). A 40 mL vial would require 0.03 mL of sodium thiosulfate or smaller than standard drop from eye dropper.

Cyanide with Chlorine present.

Site water should be tested for the presence of residual chlorine with chlorine test strips before pH adjustment with sodium hydroxide.

If chlorine is detected, there are two options. First is to dechlorinate the sample with ascorbic acid. Second is to not collect the sample. A sample testing positive for chlorine will either be discarded or dechlorinated.

Holding time is 14 days if chlorine is not present but if chlorine is present and ascorbic acid is added to dechlorinate the sample, then holding time is only 24 hours. Most likely the sample dechlorinated with ascorbic acid will have a hold time qualifier attached to it.

Therefore, due to short hold times and quality of the sample with added acid, the WSS Chemistry Laboratory, AMS, and DWR Quality Assurance Work Group (QAWG) decided that if the sample can be skipped, then it is recommended that the sample be dropped if chlorine is present. Cross cyanide off the field sheet and write "Cyanide not sampled due to presence of chlorine."

<u>If the sample is mandatory, then dechlorinate with 0.6 grams of ascorbic acid (one vial of ascorbic acid contains 0.6 g ascorbic acid) per bottle. Ascorbic acid is supplied by the WSS Chemistry Laboratory as a pack of 10 vials. Each vial contains 0.6 grams of ascorbic acid. Cap and shake. Samples can be tested with chlorine strips again to make sure the sample is dechlorinated. Pour a little of the sample water into an extra cap (not sample bottle cap), dip the test strip in the cap with water and swirl test strip for 10 seconds, if samples are dechlorinated with ascorbic acid, note on the field sheet that cyanide samples were dechlorinated with ascorbic acid.</u>

<u>After performing the chlorine test and dechlorinating if needed, adjust pH with 6N sodium hydroxide to pH>10 but <11. The amount of 6N sodium hydroxide used is usually 3 droppers full of NaOH (not drops).</u>

<u>Note: These directions **underlined** were supplied by the WSS Chemistry Laboratory for a 1 L Cyanide Bottle. In September 2024 the Laboratory switched to a 250 ml dark bottle. Field personnel should contact the WSS Chemistry Laboratory for directions with a new bottle or follow the procedures and adjust the 6N sodium hydroxide needed to reach a pH>10 but <11.</u>

Chlorine test strips, ascorbic acid, and sodium hydroxide can be ordered from the WSS Chemistry Laboratory. Ascorbic acid is supplied by the Laboratory as a pack of 10 vials (0.6 grams per vial). Wear gloves and eye protection while using preservatives. Consult the ascorbic acid and sodium hydroxide SDS for further safety information.

Cyanide samples should also be tested for sulfides. Since the WSS Chemistry Laboratory tests for sulfides, the presence of sulfides will be determined at the laboratory level.

Nutrients with Chlorine present.

If chlorine is present for nutrient samples, then two nutrient sample bottles are needed.

One bottle of sample water without sodium thiosulfate is needed for nitrate+nitrite, total Kjeldahl nitrogen, and total phosphorous (non-dechlorinated bottle, first bottle). The non-dechlorinated bottle is preserved with sulfuric acid.

One sample bottle dechlorinated with sodium thiosulfate ($Na_2S_2O_3$) (0.1 mL 10% $Na_2S_2O_3$/125 mL) is needed for ammonia (dechlorinated bottle, second bottle). Approximately 0.4 mL of sodium thiosulfate ($Na_2S_2O_3$) (0.1 mL 10% $Na_2S_2O_3$/125 mL) per 500 mL plastic disposable bottle (10 drops or ½ full eye dropper). A standard eye dropper is said to hold 1 ml or 20 drops. The ammonia sample bottle should be dechlorinated with sodium thiosulfate before adding sulfuric acid. A second chlorine check may be needed before preserving with sulfuric acid.

Summary of Nutrient Instructions When Chlorine is Detected.

Parameter	Dechlorinated or Non-dechlorinated	Preservative
Nutrients (chlorine detected): *Nitrate+Nitrite (NO_3^- + NO_2^-)* *Total Kjeldahl Nitrogen (TKN)* *Total Phosphorous (TP)*	Non-dechlorinated Bottle.	Sulfuric acid (H_2SO_4) to pH<2. 
Nutrients (chlorine detected): *Ammonia (NH_3-N)*	Dechlorinated Bottle.	Sulfuric acid (H_2SO_4) to pH<2.  Add sodium thiosulfate ($Na_2S_2O_3$) (0.1mL 10% $Na_2S_2O_3$/125 mL) to de-chlorinate ammonia (NH_3-N) nutrient samples if chlorine is detected. Approximately 0.4 mL of sodium thiosulfate solution (1/2 of full eye dropper).

Pesticides with Chlorine Present.

Water from the sample site should be checked for the presence of chlorine with chlorine test strips. If chlorine is present, sodium thiosulfate ($Na_2S_2O_3$) (0.1mL 10% $Na_2S_2O_3$/125 mL) should be added to the sample. A 4 L pesticide amber glass jug would require approximately 3.2 mL of sodium thiosulfate or 3.2 full eye droppers full. Chlorine test strips and sodium thiosulfate can be ordered from the WSS Water Chemistry Laboratory.

Semi-volatiles with Chlorine Present.

Water from the sample site should be checked for the presence of chlorine with chlorine test strips. If chlorine is present, sodium thiosulfate ($Na_2S_2O_3$) (0.1mL 10% $Na_2S_2O_3$/125 mL) should be added to the sample. A 4 L Semi-volatile amber glass jug would require approximately 3.2 mL of sodium thiosulfate or 3.2 full eye droppers full. Chlorine test strips and sodium thiosulfate can be ordered from the WSS Water Chemistry Laboratory.

VOAs with Chlorine present.

Water from the sample site should be checked for the presence of chlorine with chlorine test strips. If chlorine is present, sodium thiosulfate ($Na_2S_2O_3$) (0.1mL 10% $Na_2S_2O_3$/125 mL) should be added to the sample. Chlorine test strips and sodium thiosulfate can be ordered from the WSS Water Chemistry Laboratory. A 40 mL vial would require 0.03 mL of sodium thiosulfate or smaller than standard drop from eye dropper.

APPENDIX 6: Total Metals and Dissolved Metals Sampling

Metals descriptions:

Cadmium (Cd) - In the elemental form, cadmium is insoluble in water. It occurs in nature largely as the sulfide salt, greenockite or cadmium blend, often as an impurity in zinc-lead ores. Cadmium is used in metallurgy to alloy with copper, lead, silver, aluminum, and nickel. It is also used in electroplating, ceramics, pigmentation, photography, and nuclear reactors. Cadmium salts are sometimes employed as insecticides and anthelminthics. Cadmium salts may be found in wastes from electroplating plants, pigment works, textile printing, lead mines, and chemical industries. Cadmium has been shown to be toxic to man when ingested or inhaled.

Chromium (Total Cr) - The principal chromium emissions into surface waters are from metal finishing processes such as electroplating, pickling, and bright dipping. Other smaller discharges of chromium are from the additive in circulating cooling waters, laundry chemicals and animal glue manufacture, leather tanning, and textile dyeing. Chromium is one of the least toxic of the trace elements. Chromium is not acutely toxic to humans.

Copper (Cu) - Copper salts in natural waters are generally the result of pollution attributable to the corrosive action of water on copper and brass tubing, to industrial effluents, and algaecide. Copper salts are used in textile processes, pigmentation, tanning, photography, engraving, electroplating, insecticides, and fungicides. Because copper in concentrations high enough to be dangerous to human beings renders water disagreeable to taste, it is believed that copper is probably not a hazard in domestic water supplies. However, copper in water may be disadvantageous or detrimental for certain industrial uses. In trace amounts, copper may be beneficial or even essential for the growth of living organisms. In excessive quantities it has been found toxic to a wide variety of aquatic forms, from bacteria to fish.

Nickel (Ni) - Nickel toxicity to man is believed to be very low. Systemic poisoning of human beings by nickel or nickel salts is almost unknown. Nickel does not merit serious consideration as a water pollutant, but nickel ions may be detrimental to beneficial uses. Nickel is toxic to some plants. Nickel is used in metal plating, batteries, as a catalyst in the preparation of edible oils, and in solar energy equipment.

Lead (Pb) - Lead is a cumulative poison. The poisoning usually results from the cumulative toxic effects of lead after continuous, long-term consumption rather than from occasional small doses. Lead exists in nature mainly as the sulfide (galena). Some natural waters contain lead in solution where mountain limestone and galena are found. Lead may also be introduced into water as a constituent of various industrial and mining effluents or as a result of the action of the water on lead in pipes. Atmospheric fallout and rainout of particulate lead are considered the most significant sources of lead input into natural surface waters, especially in urban areas. Storm runoff originating in urban areas will tend to be high in lead concentration. The low solubility of lead in the aqueous phase of natural systems and the formation of stable complexes with organic matter are manifested in the low uptake by some plants and animals. There are extremely low concentrations of lead in natural bodies of water in proportion to the concentration in the beds of lakes and streams. The net effect of these sluggish dynamics is a high degree of accumulation with prolonged exposure.

Zinc (Zn) - Zinc is used extensively for galvanizing, in alloys, for electrical purposes, in printing plates, for dye manufacture, and dyeing processes. Zinc salts are used in paint pigments, cosmetics, pharmaceutics, dyes, and insecticides. Zinc is found in high concentrations in natural waters in zinc mining areas and in effluents from metal plating works. In most surface and ground waters it is present only in trace amounts. There is some evidence that zinc ions are absorbed strongly and permanently on silt with a resultant inactivation of the zinc. Zinc has no known adverse physiological effects upon man except at very high concentrations. For esthetic considerations, high concentrations of zinc in domestic water are undesirable. At 30 mg/l, zinc gives water a milky appearance and causes a greasy film on boiling. It is toward fish and aquatic organisms that zinc exhibits its greatest toxicity at much lower concentrations.

Silver (Ag) - Silver metal is used in jewelry and silverware, in alloys, for electroplating, and in the processing of food and beverages. Silver nitrate is used in photography, ink manufacture, electroplating, coloring porcelain, and as an antiseptic. Traces of silver can be expected to reach natural waters from such sources. Silver is bactericidal and toxic at low concentrations.

Aluminum (Al) - Aluminum is the third most abundant element of the earth's crust. Aluminum occurs in many rocks and ores and clays, but never as a pure metal in nature. The metal itself is insoluble, but many of its salts are readily soluble. Aluminum is not likely to occur for long in surface waters because it precipitates and settles, or is absorbed as aluminum hydroxide or aluminum carbonate. In streams the presence of aluminum ions may result from industrial wastes or more likely from wash water containing alum from water treatment plants.

Beryllium (Be) - A relatively rare element, found chiefly in the mineral beryl, this substance is not likely to occur in natural waters. Although the chloride and nitrate forms are very soluble in water and the sulfate form moderately so, the carbonate and hydroxide forms are almost insoluble in cold water. Beryllium is used primarily in metallurgy to produce special alloys, in the manufacture of X-ray diffraction tubes and electrodes for neon signs, and in nuclear reactors. Beryllium is not harmful when taken internally through the digestive tract but has been incriminated in pulmonary ailments of workers exposed to beryllium dusts.

Calcium (Ca) - Calcium is the most abundant dissolved cationic constituent of natural fresh waters. This element is widely distributed in the minerals of rocks and soils. Calcium carbonate is frequently found as a cementing agent between mineral particles of sandstone and other detrital rocks. Calcium is one of the constituents of hard water and is a scale former in hot water systems. Prevention of corrosion of cast iron water distribution systems may be obtained through controlled precipitation of calcium carbonate. Lime ($CaOH_2$), and dolomite [$CaMg(CO_3)_2$] are frequently employed as neutralizing agents in water and wastewater treatment.

Cobalt (Co) - Cobalt and its salts are used for making alloys, in nuclear technology, as pigment in the china and glass industry, and as binders in the tungsten-carbide tool industry. Cobalt has a relatively low toxicity to man, and traces of cobalt are essential to nutrition.

Iron (Fe) - Iron interferes with laundering operations, imparts objectionable stains to porcelain fixtures, and causes difficulties in distribution systems by supporting growths of iron bacteria. Iron also imparts a taste to water, which is detectable at very low concentrations. In addition to corrosion products, natural waters may be polluted by iron-bearing ground water.

Lithium (Li) - An alkali metal, it is not widely distributed in nature, being found in a few minerals and in certain spring waters. Lithium is used in metallurgy, medicinal waters, some types of glass, and, as lithium hydroxide, in storage batteries. Lithium is toxic at high concentrations.

Magnesium (Mg) - Magnesium ions are of particular importance in that they occur in significant concentration in natural waters, and along with calcium, form the bulk of the hardness reaction. Magnesium is considered relatively non-toxic to man and not a public health hazard because, before toxic concentrations are reached in water, the taste becomes quite unpleasant. At high concentrations, magnesium salts have a laxative effect, particularly upon new users, although the human body can develop a tolerance to magnesium over a period of time.

Manganese (Mn) - Manganese is essential for the nutrition of both plants and animals. Manganese is undesirable in domestic water supplies because it causes an unpleasant taste, deposits on food during cooking, stains, and discolors laundry and plumbing fixtures, and fosters the growth of some microorganisms in reservoirs, filters, and distribution systems. Manganese frequently appears in surface waters as the result of decaying vegetation, in waters with acid pH values, and acidic waters from coal mine drainage. In ground water subject to reducing conditions, manganese can be leached from the soil and occur in high concentrations.

Sodium (Na) - Sodium salts are extremely soluble in water; any sodium that is leached from soil or discharged to streams by industrial wastes will remain in solution. Sodium is the cation of many salts used in industry and as such is one of the most common ions in process wastes. Sodium in drinking water may be harmful to persons suffering from cardiac, renal, and circulatory diseases.

Potassium (K) - One of the more common elements, potassium is one of the most active metals, and for that reason it is not found free in nature but only in the ionized or molecular form. Potassium is used for fertilizers and some varieties of glass. It is an essential nutritional element, but in excessive quantities it acts as a cathartic.

Barium (Ba) - Barium ions are not normally present in natural surface or ground waters in measurable concentrations although they have been detected in a few springs and in effluents from areas where barytes, BaSO4, or witherite, BaCO3, are mined. Barium and its salts are used in the metallurgical industry for special alloys, in the paint industry, in cements designed to withstand salt water, and in the ceramic and glass industries. Because of possible toxic effects on the heart, blood vessels and nerves a surface water supply standard of 1.0 mg/l was established.

Arsenic (As) - Arsenic may occur in water as a result of mineral dissolution, industrial discharges, or the application of insecticides. Arsenic is toxic to humans and accumulates in the body.

Selenium (Se) - Elemental selenium is practically nontoxic, but hydrogen selenide and other selenium compounds are extremely toxic and resemble arsenic in their physiological reactions. Selenium poisoning occurs mostly among livestock, and the toxic effects appear to be associated with the consumption of high concentrations of selenium in food, such as locoweed or grains grown in soils with high concentrations of selenium, rather than from water consumption. Selenium occurs in sulfur deposits, sulfides of metals, volcanic emissions, sedimentary rocks, organicrich soils, and coal. Selenium is used in the electronics industry, xerographic copying machines, photoelectric cells, glass and ceramics, pigment manufacture to color plastics, paints, enamels, inks, and rubber. It is

also used as a component of plating solutions. It can also be found in discharges from coal-fired power plants.

Metals and Dissolved Metals SOP

Total metals samples are collected by surface grab samples and dissolved metal samples are surface grab samples that require filtering. To support assessment of NC Surface Water Standards 2 separate filtered samples (dissolved metals samples = DISS1 and DISS2) are required. Also, one unfiltered sample (total metals sample) is required for hardness calculation (some standards are based on total metals and not total dissolved metals). Dissolved metal samples must be filtered and preserved within 15 minutes of collection. The 2 separate dissolved (filtered DISS1 and DISS2) samples should use two separate metals kits. The two dissolved samples (DISS1 and DISS2) should be collected and filtered at least 15 minutes apart from each other but within 1 hour of each other. The two dissolved samples (DISS1 and DISS2) are discrete samples (not duplicates or replicates) and are averaged for regulatory and assessment purposes as stated in 15A NCAC 02B .0211 FRESH SURFACE WATER QUALITY STANDARDS FOR CLASS C WATERS. *"11 (f) Compliance with acute instream metals standards shall only be evaluated using an average of two or more samples collected within one hour."*

For regulatory and assessment purposes, dissolved metals is calculated based on averaging two discrete samples (DISS1 and DISS2) taken within 1 hour. These samples are not duplicates. A duplicate metals sample is taking two samples instantaneously (not 15 minutes apart).

Improper sampling techniques and non-adherence to time limits cause those dissolved metals and total metals samples to be qualified and not used for assessment purposes. Factors for metals data exclusion from DWR's assessment process include missing times, same time for both dissolved samples (DISS1 and DISS2), time between DISS1 and DISS2 <15 min or > 1hr, and not having 2 dissolved metals samples and 1 total metals sample during the same site visit.

WSS Chemistry Laboratory Supplied Metals Kits.

Field personnel should request 2 metals kits per site (one kit for DISS1 and one kit for DISS2) from the WSS Chemistry Laboratory using the

Lab_Sample_Supply_Request form from Supplies, Fees & Assistance | NC DEQ website and contacting the Inorganic Chemistry Branch, Metals Unit Supervisor joseph.jurgevitch@deq.nc.gov.

Metals kits must be requested by writing metals kits and the number of kits needed on the bottom of the Lab_Sample_Supply_Request form. Each metals kit contains a 1 L plastic reusable sample collection bottle filled with deionized water, a 4-foot length of peristaltic tubing, and two 500 mL cleaned plastic disposable bottles marked with M to signify that the bottles were cleaned for metals according to protocols. Normal dissolved metals and total metals sampling involves two metals kits. You will only use three of the four 500 mL disposable plastic bottles supplied in the two kits. Return the used two 1 L deionized water/sample bottles from the 2 kits, one un-used 500 mL plastic disposable sample bottle, and two used tubing sections from the 2 kits to the WSS Chemistry Laboratory to be cleaned and reused.

Metals Filters.

Field personnel should contact the AMS Coordinator for a supply of Millipore Disposable Groundwater Filter Capsules. At least two Millipore Disposable Groundwater Filter Capsules 0.45 micron (0.45 µm) pore size are required per site. The filters should have been previously tested for metals by the manufacturer and the Ecosystems Branch with the WSS Chemistry Laboratory. Therefore, the filters used for AMS and RAMS Program sampling must be supplied by the AMS Coordinator.

Filters are single use, used for only one sample (except when used for the equipment blank and DISS1). Discard after one use. Use a new filter and a new kit for each sample.

Protocol and Suggestions for Minimizing Metal Contamination.

Method 1669. Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, July 1996 lists many possible sources of contamination for trace level metal sampling including metal bridges, dirt and dust from automobile exhaust, and sampling equipment.

- If possible, turn the vehicle off when sampling and filtering for metals. Avoid filling the samples near a running motor or any type of exhaust system because discharged fumes and vapors may contaminate the samples.
- Some vehicles must be turned on to operate the 12V peristaltic pump to filter samples. If this is the case, keep the vehicle running and do your best to filter away from possible contaminants by filtering inside the vehicle and/or away from the exhaust pipe. Field personnel should get a rechargeable battery for use with the 12V peristaltic pump from the AMS Coordinator. Using a rechargeable portable battery allows use of the pump without having the vehicle running.
- Use caution when handling sample equipment to avoid placing equipment on dirty surfaces or ground.
- Though metals is not a trace level analysis, it is advisable to sample upstream of bridges, turn the car/truck off during sampling/filtration, and use only clean and/or laboratory provided equipment (filter tubing, etc.).
- Field personnel should consult with the QA Coordinator, AMS Coordinator, and Ecosystems Branch Supervisor if a metal bridge sampler is needed to sample for metals. Only supplies provided by the WSS Chemistry Laboratory and AMS and RAMS Program should be used to sample for metals.

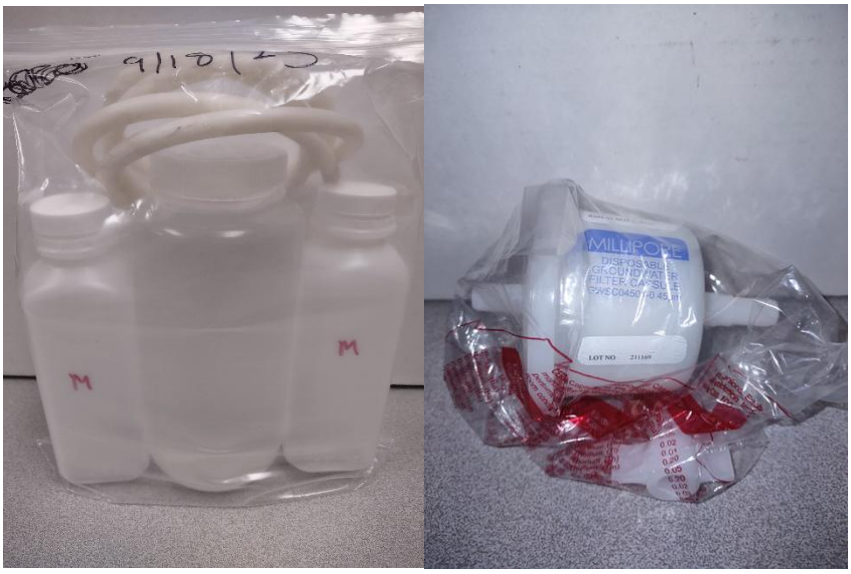


Figure 21. Metals Kit from the WSS Chemistry Laboratory and Millipore Disposable Groundwater Filter Capsule

DISSOLVED Metals Filtration.

Obtain and arrange all filtering equipment including peristaltic pump, metals sampling kit (cleaned peristaltic pump tubing, 1 L collection bottle with deionized water, disposable plastic bottles), capsule filters, and nitrile gloves. Ensure that the workspace is clean. Wear clean gloves before handling any equipment that will come into contact with the sample(s). Change gloves as needed to minimize contamination risk throughout the process.

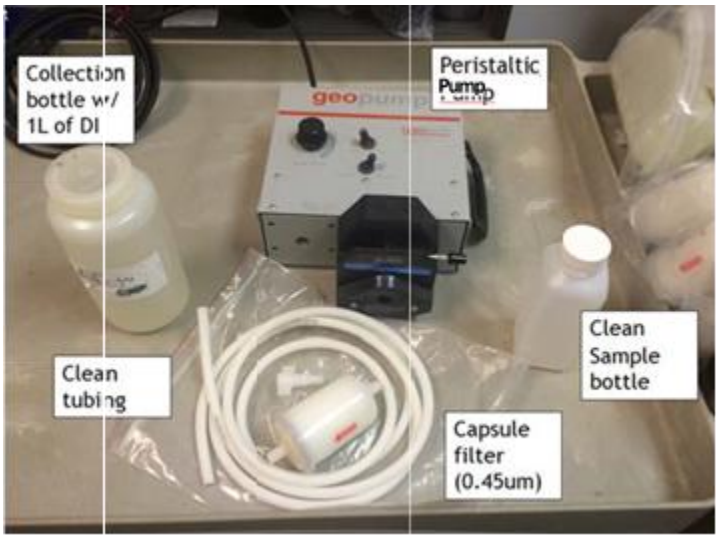


Figure 22. Dissolved Metals Filter Equipment.

Arrange workspace such that one end of the pump tubing is attached to a capsule filter, and the other is free to go into the deionized water contained in the 1 L sample collection bottle. Capsule filters have an arrow indicating the direction of the water flow. Screw the filter nozzle on the end of the filter that the DI water will flow into and then attach one end of the pump tubing to this nozzle. The arrow on the side of the filter should be pointing away from the nozzle and attached tubing.

Extra care must be taken so that the ends of the pump tubing are always kept clean. The tubing ends will have the most direct contact with the sample and therefore most easily contaminated. The free end of the tubing and filter end can be placed in a large clean Ziploc bag when not in use.

Conditioning a filter is pumping deionized water through the filter before sampling thereby wetting the filter media and rinsing the filter body of any loose debris or impurities. This is not a filter blank or an equipment blank. The deionized water is never analyzed. The deionized water is used to “condition” the filter.

All samples (environmental and QC samples) (e.g., Equipment Blank Dissolved (EBD)) must be collected after this conditioning step has been completed.

Load the pump with the tubing making sure the tubing is between the v clamps (Figure 23). Lower the clamps with the tightening lever. To condition the filter, insert the loose end of tubing into the container of deionized water supplied by the WSS Chemistry Laboratory. Some peristaltic pumps have a directional flow switch. Note the direction of the pump dial so the pump removes water from the bottle pumping it through the filter.

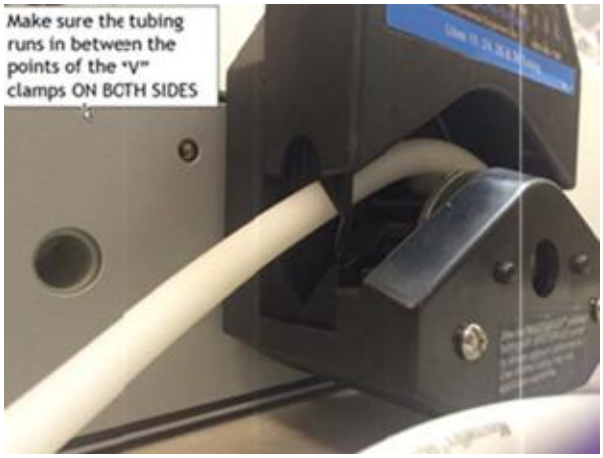


Figure 23. Loading the Tubing in the Peristaltic Pump.

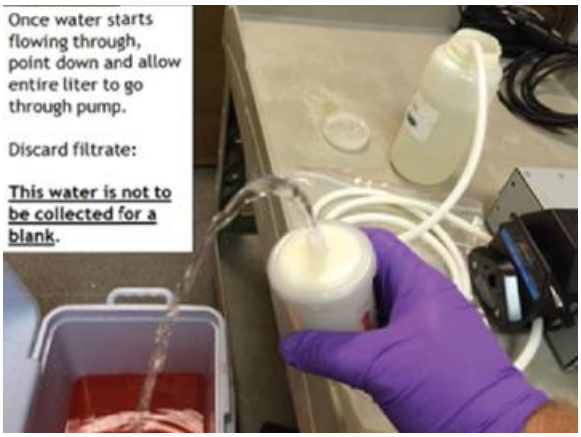


Figure 24. Conditioning the Filter.

Point the filter up in the air. Once the filter is full of water and water flows out of the filter, point the filter outlet down and allow filtrate to go to waste. Pump 1 L of deionized water through the filter. After filtering the 1 L of deionized water, shake the filter removing any excess deionized water from the filter. The filter is now conditioned.

Figure 25. Conditioning the Filter and Shaking Excess DI Water Out.

Keep the filter and loose tubing end from touching any other surfaces. The loose tubing end and filter can be placed in a clean Ziploc gallon bag to keep them protected from contamination.

The 1 L sample collection plastic bottle that contained the deionized blank water is now the sampling bottle. Rinse the sample bottle 3 times at the site to remove any distilled water remaining in the bottle. Collect a surface grab sample from the site. Cap the bottle underwater if possible.

Filter the surface grab sample by filtering some of the sample water through the filter onto the ground before collecting the filtrate in one of the 500 mL plastic disposable bottle marked with M. Metals plastic disposable bottles (M) supplied by the WSS Chemistry Laboratory in the metals kits do not need field rinsing. Preserve dissolved metals samples with one vial of nitric acid. This first dissolved metals sample is DISS1. Filter and preserve the first dissolved metals (DISS1) sample within 15 minutes of collection.

Repeat this procedure with the second kit, new filter, and new tubing after at least 15 minutes have elapsed since collecting the first dissolved metals sample (DISS1). The second dissolved metals sample (DISS2) should be collected 15 minutes after the first dissolved metals sample (DISS1) and within 1 hour of the first sample

(DISS1). Note the time collected for both dissolved metals samples on the dissolved metals DISS1 and DISS2 field sheets.

Write the lot numbers of the nitric acid vials used on the DISS1 and DISS2 field sheets in the Lots/Comments section.

Write the lot numbers of the Millipore Disposable Groundwater Filter Capsule 0.45 µm on the DISS1 and DISS2 field sheets in the Lots/Comments section.

If lot numbers of the nitric acid vials or Millipore Disposable Groundwater Filter Capsules 0.45 µm are different for the 2 dissolved metals samples, be sure to write the correct lot number on the appropriate dissolved metals field sheet.

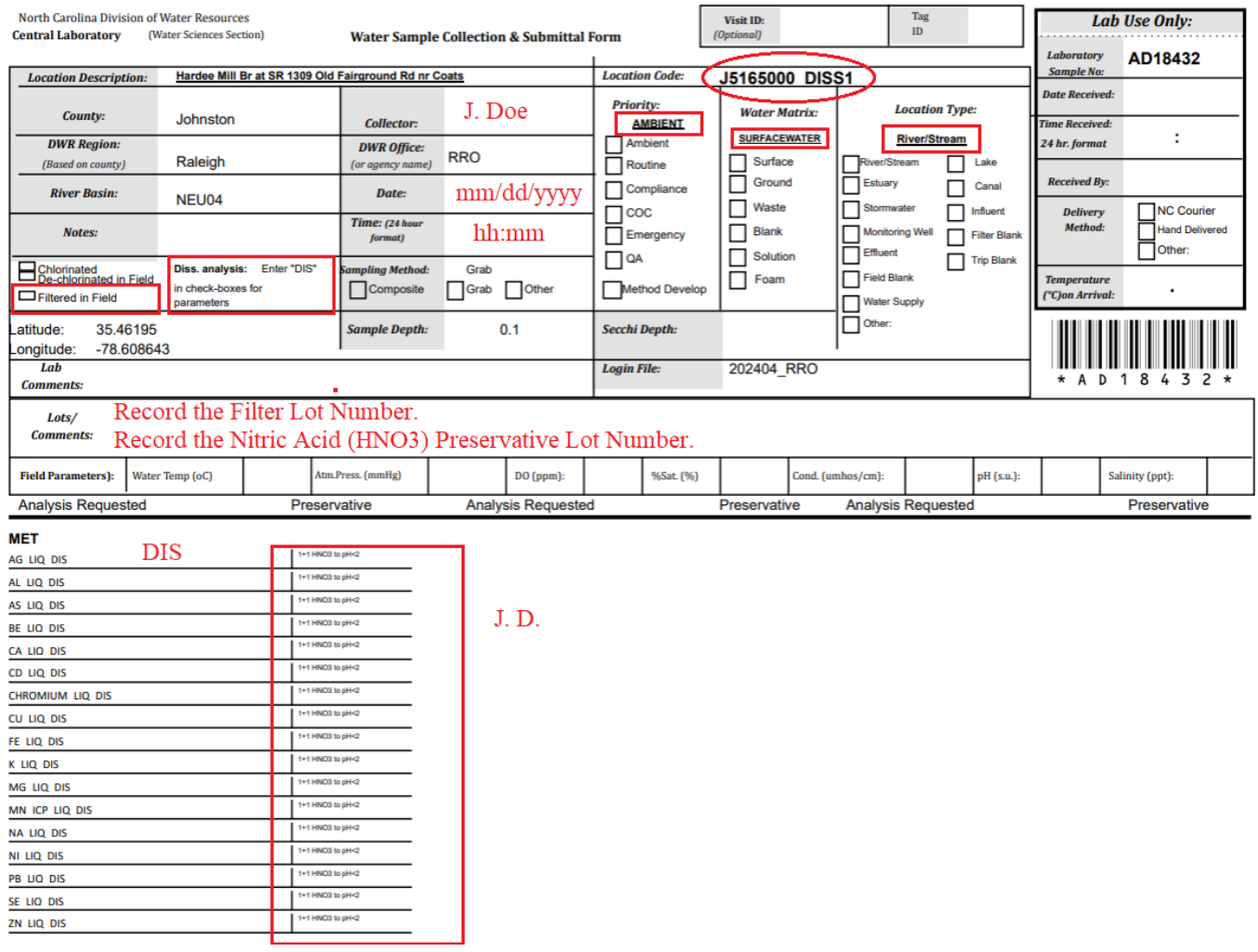


Figure 26. Dissolved Metals (DISS1) Field sheet Listing the Entries and Sections to be Filled Out.

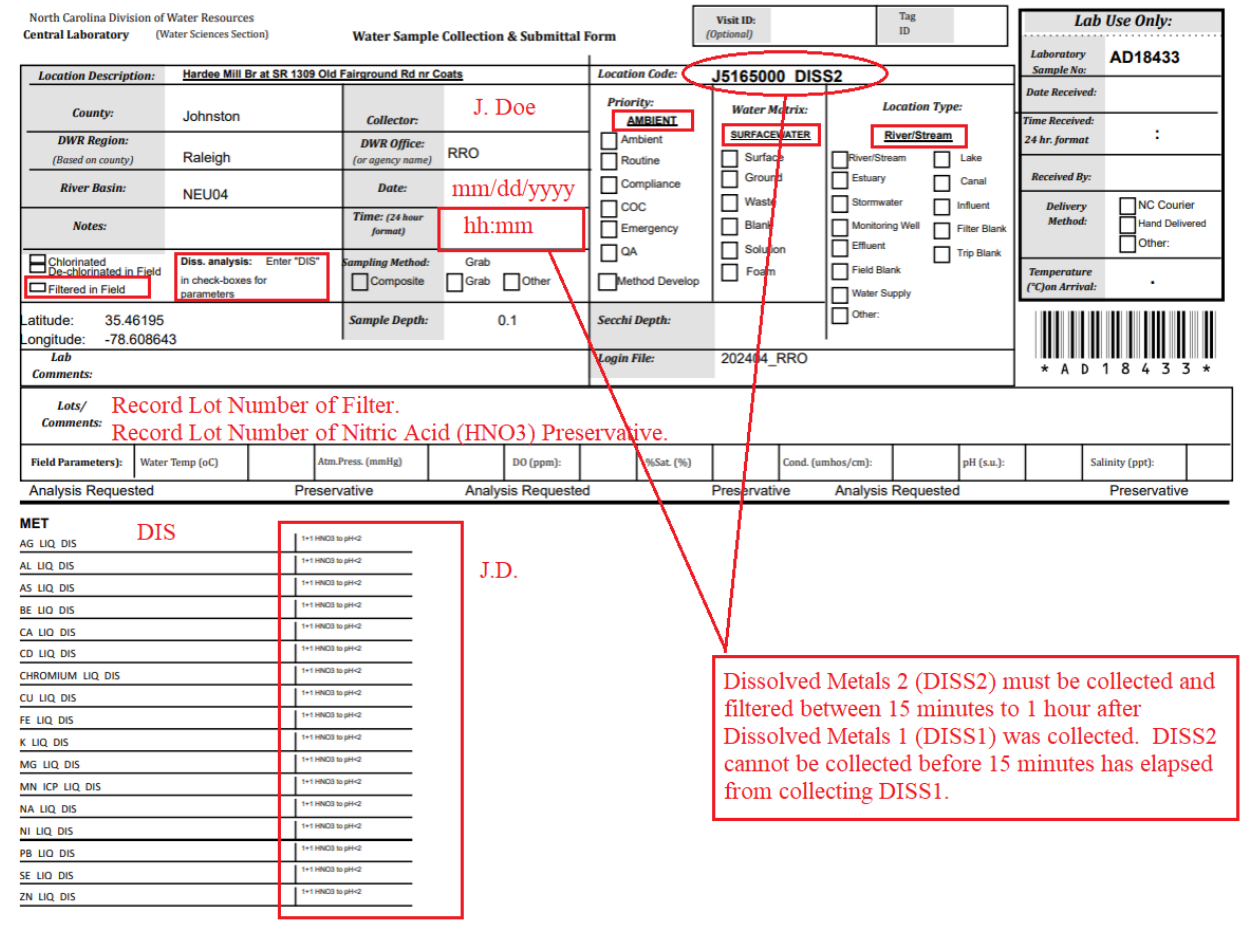


Figure 27. Dissolved Metals Two (DISS2) Field sheet Listing the Entries and Sections to be Filled Out.

TOTAL Metals (not filtered).

The total metals sample is a direct surface grab sample from the site using one of the 500 mL plastic disposable bottles marked with M. The plastic disposable bottles marked with a M do not need field rinsing.

The total metals sample (not filtered) is on another field sheet that may list several other unfiltered grab sample parameters. The total metals sample should be collected close to the dissolved samples but at the same time as the other grab samples. The total metals surface grab and the other grab samples are collected at the same time so the times of collection for all the grab samples listed on the

"grab/unfiltered" field sheet and tags are the same. Write the lot number of the nitric acid preservative on the total metals "grab/unfiltered" field sheet.

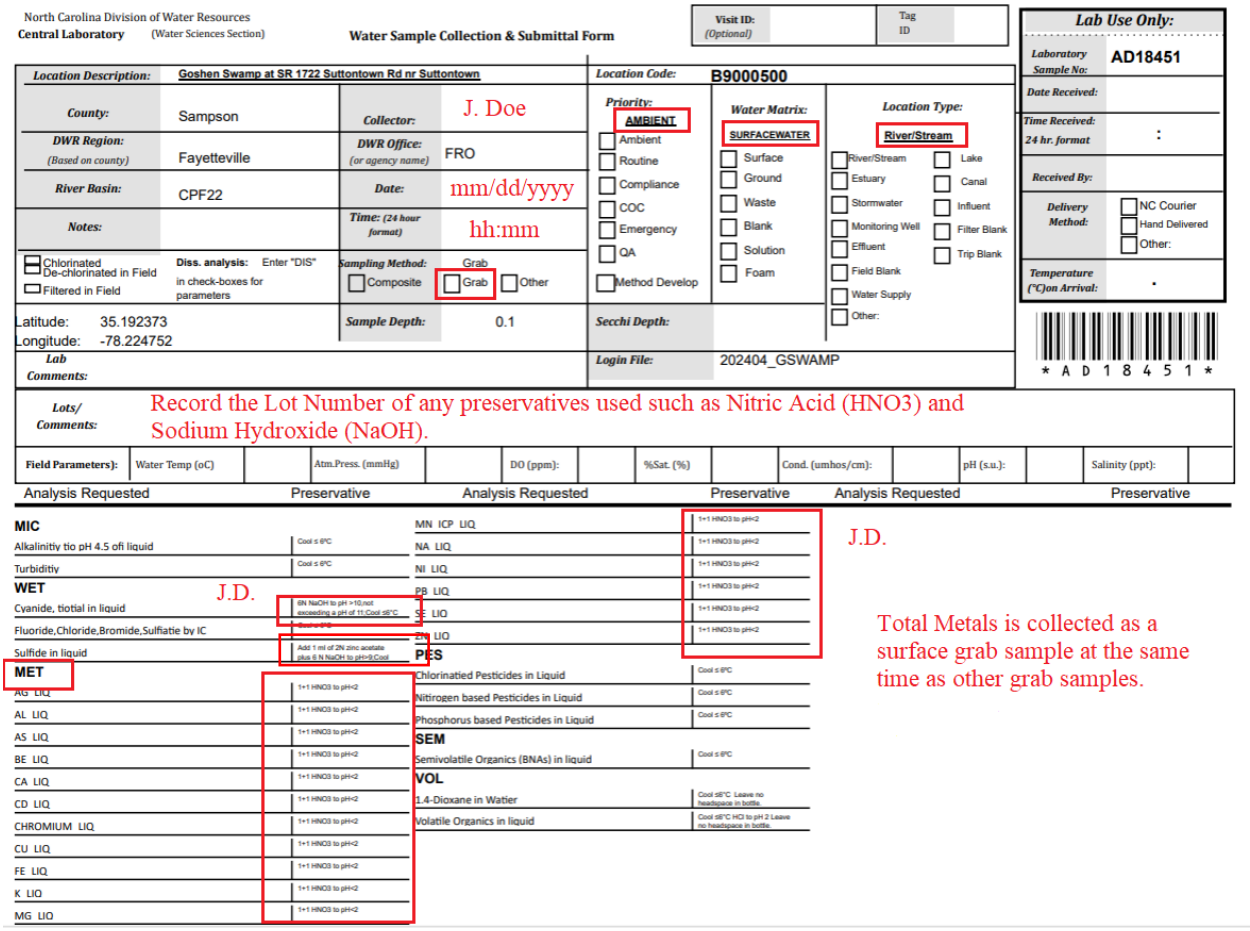


Figure 28. Total Metals Field sheet Includes Total Metals Grab Sample with Other Grab Samples.

Metals Preservatives.

Both total metals and dissolved metals samples are preserved with nitric acid vials. Nitric acid preservatives are supplied in vials (Thermo Fisher Scientific Nitric Acid 20-70% EP vials with 5.0 mL 1:1 Nitric Acid). Nitric acid preservative vials should be ordered from the WSS Chemistry Laboratory.

Equipment Blank Dissolved (EBD).

An equipment blank dissolved (EBD) for dissolved metals is collected once per year per site in conjunction with duplicates. An equipment blank is collected by filtering

a second bottle of 1 L conditioning deionized water into a 500 mL plastic disposable bottle with the letter M before site samples are filtered. If your site is scheduled for an equipment blank, field personnel should request an extra metals kit from the WSS Chemistry Laboratory for the equipment blank. Requests can be submitted to the Metals Unit supervisor, Joe Jurgevich, at joseph.jurgevich@deq.nc.gov.

Equipment blanks for the dissolved metals are completed in the field and are designed to detect contamination from filtering equipment (filters, tubing, etc.). Equipment blanks will be scheduled following the RAMS duplicate schedule. When a duplicate metals sample is scheduled at a given site, an equipment blank will also be scheduled for that site.

The location code for the equipment blank will take the format of "locationcode_EBD". For example, the equipment blank at N8990000 would be N8990000_EBD (**E**quipment **B**lank **D**issolved=EBD). An equipment blank field sheet and possibly a label will be provided by the AMS coordinator.

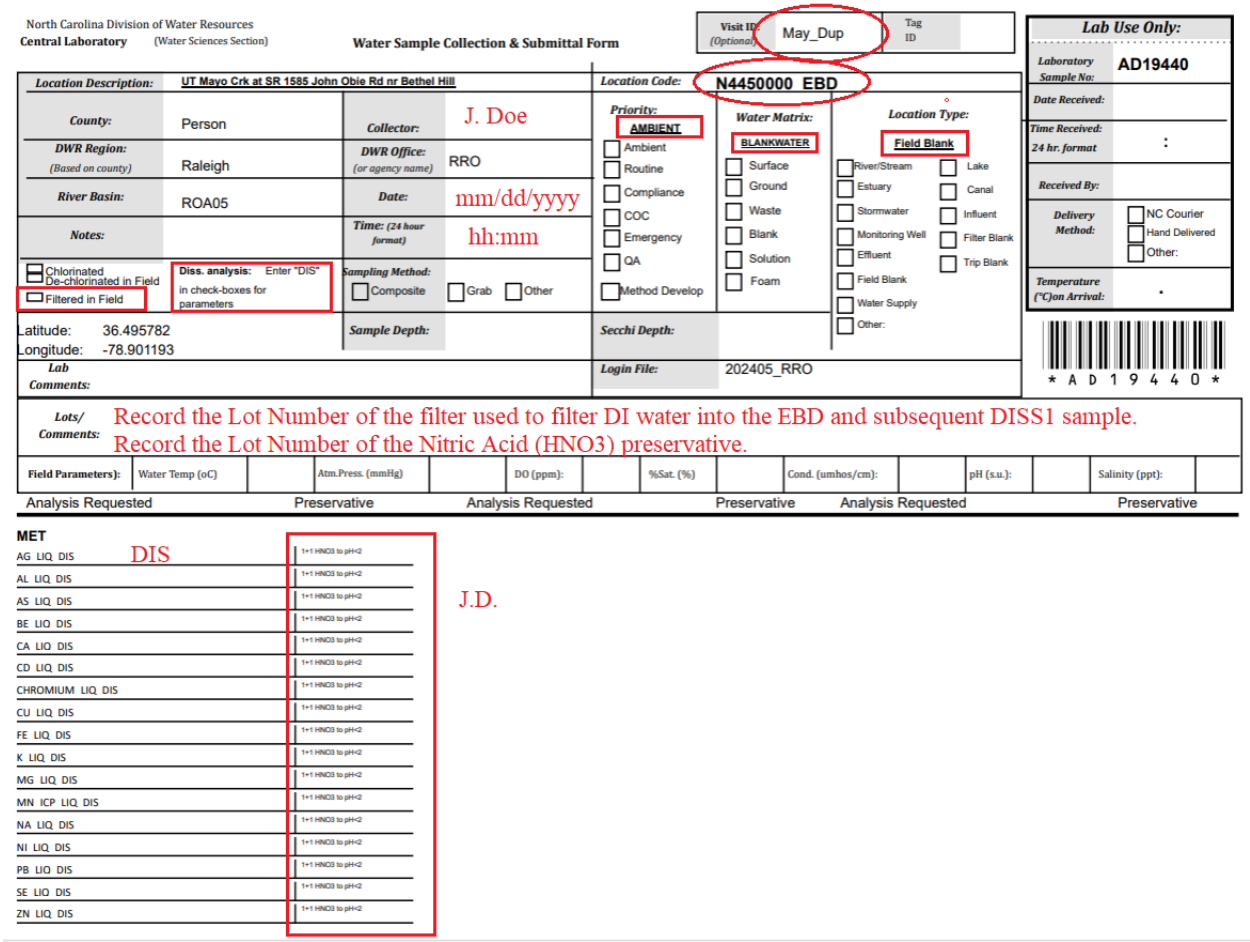

North Carolina Division of Water Resources
Central Laboratory (Water Sciences Section)
Water Sample Collection & Submittal Form
Visit ID (Optional): May_Dup
Tag ID
Lab Use Only:
Laboratory Sample No: AD19440
Date Received:
Time Received: 24 hr. format :
Received By:
Delivery Method: NC Courier, Hand Delivered, Other:
Temperature (°C)on Arrival: .
Location Description: UT Mayo Crk at SR 1585 John Obie Rd nr Bethel Hill
Location Code: N4450000 EBD
County: Person
Collector: J. Doe
DWR Region: (Based on county) Raleigh
DWR Office: (or agency name) RRO
River Basin: ROA05
Date: mm/dd/yyyy
Notes:
Time: (24 hour format) hh:mm
Chlorinated / De-chlorinated in Field
Filtered in Field
Diss. analysis: Enter "DIS" in check-boxes for parameters
Sampling Method: Composite Grab Other
Latitude: 36.495782
Longitude: -78.901193
Sample Depth:
Secchi Depth:
Lab Comments:
Login File: 202405_RRO
Priority: AMBIENT; Ambient, Routine, Compliance, COC, Emergency, QA, Method Develop
Water Matrix: BLANKWATER; Surface, Ground, Waste, Blank, Solution, Foam
Location Type: Field Blank; River/Stream, Estuary, Stormwater, Monitoring Well, Effluent, Field Blank, Water Supply, Other: Lake, Canal, Influent, Filter Blank, Trip Blank
* A D 1 9 4 4 0 *
Lots/ Comments: Record the Lot Number of the filter used to filter DI water into the EBD and subsequent DISS1 sample.
Record the Lot Number of the Nitric Acid (HNO3) preservative.
Field Parameters): Water Temp (oC) | Atm.Press. (mmHg) | DO (ppm): | %Sat. (%) | Cond. (umhos/cm): | pH (s.u.): | Salinity (ppt):
Analysis Requested | Preservative | Analysis Requested | Preservative | Analysis Requested | Preservative
MET
AG LIQ DIS DIS
AL LIQ DIS
AS LIQ DIS
BE LIQ DIS
CA LIQ DIS
CD LIQ DIS
CHROMIUM LIQ DIS
CU LIQ DIS
FE LIQ DIS
K LIQ DIS
MG LIQ DIS
MN ICP LIQ DIS
NA LIQ DIS
NI LIQ DIS
PB LIQ DIS
SE LIQ DIS
ZN LIQ DIS
1+1 HNO3 to pH<2
J.D.

Figure 29. Metals Equipment Blank Dissolved (EBD) Field sheet.

Collecting the Equipment Blank (EBD).

When on site, set up your tubing and peristaltic pump as normal. Prime your filter using the first 1 L bottle of deionized water from the metals kit as instructed in the metals SOP. Hold the filter upright until the filter fills completely and DI water is flowing out of the top. Allow the full 1 L to flow through the filter. Shake the filter dry. Your filter is now primed.

Next, follow the steps below to collect the equipment blank:

- Place the tubing in the second 1 L bottle of deionized water.
- Draw water from the second 1 L bottle through the tubing and filter. Again, hold the filter upright until the water flows out of the top.
- Fill the 500 mL plastic disposable bottle marked with a M with the filtered deionized water. This is your equipment blank sample (EBD). The plastic disposable bottle marked with a M does not need field rinsing.

- Preserve the filtered equipment blank (EBD) with nitric acid within fifteen minutes of collection and place on ice.

Prepare for collecting DISS1 after collecting the EBD.

- Shake the filter dry again. Place the tubing and filter in a plastic bag to keep clean.
- This is the only time the filter and tubing can be used twice. The filter and tubing can be reused to filter the first dissolved metals sample (DISS1) at the site.
- Continue to sample DISS1 and DISS2 according to regular dissolved metals protocol.

Total Metal Duplicates.

Total metals (not filtered) duplicates are collected once per year with the other site duplicates. Dissolved metals (filtered) are not sampled as duplicates, only total metals have duplicates.

A duplicate total metals grab sample is collected simultaneously with the total metals grab environmental sample. Duplicates and environmental samples must have the same sampling time for both samples.

Metal parameters sampled during a metals duplicate month include:

- Total metals sample.
- Total metals sample duplicate.
- EBD. Equipment blank dissolved sample.
- DISS1. Normal dissolved metals 1 sample (no duplicate).
- DISS2. Normal dissolved metals 2 sample (no duplicate).

APPENDIX 7: Mercury - EPA1631 Trace-Level Mercury Sampling SOP

Mercury (Hg) Description:

Mercury and mercuric salts are considered to be highly toxic to humans and aquatic life. Elemental mercury is inert chemically and insoluble in water and is not likely to occur as a water pollutant. Mercuric salts occur in nature chiefly as the sulfide HgS, known as cinnabar, but numerous synthetic organic and inorganic salts of mercury are used commercially and industrially. They are used in medicinal products, disinfectants, detonators, pigments, and photoengraving. Many of the mercuric and mercurous salts are highly soluble in water.

EPA1631 Trace-level Mercury Sampling Standard Operating Procedure:

Samples for EPA1631 trace-level mercury analysis must be packaged and transported to the WSS Chemistry Central Laboratory using the sample kits provided by the laboratory. No exceptions are allowed.

The WSS Chemistry Laboratory conducts Mercury Cold Vapor Atomic Fluorescence Spectrometry (CVAFS) (Trace-level total Hg) testing.

From: EPA-821-R-05-001, February 2005, Method 245.7, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry. Revision 2.0, February 2005. "*EPA Method 245.7, Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry, was developed through a collaboration between EPA's Environmental Monitoring Systems Laboratory, EPA Region 4, and Technology Applications, Inc. In developing this method, EPA sought to provide the environmental monitoring community with a rugged analytical protocol capable of determining mercury (Hg) at the concentrations typically regulated under State water quality standards. EPA developed this version of the method specifically to address State needs for measuring toxic metals at ambient water quality criteria (WQC) levels, when such measurements are necessary to protect designated uses."*

"This method is accompanied by Method 1669: Sampling Ambient Water for Determination of Trace Metals at EPA Water Quality Criteria Levels. This sampling

guidance is recommended to preclude contamination during the sampling process."

"The ease of contaminating ambient water samples with mercury and interfering substances cannot be overemphasized. This method includes suggestions for improvements in facilities and analytical techniques that should minimize contamination and maximize the ability of the laboratory to make reliable trace metals determinations. Certain sections of this method contain suggestions, and other sections contain requirements to minimize contamination."

Protocol and Suggestions for Minimizing Contamination for Mercury Hg1631 Samples.

- Follow "Clean Hands" and "Dirty Hands" Mercury Hg 1631 sampling procedures.
- Field personnel should consult with the QA Coordinator, AMS Coordinator, and Ecosystems Branch Supervisor if a metal bridge sampler is needed to sample for Mercury Hg 1631.
- Avoid possible airborne contamination from smoking, automobile exhaust, airborne dust/particulate matter, windblown dirt, and vapors.
- Use caution when handling sample equipment to avoid placing equipment on dirty surfaces or ground.
- Collect samples away from metal objects, bridges, and posts.
- When completing RAMS sampling, low-level trace mercury should be the first parameter sampled after arrival to the RAMS site due to high possibility and likelihood of contamination from carryover from other sampling collection and preservation activities.
- Low-level trace mercury should not be sampled during rain events, wind events, or near running vehicles.

WSS Chemistry Laboratory Supplied Mercury Sampling Kits**.**

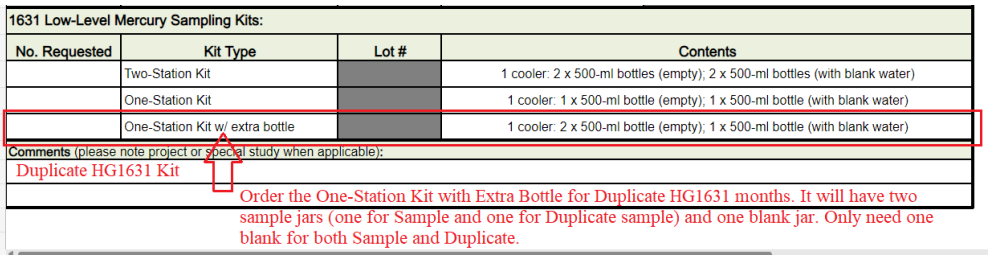
A Water Sciences Section Inorganic Chemistry Branch Metals Unit memorandum on Sampling Kits for Low Level Mercury Analysis by EPA Method 1631 Memorandum can be found online.

Order EPA-1631 Hg Sampling kits from the WSS Chemistry Lab by visiting Supplies, Fees & Assistance NC DEQ website and fill out the Excel spreadsheet order form. The one station kit contains: 1 cooler, 1 x 500 mL glass jar (empty), and 1 x 500 mL

glass jar (with blank water). The Excel spreadsheet can then be emailed to dwr.mercurykit@deq.nc.gov. Each site requires one kit. There should never be ice inside of the cooler. Collected mercury samples should be transported inside of the cooler originally provided in the mercury kit from the office to the sample site to the lab, without ice.

During duplicate mercury (Hg 1631) months, field personnel collect one field blank, similar to regular monthly mercury sampling, and then immediately collect two environmental samples.

Duplicate mercury Hg 1631 months require ordering a One-Station Kit with an extra bottle from the WSS Chemistry Laboratory to complete duplicate Hg 1631 samples (Figure 30). The One-Station Kit with an extra bottle will contain two sample jars and one field blank jar. Collect the duplicate Hg 1631 sample immediately after completing the field blank and regular mercury sample. Only one Hg 1631 field blank is required for both Hg 1631 field sample duplicate and regular field environmental sample during the duplicate month.



1631 Low-Level Mercury Sampling Kits:

No. Requested	Kit Type	Lot #	Contents
	Two-Station Kit		1 cooler: 2 x 500-ml bottles (empty); 2 x 500-ml bottles (with blank water)
	One-Station Kit		1 cooler: 1 x 500-ml bottle (empty); 1 x 500-ml bottle (with blank water)
	One-Station Kit w/ extra bottle		1 cooler: 2 x 500-ml bottle (empty); 1 x 500-ml bottle (with blank water)

Comments (please note project or special study when applicable):

Duplicate HG1631 Kit

Order the One-Station Kit with Extra Bottle for Duplicate HG1631 months. It will have two sample jars (one for Sample and one for Duplicate sample) and one blank jar. Only need one blank for both Sample and Duplicate.

Figure 30. Ordering Duplicate HG 1631 kit known as a One-Station Kit w/ extra bottle.

Trace-level mercury sampling is a surface grab sample. Mercury sampling protocol is based on Method 1669: Sampling Ambient Water for Determination of Trace Metals at EPA Water Quality Criteria Levels to reduce contamination during the sampling process.

<u>"Clean Hands" and "Dirty Hands" Mercury Sampling Protocol.</u>
Mercury sampling requires two field personnel, one of which is referred to as "**Dirty Hands**" and one as "**Clean Hands**."

"Dirty Hands" refers to the personnel who will hand clean gloves to "Clean Hands" and to themselves, opens the cooler to retrieve the plastic bags, closes cooler to

return the bags, opens up the outside plastic bag which houses a second plastic bag, and closes the outside bag. “Dirty Hands” should not touch the inside of the bags, the inside bag, any clothing or skin, or anything other than what is listed in the directions.

“Clean Hands” refers to the personnel who will open up the inside bag and reach into the inside bag to retrieve glass jars, pour the field blank DI water from the sample jar (marked with a star (*)) into the field blank jar (empty at first; with no star (*)), and collect the sample from the water body into the now empty jar with a star (*). “Clean Hands” should never touch the outside of the outside bag, any clothing or skin, or anything other than the inside bags and sample containers.

Figure 31 lists duties of “Dirty Hands” and “Clean Hands”. From Brooks Applied Labs, environmental-sampling-for-low-level-trace-metals-per-epa-method-1669.pdf (brooksapplied.com). “*EPA Method 1669 also requires that a two-member team participate in the collection of samples. “Clean Hands” is responsible for all procedures involving direct contact with the sample and the sample container. “Dirty Hands” is responsible for preparing the sample containers for collection, operating any machinery, and all other activities that could lead to contamination of the sample.”*

“Dirty Hands”	“Clean Hands”
does not disturb sample source	directly contacts sample source
does not touch primary container bags/inside bag (inner bag)	opens/closes primary container bags
opens/closes secondary container bags (outer bag)	does not touch secondary container bags (outer bag)
does not touch “clean equipment”	handles all “clean” materials
handles all “non-clean” materials	does not touch “non-clean” materials
opens/closes shipping containers	does not touch shipping containers
does not touch sample containers	directly holds sample container

Figure 31. Duties of "Dirty Hands" and "Clean Hands"

Modified AMS/RAMS Mercury – EPA 1631 Trace-Level Mercury Sampling Standard Operating Procedure. The AMS/RAMS Mercury – EPA1631 Trace-Level Mercury Sampling Standard Operating Procedure uses some of the steps copied from EPA Method 1669 and other steps have been modified.

- *Instructions in regular italics are copied from EPA Method 1669.*
- ***Instructions in bold italics are specific to NC DEQ AMS/RAMS Sampling. The instructions may be modifications of EPA Method 1669, clarifications, or specific to AMS and RAMS Program sampling.***

At the site, all sampling personnel must put on clean gloves before commencing sample collection activity. ***"Dirty Hands" puts on a pair of nitrile gloves, then offers a box of clean gloves for "Clean Hands" to put clean nitrile gloves on. "Dirty Hands" then allows "Clean Hands" to take a pair of shoulder-length gloves from the bag of shoulder length gloves for "Clean Hands" to put on. After this, both personnel put on a final pair of nitrile gloves. Field personnel (both "Dirty Hands" and "Clean Hands") may wear 2 sets of outer nitrile gloves removing one set of gloves to avoid contamination if they may have touched things not listed in the directions. If contamination is believed to be a factor in the sampling, then note the incidence on the field sheets.***

Note that "Clean Hands" should put on shoulder-length polyethylene gloves and both "Clean Hands", and "Dirty Hands" should put on the nitrile gloves. ***"Dirty Hands" should not need shoulder length gloves as they will not reach into the bags. Shoulder-length gloves are 35" length, 1.25 mil, poly gloves.***

"Dirty Hands" places a new, unused, clean trash bag either supplied by the AMS Coordinator or by field personnel on top of another cooler to be used as a clean table. No trash bag specifics are given other than trash bags should be new and clean.

In the Mercury cooler provided by the WSS Chemistry Laboratory there will be one double bag with the field blank water in a glass jar with a cap marked with a star (*) and one double bag with an empty glass jar with a cap with no star (*).

Start by removing either jar. *"Dirty Hands" must open the cooler, remove the double-bagged sample jar from storage, and unzip the outer bag without touching the inside of the bag.*

Next, "Clean Hands" opens the inside bag containing the jar, removes the jar, and zips/reseals the inside bag. "Dirty Hands" then zips/reseals the outer bag.

After removing the jar from the bag, both double bags should be resealed empty to prevent contamination while sampling. "Clean Hands" should not touch the outside bags.

Complete this process again to remove the second jar.

Both jars are placed on the trash bag on top of the cooler after being removed from the plastic bags. "Clean Hands" handles the sampling jars. "Dirty Hands" should not touch sampling jars or items that come into contact with the sample jars.

Field Blank Procedure.

"Clean Hands" removes the cap from the empty jar with no star (*) and sets the cap upside down on the trash bag table. "Clean Hands" removes the cap from the jar with the blank water (marked with a star (*)) and transfers the blank water to the empty jar with no star (*).

"Clean Hands" may hold the cap (with a star (*)) in one hand and recap immediately the empty jar after transferring the blank water to the field blank jar with no star (*) or set the cap upside down on the trash bag table.

1. ***After filling the blank water field blank jar "Clean Hands" immediately screws the cap marked with a star (*) back on its now empty jar so it is covered until sampling takes place.***
2. ***If the cap marked with a star (*) was put on the trash bag table, "Clean Hands" now caps the empty jar with the cap marked with a star (*) so it is covered until sampling takes place.***

"Clean Hands" now caps the blank water filled field blank jar with the cap with no star (*).

"Dirty Hands" opens the outer bag with no star (*). "Clean Hands" opens the inner bag, then places the jar filled with field blank water (i.e., jar with no star (*) cap) into the inner bag. "Clean Hands" zips/seals the inner bag and "Dirty Hands" zips/seals the outer bag.

"Dirty Hands" returns the field blank double-bagged Field Blank (jar with no star (*)) sample (both inner and outer bags should be sealed) to the mercury cooler.

The Field Blank (jar with no star (*)) sampling procedure is complete.

Mercury Environmental Sample Procedure.

"Clean Hands" will now take the capped empty jar with the screwed on cap marked with a star (*) and use the jar to collect water from the site for the WSS Chemistry Laboratory to test for mercury. To sample for mercury, "Clean Hands" first dumps remaining field blank water in the jar out.

"Clean Hands" should rinse the sample collection jar with a star (*) with site water to remove Hg-free blank water remaining in the jar. Specifically, as mentioned in EPA 1669, *"Clean Hands" then submerges the sample jar* ***(cap marked with a star (*))*** *and allows the jar to partially fill with sample. "Clean Hands" screws the cap on the jar, shakes the jar several times, and empties the rinsate away from the site. After two more rinsings, "Clean Hands" holds the jar under water and allows jar to fill with sample. After the jar has filled (i.e., when no more bubbles appear), and while the jar is still inverted so that the mouth of the jar is underwater, "Clean Hands" replaces the cap on the jar. In this way, the sample has never contacted the air.*

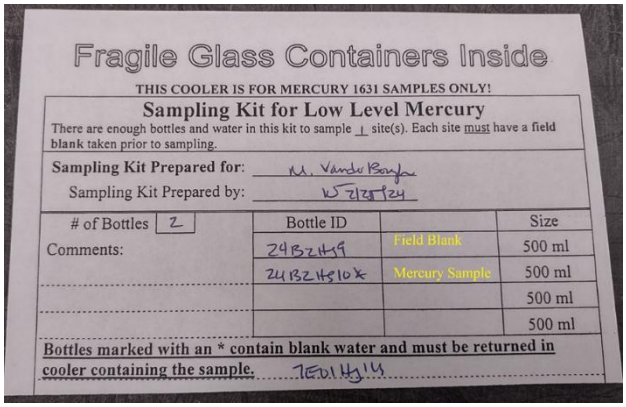
*Once the jar cap has been replaced...****the filled and capped sample jar can be placed on the garbage bag and cooler while the double bags are opened.*** *"Dirty Hands" reopens the outer plastic bag, and "Clean Hands" opens the inside bag, places the* ***sample filled*** *jar with a star (*) inside the inner bag, and zips/seals the inner bag. "Dirty Hands" zips/seals the outer bag.*

"Dirty Hands" returns the mercury double-bagged sample (both inner and outer bags sealed) to the mercury cooler.

The samples are returned to the cooler without ice. Mercury samples are not put on ice. There are no field preservatives for low-level mercury samples.

After the samples have been placed in the cooler with both inner and outer bags closed, "Dirty Hands" can re-tape the cooler shut. Both personnel can now remove their gloves. Mercury Sampling and "Clean Hands" / "Dirty Hands" is complete.

On top of the cooler, remove the WSS Chemistry Laboratory Supplied Sampling Sheet. Write <u>Mercury Sample</u> in the middle section next to number + star (*). Write <u>Field Blank</u> in the middle section next to the number with no star (*). The bottle with no star (*) is always the field blank, while the bottle with the star (*) is the mercury sample. Place this low-level mercury sampling sheet back into the shipping pocket on top of the mercury kit cooler.


Fragile Glass Containers Inside

THIS COOLER IS FOR MERCURY 1631 SAMPLES ONLY!

Sampling Kit for Low Level Mercury

There are enough bottles and water in this kit to sample 1 site(s). Each site must have a field blank taken prior to sampling.

Sampling Kit Prepared for: M. VanderBoyle

Sampling Kit Prepared by: WS 7/25/24

# of Bottles 2	Bottle ID		Size
Comments:	24BZH59	Field Blank	500 ml
	24BZH510*	Mercury Sample	500 ml
			500 ml
			500 ml

Bottles marked with an * contain blank water and must be returned in cooler containing the sample. TEDI Hg1/4

Figure 32. WSS Chemistry Laboratory Supplied Sampling Sheet for Sampling Low-Level Mercury (Mercury 1631).

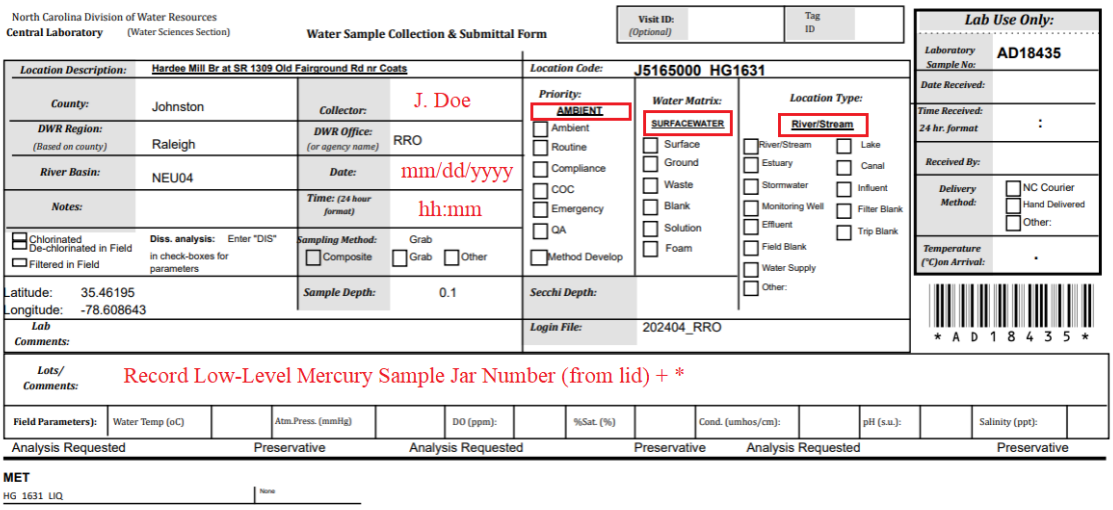
<u>Mercury Field sheets.</u>

There will be one field sheet (**HG1631**) for the low-level mercury sample and one field sheet for the mercury field blank (**FB**).

Mercury Sample (HG1631) Field sheet.

The field sheet for the low-level mercury sample will have the Location Code and **HG1631** listed.

- Write the sample number + star (*) on the mercury sample (HG1631) field sheet in the Lots/Comments section (Figure 33). The sample number is written on the lid of the sample jar. Mercury sample has a star (*).



North Carolina Division of Water Resources
Central Laboratory (Water Sciences Section)

Water Sample Collection & Submittal Form

Visit ID: (Optional)

Tag ID

Lab Use Only:
Laboratory Sample No: AD18435
Date Received:
Time Received: 24 hr. format :
Received By:
Delivery Method: NC Courier; Hand Delivered; Other:
Temperature (°C)on Arrival:

Location Description: Hardee Mill Br at SR 1309 Old Fairground Rd nr Coats
Location Code: J5165000_HG1631

County: Johnston
Collector: J. Doe
DWR Region: (Based on county) Raleigh
DWR Office: (or agency name) RRO
River Basin: NEU04
Date: mm/dd/yyyy
Notes:
Time: (24 hour format) hh:mm
Chlorinated De-chlorinated in Field; Filtered in Field
Diss. analysis: Enter "DIS" in check-boxes for parameters
Sampling Method: Composite; Grab; Other
Latitude: 35.46195
Longitude: -78.608643
Sample Depth: 0.1
Secchi Depth:
Lab Comments:
Login File: 202404_RRO

Priority: AMBIENT; Ambient; Routine; Compliance; COC; Emergency; QA; Method Develop
Water Matrix: SURFACEWATER; Surface; Ground; Waste; Blank; Solution; Foam
Location Type: River/Stream; River/Stream; Estuary; Stormwater; Monitoring Well; Effluent; Field Blank; Water Supply; Other: Lake; Canal; Influent; Filter Blank; Trip Blank

* A D 1 8 4 3 5 *

Lots/ Comments: Record Low-Level Mercury Sample Jar Number (from lid) + *

Field Parameters): Water Temp (oC); Atm.Press. (mmHg); DO (ppm): ; %Sat. (%); Cond. (umhos/cm): ; pH (s.u.): ; Salinity (ppt):

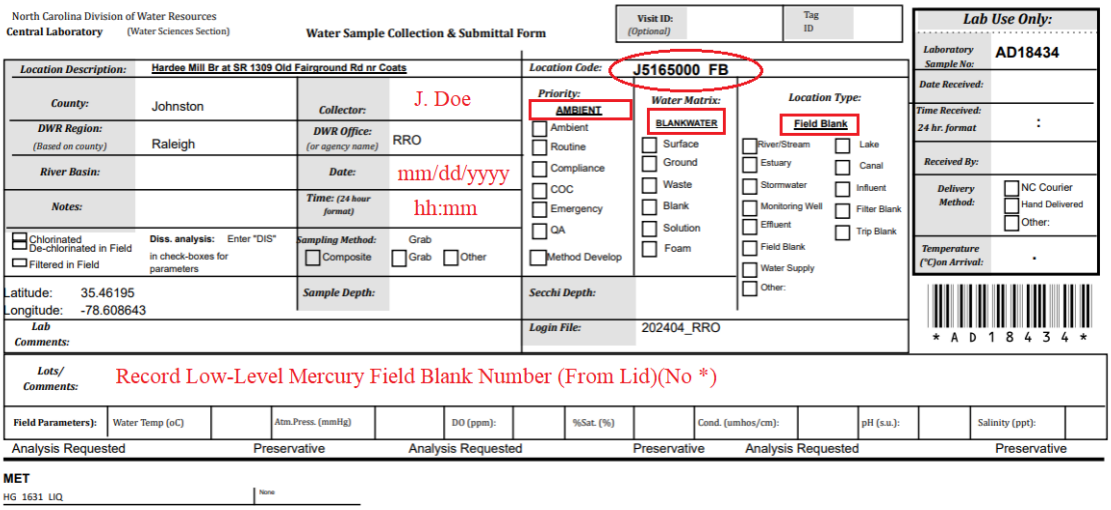
Analysis Requested	Preservative	Analysis Requested	Preservative	Analysis Requested	Preservative
MET					
HG 1631 LIQ	None				

Figure 33. Low-Level Mercury (HG1631) Field sheet Indicating Entries to Fill Out and List the Low-Level Mercury Sample Number + star (*)

Mercury Field Blank (FB) Field sheet.

The field sheet for the field blank (**FB**) will have the Location Code and FB listed.

- Write the field blank number (number with no star (*)) on the mercury field blank (FB) field sheet in the Lots/Comments section. The field blank number is written on the lid of the field blank jar. Field blank does not have a star (*).



North Carolina Division of Water Resources
Central Laboratory (Water Sciences Section)

Water Sample Collection & Submittal Form

Visit ID: (Optional) | Tag ID

Lab Use Only:
Laboratory Sample No: AD18434
Date Received:
Time Received: 24 hr. format :
Received By:
Delivery Method: NC Courier; Hand Delivered; Other:
Temperature (°C)on Arrival: .

* A D 1 8 4 3 4 *

Location Description: Hardee Mill Br at SR 1309 Old Fairground Rd nr Coats
Location Code: J5165000_FB
County: Johnston
Collector: J. Doe
DWR Region: (Based on county) Raleigh
DWR Office: (or agency name) RRO
River Basin:
Date: mm/dd/yyyy
Notes:
Time: (24 hour format) hh:mm
Chlorinated; De-chlorinated in Field; Filtered in Field
Diss. analysis: Enter "DIS" in check-boxes for parameters
Sampling Method: Composite; Grab; Other
Latitude: 35.46195
Longitude: -78.608643
Sample Depth:
Lab Comments:

Priority: AMBIENT; Ambient; Routine; Compliance; COC; Emergency; QA; Method Develop
Water Matrix: BLANKWATER; Surface; Ground; Waste; Blank; Solution; Foam
Location Type: Field Blank; River/Stream; Estuary; Stormwater; Monitoring Well; Effluent; Field Blank; Water Supply; Other; Lake; Canal; Influent; Filter Blank; Trip Blank
Secchi Depth:
Login File: 202404_RRO

Lots/ Comments: Record Low-Level Mercury Field Blank Number (From Lid)(No *)

Field Parameters): Water Temp (oC); Atm.Press. (mmHg); DO (ppm); %Sat. (%); Cond. (umhos/cm); pH (s.u.); Salinity (ppt);

Analysis Requested | Preservative | Analysis Requested | Preservative | Analysis Requested | Preservative

MET
HG 1631 LIQ | None

Figure 34. Low-Level Mercury Field Blank (FB) Field sheet Indicating Entries to Fill Out and Record the Low-Level Mercury Field Blank Number (number with no star (*))

Mercury Sample Shipping:

The completed field sheets can be placed under the tag on the mercury cooler (or with the other field sheets). Mercury coolers should not contain ice and mercury samples should not be put on ice. The samples are shipped and returned in the cooler without ice. There are no field preservatives for low-level mercury samples.

APPENDIX 8: Thermistor Check Procedure.

Figure 35. In-Situ Thermistor Check Procedure

Thermistor Check Schedule

- It is recommended to conduct the AMS and RAMS Thermistor Check once every four months (Tri annually), if possible.
- At a minimum, the AMS and RAMS Thermistor Check will be conducted once per year (2024 AMS and RAMS QAPP).
- The annual In-Situ Factory Calibration in September through December of each year will be used for an additional check point, but not used to qualify data.
- The In-Situ Field Meter Temperature Probes are starting to fail 3 years after purchase so it is imperative to perform more thermistor checks throughout the year in order to reduce the number of possible data qualifications based on thermistor check failures.

Thermistor Check Data Entry.

The thermistor results should be recorded in the ArcGIS Survey 123 form called “AMS and RAMS Thermistor Check”.

AMS and RAMS Thermistor Check Performance Criteria.

- The performance criteria for a thermistor check is ≤ 0.5 °C difference between the field meter temperature and the NIST-certified digital thermometer temperature for both the ambient and cool water temperature checks. A thermistor check failure results when the check does not meet the performance criteria (> 0.5 °C) at either one temperature (ambient or cool water) or both temperatures (ambient and cool water).
- The performance criteria for thermistor checks is from the North Carolina Wastewater/Groundwater Laboratory Certification Branch Approved Procedure for the Analysis of Temperature.

  "All compliance temperature measurements must be made with a National Institute of Standards and Technology (NIST)traceable temperature-measuring device that has a demonstrated accuracy of ± 0.5 °C and equilibrates rapidly."
  "To check a compliance temperature-measuring device, compare readings at two temperatures that bracket the range of compliance samples routinely analyzed against a Reference Temperature-Measuring Device and record all four readings. The difference between the readings from both devices must be ≤ 0.5 °C*."*

- Field meter measurements that fail a thermistor check are qualified with the qualifier code J12.

AMS and RAMS Thermistor Check Standard Operating Procedure

- I don't believe the field meters need to be calibrated in order to perform a thermistor check as the temperature probe is never calibrated (except by In-Situ), so calibration should not affect temperature.
- The AMS and RAMS program Thermistor Check will test field meter thermistors at two temperatures. Ambient water temperature and Cool water temperature.

  Note: In the past water ice bath temperatures were used as the second temperature. Starting in 2025, the AMS and RAMS Program will switch ice bath temperatures to cool water temperatures to negate problems and issues that occurred during ice bath readings. See section concerning discontinuing water ice bath temperature readings.

- One milk jug (½ gallon or 1 gallon) can be filled with water and stored in the refrigerator overnight for the cool water temperature readings.
- Another milk jug (½ gallon or 1 gallon) can be filled with water and left out at room temperature for the ambient temperature readings.
- The tops of the milk jugs can be cut off and can be used for the measurement container. The first milk jugs can be replaced with a second milk jug using 2 meter stands with the probes at fixed positions. One meter stand is for the In-Situ field meter probe and the second meter stand is for the thermometer probe.
- The two meter probe stands can be used to attach the field meter probe and NIST-certified thermometer close to each other. The probes can be lowered into the gallon or ½ gallon jug not touching the bottom of sides of the jug.
- The NIST-certified thermometer should be placed as close as possible to the temperature probe located on the field meter probe. The temperature probe on the In-Situ field meter is the small metal disc on the large conductivity probe. See the Sensor diagram for the location of the temperature probe on the In-Situ 400 model field meter.
- Both the field meter probe and the NIST-certified thermometer should not touch the sides or bottom of the plastic milk jug (sample container). The NIST-Certified Thermometer is very sensitive. If the jug of water is on a cold stainless-steel bench and the NIST-certified thermometer is touching the bottom of the jug, the cold stainless steel will affect the temperature of the NIST-certified thermometer.
- Similarly, once the field meter probe and NIST-certified thermometer are set up according to these procedures, one should wait 5 minutes or more for the temperatures of both probes to stabilize.
- Allow the temperature readings to stabilize before documenting the temperature readings from the field meter and NIST-certified thermometer. The temperature readings of the NIST-certified thermometer will vary/move up and down throughout the whole event, therefore judgement must be made when both readings are pretty stable and accurate.

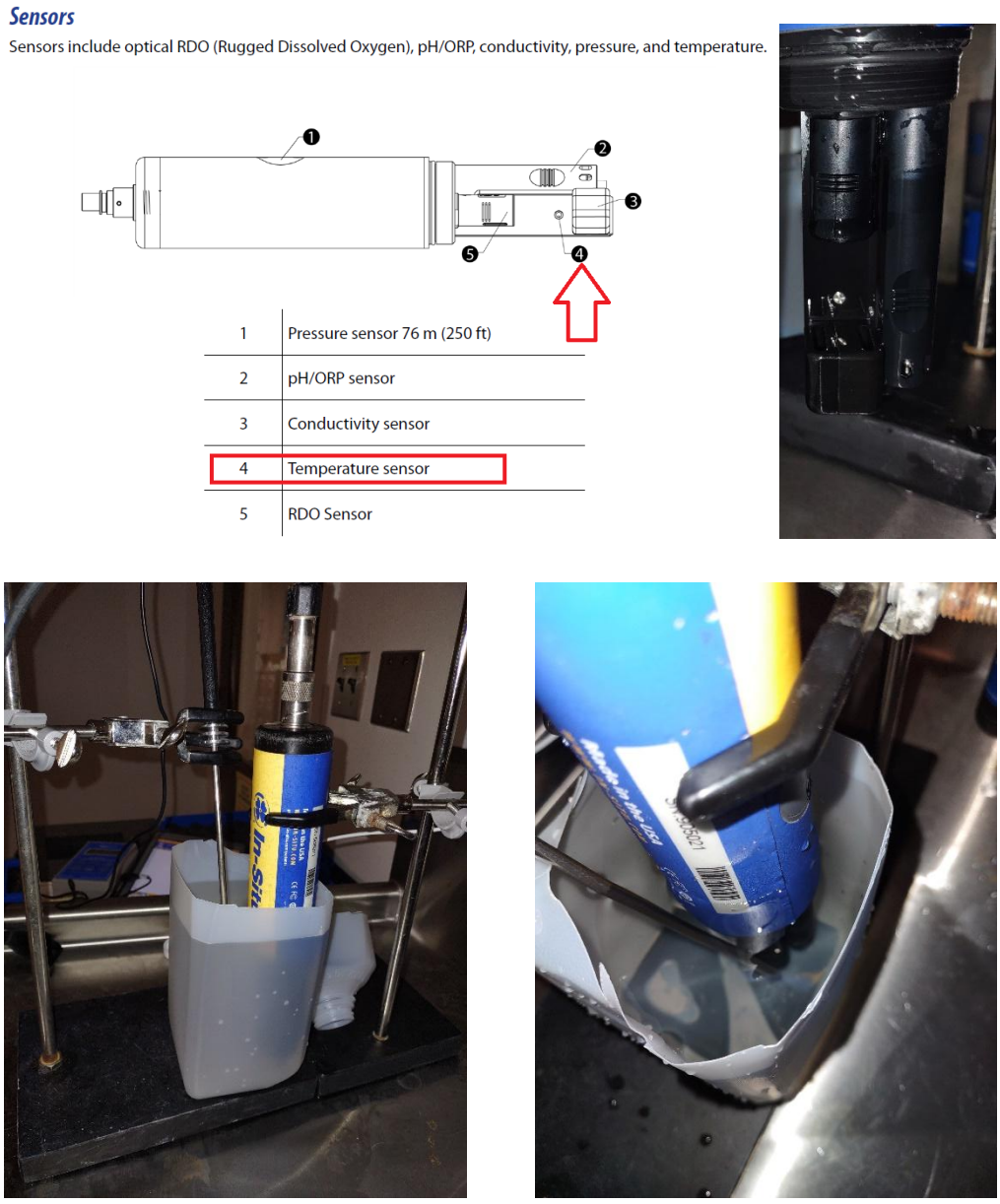


Figure 36. In-Situ Thermistor Check Ice Bath Procedure

Discontinued use of Water Ice Bath temperatures during AMS and RAMS Thermistor Checks.

In 2025 the use of water ice baths for AMS and RAMS Thermistor tests will be discontinued due to fluctuating and inconsistent temperature readings observed using water ice baths.

Water ice bath conditions require the water and ice mixture to be stirred to prevent cold pockets from forming. Cold pockets may cause differences in temperature readings between the NIST-certified thermometer and field meter that are related to position of the probes in the water and not due to field meter or thermometer error.

The multiparameter probe guard should also been removed during the test since it is metal and the metal probe could possibly cool the probe down further than the water ice bath and/or NIST-certified thermometer reading. The metal probe guard also trapped ice causing cooler temperatures for the field meter probe.

Switching to cool water temperature should provide more representative readings of conditions observed in the field and hopefully less errors in measurements not related to the actual performance of the thermistor/probes. Ice cold temperatures are rarely observed in AMS and RAMS sampling. Cool water temperatures represent the lower end of the temperature range observed in AMS and RAMS sampling than Ice temperatures.

Responsibilities.

Field Personnel.
Conduct thermistor checks and record data in ArcGIS Survey123 form called "AMS and RAMS Thermistor Check" according to prescribed schedule.

QA Coordinator.
The QA Coordinator will monitor thermistor checks quarterly. The thermistor check data is compiled into a spreadsheet on annual basis and stored on the WSS Ecosystem Branch QAQC SharePoint site. From this data, the PQAM QA Coordinator will create a "Yearly Thermistor Check Report" with a list of field

meters that have failed the thermistor check performance criteria. This report will list dates, field collector's name, field meter serial numbers, and any other information pertaining to the thermistor check failure. The completed report is submitted to the AMS Coordinator by the end of January of the following year.

AMS Coordinator.
The AMS Coordinator is responsible for applying data qualifications to appropriate field meter measurements based on thermistor check performance criteria and guidelines. Purchases new thermometers. Responsible for the completion of thermistor checks by field personnel.

Batteries**.**
It is recommended to remove the battery when not in use. Each year it seems like when the Thermometers are sent in or when used, the battery is dead.

Other Information

Refer to the Thermistor Check Presentation at the 11/6/2024 AMS Workshop kept on the WSS Ecosystems Branch QAQC SharePoint site.

Other Thermistor Checks**.**
The annual In-Situ factory calibration and maintenance check as part of the Annual Service Agreement should be performed from September through December of each year. The annual In-Situ factory calibration and maintenance check performed by In-Situ will provide an end of the year thermistor check. It will not be used to qualify data though as only one temperature is used.

Results of the annual In-Situ factory calibration and maintenance check should be kept with the AMS/RAMS Thermistor check data.

In-Situ annual factory calibration and maintenance check thermometer check data (Room Temperature Check - Performance Tests Reading in °C, Pass <+/- 0.25 °C) should be entered into ESRI Survey123 Thermistor Check Data so both In-Situ results and AMS and RAMS Thermistor Check data results are in the same table.

In-Situ Annual Service Agreement and Temperature Checks.

Some meters purchased in 2022 have a 5-year warranty and are enrolled in annual service agreement. In-Situ Field Meters covered under the annual service

agreement should be sent in to In-Situ for factory calibration and maintenance checks. In-Situ has reported to conduct several QA checks including:

- Room Temperature Check - Performance Tests Reading in °C, Pass <+/- 0.25 °C
- 900 µS/cm Check -Performance Tests Reading in µS/cm, Pass <+/- 5% µS/cm
- 9000 µS/cm Check - Performance Tests Reading in µS/cm, Pass <+/- 5% µS/cm
- 90000 µS/cm Check - Performance Tests Reading in µS/cm, Pass <+/- 5% µS/cm
- 100% Saturation Check - Performance Tests Reading in percent saturation, Pass <+/- 10% from 100%

NIST-Certified Digital Thermometer Annual Calibration.

- On an annual basis (in September) the digital thermometers used for thermistor checks will need to be calibrated. Digital Thermometers are NIST Certified Fisherbrand Thermometers, Model 15-081-102.
- Precision Weighing conducts the calibrations. Thermometers must be collected from the field personnel and delivered to Precision Weighing for calibration.
- Contact: Shauna Brabble, Precision Weighing, 1949 Evans Road, Cary, NC 27513, 919-678-0077, pweighing@aol.com.
- Precision Weighing Technician, Curt: 919-218-8360
- Precision Weighing Technician, Rick: 919-600-4520

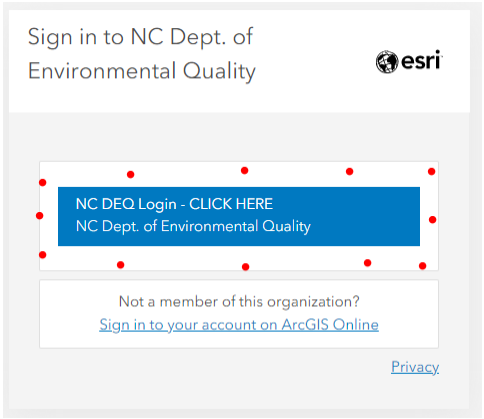
NIST-Certified Digital Thermometer Inventory as of 2024.

2024				
NIST Certified Thermometer Brand	Model	Serial Number	Date of Last Calibration	Region
Fisherbrand	15-081-102	192279069		ARO
Fisherbrand	15-081-102	200545193		EMT/WaRO
Fisherbrand	15-081-102	200545192		FRO
Fisherbrand	15-081-102	200545183		MRO
Fisherbrand	15-081-102	202545190		RRO
Fisherbrand	15-081-102	200545185		WiRO
Fisherbrand	15-081-102	200545199		WSRO
Fisherbrand	15-081-102	200545189		WSS
Fisherbrand	15-081-102	200545197		WSS
Fisherbrand	15-081-102	151978321		WSS
Fisherbrand	15-081-102	200545194		WSS

ArcGIS Online.

ArcGIS Online account for Field Personnel are set up with a Basic User role. New employees should contact their IT representative and/or kerry.hanko@deq.nc.gov to be added to the NC DEQ ESRI AGOL. Once added to the site, the directions for access to the ESRI AGOL site are as follows:

- Go to NC DEQ AGOL home page
- Click Sign In in the top right corner.
- Click the **Enterprise login** button.



- Then click the blue **NC Enterprise** button.
- Type in your email address and password.
- You will land back on the NC DEQ AGOL homepage. We recommend that you bookmark the home page.

AMS and RAMS Field Meter Thermistor Check

Collector: _____ **Agency/Study/Location:**_____

Field Meter Model: _____**Field Meter Probe Serial #:** (not TROLL COM Bluetooth #) _____

	Date mm/dd/yyyy	24 HR Time (hh:mm)	Initials
Thermistor Check Information			

	Serial Number
NIST-Certified Fisherbrand Thermometers, Model 15-081-102	

NIST-Certified Thermometers are calibrated once per year in September by Precision Weighing, 1949 Evans Road, Cary, NC 27513, 919-678-0077. Contact Shauna Brabble pweighing@aol.com.

Field Meter and NIST-Certified Thermometer TEMPERATURE MEASUREMENTS

Temperature	Temperature from Field Meter (In-Situ)	Temperature from NIST-Certified Thermometer	Record the difference in Temperature between the Field Meter and NIST-Certified Thermometer. Field Meter Temperature minus NIST-Certified Thermometer Temperature reading.	Is the temperature difference between the readings of the Field Meter and NIST-Certified Thermometer less than or equal to 0.5 ºC (= 0.5 ºC)? Circle Yes or No.	A pass is where the difference between the readings from both devices is less than or equal to 0.5 ºC (= 0.5 ºC). A failure is where the difference between the readings from both devices is greater than 0.5 ºC (>0.5 ºC) (0.6 ºC or higher). Circle Pass or Failure.
Ambient Water				Yes or No	Pass or Failure
Cool Water Temperature				Yes or No	Pass or Failure

Contact randy.sloup@deq.nc.gov with any questions and comments, or for help in completing the sheet. Revised 8/30/2024.

Figure 37. In-Situ Thermistor Calibration Sheet

APPENDIX 9. Legal or Evidentiary Chain of Custody Procedures.

Purpose: To ensure samples or physical evidence collected as part of an investigation for legal proceedings or as required by a study plan are traceable from collection to presentation in legal proceedings, maintaining integrity and preventing tampering.

Scope: This procedure applies to all personnel involved in collecting, handling, transporting, or storing samples or evidence intended for legal or regulatory purposes.

Sample Custody

A sample or other physical evidence is considered under custody if it is:

- In the field investigator's actual possession.
- Within the field investigator's view after being in their possession.
- Secured by the field investigator to prevent tampering after being in their possession; or
- Stored in a designated secure area with restricted access.

Key Principle: To minimize litigation risks and simplify the chain-of-custody record, limit the number of individuals handling samples or evidence. The field investigator is responsible for the care and custody of samples from collection until properly transferred to an authorized person or facility.

Field Custody Procedures

Security Seal

- Complete sample tags for each sample, including:
  - Sample ID, collection date, time, location, and investigator's name.
  - Use legible, waterproof tags securely attached to the sample container.

- Place laboratory sheets and chain-of-custody forms in a waterproof Zip-loc bag and store in a cooler with the samples.
- Seal coolers with tamper-evident filament tape and a DWR custody seal (figure 38)
- The field investigator must write their name, date, and time on the custody seal. This requirement is waived if the field investigator maintains continuous custody from collection to laboratory delivery.
- Optional Digital Seal Verification: Where available, use tamper-evident digital tracking (e.g., QR codes or RFID tags) to supplement physical seals for real-time custody verification.

Chain-of-Custody Form

Record all samples in the **field logbook** or on the **Chain-of-Custody Record** (Figure 39, available at: **Sample Submission | NC DEQ. Include**:

- Sample ID, collection date, time, location, and description.
- Names and signatures of all personnel handling the sample.
- Details of any transfer, including date, time, and recipient.

For **documents** collected during investigations:

- Place documents in a large envelope and seal with a DWR custody seal to prevent opening without breaking the seal.
- Note the contents on the envelope and include a chain-of-custody record.
- If a seal is broken, document the reason, investigator's signature, date, and time on the chain-of-custody record, then affix a new seal.

For other physical evidence (e.g., videotapes, small items):

- Place items in a Zip-loc bag and seal with a DWR custody seal to prevent opening without breaking the seal.
- Include a chain-of-custody record inside or securely attached to the bag.
- If a seal is broken, document the reason, investigator's signature, date, and time on the chain-of-custody record, then affix a new seal.

Acceptance of External Samples:

- Do not accept samples from external sources unless:
  - Collection procedures are legally defensible and documented.

- The chain-of-custody is fully traceable from collection to receipt.

- If accepted, complete a sample tag and DWR chain-of-custody form for each sample, documenting all relevant details.

Digital Chain-of-Custody (Optional): Use electronic chain-of-custody systems, where available, to log sample details, transfers, and custody events in real time, ensuring secure access and audit trails.

Figure 38. DWR Chain of Custody Security Seal

NC DEQ DWR WSS **Chain of Custody**
Chemistry Laboratory

Samples Submitted to:	( ) Central Lab - Raleigh ( ) Asheville Regional Lab - Swannanoa	
Purpose / Investigation:		
Sample Collector(s): Please Print #1 Reserved for Lead Sampler	#1	#3
	#2	#4
Field Storage conditions & location: *(when applicable)*		

Lab Use Only Lab Sample No.	Location Code or Tag Number	Location Description	Date Collected	Time Collected	Collector	# Of Sample Containers

Relinquished by: *(signature)*	Date Relinquished	Time Relinquished	Received by: *signature)*	Date Received	Time Received

Method of Shipment *(circle one)* : State Courier Hand-Delivered Federal Express UPS Other:

Shipment Container Sealed By: *(signature)*	**Security Tape Broken By**: *(signature)*
Phone # (To reach you in the field):	Email:

Container / Security Tape Comments: o Good Condition o Signed and Dated o Not Signed and Dated o Seal Ragged o Seal Torn o No Seal

Intra-Laboratory Chain of Custody - *Lab Use Only*

Lab Sample Numbers Starting with:	Ending with:	Number of Bottles	Analyses Requested	Relinquished By:	Received By:	Date	Time

Revised 3/2/2021 - AW Page _____of _____

Figure 39. WSS Chain of Custody Form

Transfer of Custody and Shipment

When transferring the possession of chain of custody samples, the individuals receiving the samples shall sign, date, and note the time that they received the samples on the field form or in the field log book. This action documents transfer of custody of samples from the field investigator to another person (e.g. to the laboratory).

After properly packing samples for shipment to the appropriate laboratory for analysis, secure the shipping containers using nylon strapping tape and custody seals. The seal shall be placed under the point on the tape where the ends are located and wrapped over the top of it. The seal shall be signed, dated, and the time recorded by the field investigator.

Samples split with a facility, state regulatory agency, or other government agency must be signed for on the Chain of Custody Form by the facility, state regulatory agency, or other government agency representative receiving the samples.

All samples shipped shall be accompanied by the DWR chain-of custody form(s). The original and one copy of the form will be placed in a plastic bag inside the secured shipping container. One copy of the form will be retained by the field investigator or project leader. The original of the form will be transmitted to the field investigator or project leader after samples are accepted by the laboratory.

If sent by mail, the package shall be registered with return receipt requested. If sent by common carrier, a government bill of lading or air bill should be used. Receipts from post offices, copies of bills of lading, and air bills shall be retained as part of the documentation of the chain-of-custody.

APPENDIX 10. Time-of-Travel & Dye Tracing

1. FLUORESCENT DYE

The preferred dye for use in time-of travel studies by the North Carolina Division of Water Resources is Rhodamine W. T. (20%) solution. This is a red fluorescent dye which mixes well with water and is easily detected through visual means under high concentrations and through the use of a fluorometer for concentrations to as low as 0.01 parts per billion. Rhodamine WT has properties essential for water tracing studies. Rhodamine WT is:

- water soluble,
- highly detectable-strongly fluorescent,
- fluorescent in a part of the spectrum not common to materials generally found in water, thereby reducing the problem of background fluorescence,
- harmless in low concentrations,
- inexpensive, and
- reasonably stable in a normal water environment (Wilson, Cobb & Kilpatrick, 1986).

Rhodamine dye can also be used to determine such things as short-circuiting in wastewater treatment plants, outlets from storm drains, septic tank leakage, etc.

Most of ISB's dye studies are performed as part of a waste-load allocation model. This model requires that a stream be segmented into different reaches based upon predicted stream velocities, stream morphology, total distance of the study area, and major inputs from dischargers and tributaries. A dye sampling station is required in each of these reaches.

Safety

(MSDS is kept with dye container)

Personal Protection

a. Latex or vinyl gloves (in lab and field).
b. Goggles
c. Ventilated room
d. Apron

Emergency and First Aid Procedure

a. Inhalation:

- Move to fresh air.
- Give oxygen and medical help if breathing is difficult.

b. Eye contact:

- Flush eyes with flowing water for at least 15 minutes, holding eyelids apart to irrigate thoroughly.
- Get medical attention right away.

c. Skin contact:

- Wash affected skin areas thoroughly with soap and water.
- If irritation develops, consult a physician.

d. Ingestion:

- If swallowed, dilute with water and induce vomiting.
- Get immediate medical attention.
- Never give fluids or induce vomiting if patient is unconscious or has convulsions.

Equipment - Fluorometer

Turner Designs Model 110 - reads dye concentrations directly in ppb. Operating instructions are contained in the Turner Designs Model 110 Operators manual, section 3-operations.

Turner Model 10-AU - reads dye concentrations in ppb. Operating instructions are contained in the Turner Designs Model 10-AU operating manual.

2. PRE-SURVEY

Surface Water Supplies

Identify all surface water supplies in or downstream from the study area.

Notify each water supply operator that may be affected in the study area and the NC DEQ- Division of Water Resources regional water quality supervisor that a dye study is scheduled to be performed. Explain the reason for the study and inform the water treatment operator that DWR personnel will monitor dye concentrations at their water intake. If dye concentrations in the river exceed 10 ppb, the facility will be informed to shutdown their operation until river dye concentrations fall below 10 ppb. All efforts should be made to calculate a dye dosage that will result in a dye concentration at a water supply significantly below the 10 ppb.

Field Reconnaissance

1. Select dye sampling stations. Stations are selected based upon access, distance from the dose, and model requirements.
2. Locate all USGS gage stations in the study area or sites at which flows can be performed.
3. Determine if any dam structures that can regulate flow exist in the study area. If there is such a dam structure, a station is usually set up just upstream of the dam and an additional dose is made below the dam.

3. DYE REQUIREMENTS (ESTIMATING DOSAGE)

For Rhodamine WT 20 percent dye the dosage formula is:

$$V = 3.4 * 10^{-4} * [(Qm * L)/Vm]^{0.93} * Cp$$

Where:

V is the volume of dye, in liters
Qm is the maximum discharge in the reach, in cfs
L is the distance from injection to sampling point, in miles
Vm is the mean velocity, in fps
Cp is the peak concentrations desired in µg/l

The volume of Rhodamine WT 20 percent dye required to produce a peak concentration of 1 µg/l (ppb) can be determined from the nomograph in Figure 40 for a range of flow-reach conditions.

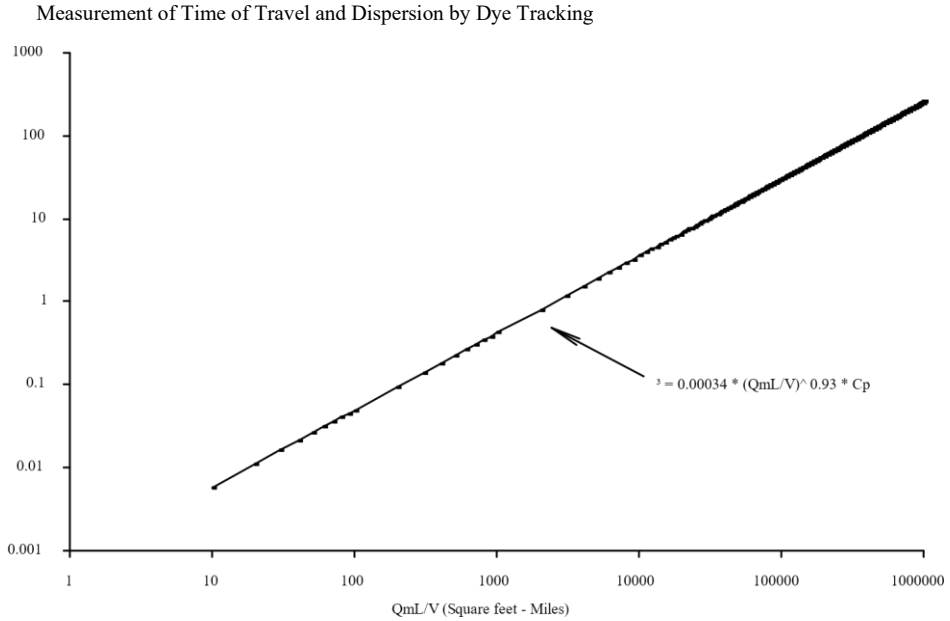


Figure 40. Nomograph for determining volume of dye necessary to produce peak concentration

4. INJECTION OF DYE

Injection Types

Single Slug Injection

- A single slug injection of dye is usually made in the center of the thread of flow.
- The desired quantity of dye as calculated in part 3 (above) can be poured into the stream from a container.

Continuous injection

- The desired quantity of dye is pumped into the water column at a fixed rate for a fixed time using an ISCO sampler or a peristaltic pump. Tubing is run from the pump to the desired dye injection point. Contaminated pump lines are first flushed with stream water and then placed in plastic bags for shipment back to the lab.

5. COLLECTION OF WATER SAMPLES

Samples should be taken in pre-numbered glass bottles by a hand sampler or by ISCO samplers. Care should be taken to collect samples in the peak concentration of the dye cloud, i.e. do not sample midstream if the dye cloud is along one stream bank.

Dye Sample Collection

Data recorded on field sheets

a. Station Location-Sampling Point
b. Date
c. Sample Bottle Number
d. Time
e. Name of Sampler

Methods and Guidance

a. At least one background sample is needed for measurement of background fluorescence at each site in the study reach before the dye arrives.
b. Sampling should begin early enough to determine the true dye peak.
c. Sampling should continue until a peak has been determined; and until a decreasing trend has been clearly established.

Sampling Schedule

a. The schedule for collecting samples at each sampling site is the most uncertain aspect of the plan.

b. Estimates of the time to begin sampling, time intervals between samples, and the duration of sampling must be made, which will ensure adequate definition of the dye cloud passing each site. It is better to start with more

frequent sampling and decrease frequency based on sampling results when travel times are unknown.

c. An estimate of the time-of-travel between sampling sites is usually based on the cloud's movement to the first sampling site downstream of the injection site.

WATER QUALITY SECTION SUB-BASIN: ___________

INTENSIVE SURVEY

DYE TRACER STUDIES

STREAM: ______________________________ LOCATION: __________________________________

SAMP. NO	SAMP Point	DATE	TIME	G. H. OR RP-WS	FIELD READINGS				LABORATORY READINGS				CONC (ppb)
					1X	3X	10X	30X	1X	3X	10X	30X	

COMMENTS:

Figure 41. Dye Tracer Study Field Sheet

Sample Collection Methods

Hand Sampling

a. Grab sample by hand dipping a bottle or by using a dye sampler.
b. Depth integrated sample a Labline sampler is needed.
c. Specific depth sample – a Van Doran bottle may be used.

Automatic Samples (ISCO Sampler)

a. Samples can be analyzed directly from the ISCO bottles, however, if the ISCO bottles are needed to continue sampling the samples can be transferred to numbered glass bottles.
b. Label bottle racks.
c. To reuse ISCO bottle, rinse three times using tap water or if tap water is unavailable uncontaminated stream water (collected upstream of the injection site) can be used.

6: FLUOROMETER OPERATION AND PROCEDURES

Purpose: To outline the proper use, calibration, and sample analysis procedures for the Turner Designs Model 10 Fluorometer in dye tracing studies.

Scope: This SOP applies to all personnel using the Turner Designs Model 10 Fluorometer for measuring dye concentrations in field and laboratory settings.

Reference: Fluorometric Procedures for Dye Tracing, Book 3, Chapter A12, Revised 1986, U.S. Geological Survey.

Equipment: Turner Designs Model 10 Fluorometer

Fluorometer Scales and Sensitivity: The fluorometer has two main scales: X1 and X100. The X100 scale is 100 times more sensitive than the X1 scale. The sensitivity is further adjusted by range settings, as shown below:

Scale	Range Setting	Concentration Range (ppb)
X100	**X10**	**0–1**
X100	**Minimum Sensitivity**	**1–10**
X1	**X10**	**10–100**
X1	**Minimum Sensitivity**	**100–1000**

For specific operating and service instructions, refer to the Turner Designs Model 10 Fluorometer manufacturer's manual.

Calibration

Preparation

- Prepare a range of dye standard solutions (e.g., 1 ppb, 10 ppb, 50 ppb, 100 ppb) following U.S. Geological Survey dye tracing procedures.
- Ensure the fluorometer is calibrated at 1 ppb, as preferred by the Department of Water Resources (DWR).

Calibration Frequency:

- Calibrate the fluorometer:
- Before field deployment.
- Prior to running samples in the field.
- Before analyzing samples in the laboratory.

Procedure:

- Turn on the fluorometer and allow it to stabilize for at least 10 minutes.
- Use a distilled water blank to zero the instrument.
- Run the prepared dye standards and adjust the fluorometer to ensure accurate readings across the desired concentration range.

Sample Collection and Analysis

Sample Preparation:

- Collect samples in clean, labeled sample bottles.
- Prepare dye standard solutions of known concentrations per U.S. Geological Survey dye tracing procedures.

Sample Analysis:

- Rinse the cuvette with distilled water, then with a small volume of the sample to be tested.
- Wipe the outside of the cuvette to remove moisture.
- Place the sample in the cuvette and insert it into the fluorometer.
- Select the appropriate scale and range setting based on the expected concentration (refer to the table above).
- Run the sample and record the measurement on the field data sheet.

Sample Storage:

- Retain samples for future analysis, particularly if peak concentrations are uncertain or require verification.

Notes:

- Ensure all personnel are trained on the fluorometer's operation and this SOP.
- Regularly inspect the fluorometer for maintenance needs as per the manufacturer's guidelines.
- Handle samples carefully to avoid contamination, which can affect measurement accuracy.

APPENDIX 11. Flow Measurements

1. INTRODUCTION

Stream-flow or discharge is defined as the volume rate of flow of the water including any sediment or other solids that may be mixed with it (Buchanan and Somers 1968). Stream-flow is usually expressed in cubic feet per second (cfs) and discharge flow in million gallons per day (MGD). Several methods of determining flow are used by DWR. Most consist of wading into the stream with a top-setting flow rod and a vertical axis type flow meter shown in Figure 42.

Other methods of determining flows (usually small, low-velocity flows) are:

- Volumetric method
- V-notched weir method
- Estimating flow mathematically method

The USGS maintains many gauging stations across the state and their stream-flow information is available online. Discharge measurements using current meters are based on the equation:

Q=AV, where Q=Discharge, A=Area, V=Velocity

How to Calculate Flow

Calculating discharge from each of the width intervals:

$q_2 = v_2 d_2 (w_3 - w_1)/2$

where: q_2 = *discharge at width interval 2 (cfs)*
v_2 = *velocity measure at width interval 2 (ft/sec)*
d_2 = *depth at interval 2 (ft)*
w_3 = *distance from the bank or initial measuring point to the point following interval 2 (ft)*
w_1 = *distance from the bank or initial measuring point to the point preceding interval 2 (ft)*

Calculate the total discharge (flow) as the sum of each of the partial discharges:

$Q = q_1 + q_2 + q_3 + q_4 \ldots q_i$

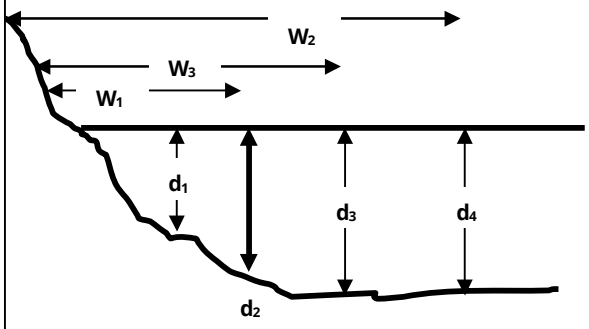


Figure 42. Calculating Stream Flow

Depth-measuring devices used by DWR include wading rods and a cable-winch bridge board. The top-setting wading rod easily sets the current meter at the proper height. The bridge board is used from a bridge handrail or the gunwale of a boat in streams and rivers where the water is too deep or the current is so strong that wading is dangerous or impossible. The winch has a depth indicating gage.

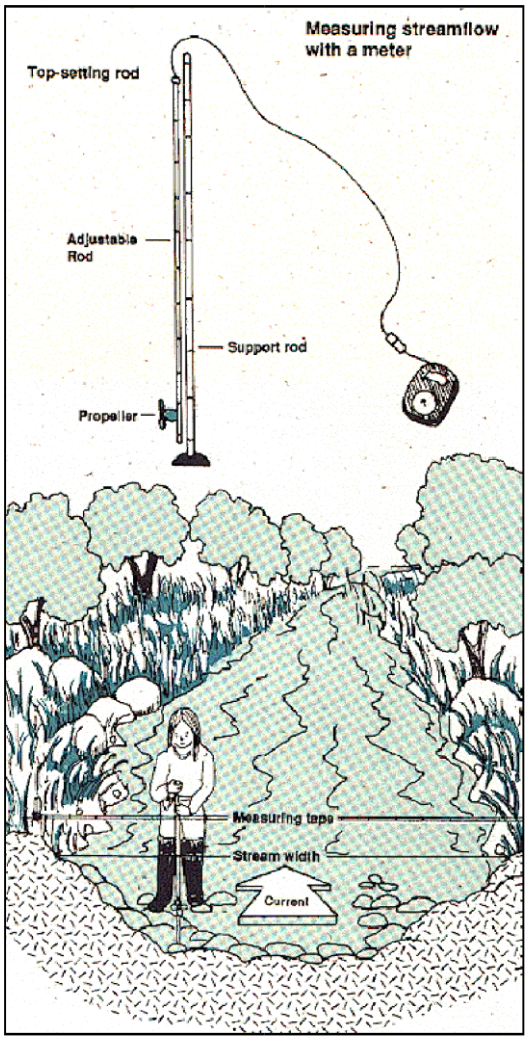


Figure 43. Instream Flow Measurement

2. ESTABLISHING AND USING A REFERENCE POINT

Before measuring any flow, a stage reference point (RP) should be found or established. This point can be located on any stationary object over the water surface. Measure the distance from that point to the water surface. It is important to measure this distance before and after doing the flow measurement, as this will indicate any changes in the water level that occurred during the flow measurement.

Many bridges already have an established RP located somewhere on the bridge. These RPs have been established by the USGS and should be used if they can be located. If the RP on the bridge is not located, establish one. Make a mark on a structure over a deep pool near where the flow measurement is made. Clearly identify the mark so that others may use it also.

If many flows are to be done at a station, then a good reference point needs to be used. Each time a flow measurement is performed, record the tape down elevation. As multiple flows are compiled, a relationship between stage and flow can be graphed; eventually allowing flow estimates to be made by measuring the reference point. Occasional flows still need to be performed to make sure the tape down/flow relationship remains constant.

Use a metal tape and dimwap (weight) when measuring the tape down. Stand at the reference point. Let the tape feed out until it barely skims the water surface. Put the tape to the reference point and record the measurement. Be sure to add the length of the dimwap in the measurement.

The reference elevation should be measured to the top of the bolt, the top of the nail head, or to the top of the bridge rail (even if it is beveled). It is important to make your measurements from the exact same point each time.

3. FLOW EQUIPMENT

Price AA current meter
Price pygmy meter
Top-setting wading rod
Headset or beeper box
Stopwatch
100 foot tape measure (in 1/10 ft)
Chaining pins
Stage tape measure with dingwap
Clipboard
Flow sheets
Pencil
Cleaning cloth
Oil
Flagging
Small flathead screwdriver
Hammer and nail
Spray paint (international orange)
Torpedo weights (15 lb., 30 lb.)
Bridge board
Hand calculator

4. FLOW MEASUREMENT PROCEDURE

Flow Measurement Method

Pre-Sampling:

a. Maintain all flow equipment in good working order. Refer to USGS publication, Discharge Measurements at Gauging Stations (Buchanan and Somers 1969).

   Note: The spin test is a good indicator of flow equipment readiness.

a. Check condition of flow equipment before leaving the office. An equipment checklist is helpful.
b. Gather all equipment necessary to do the flow, see list in section 3 of this appendix.
c. Select a reach of stream containing the following characteristics:
   - A straight reach with the threads of velocity parallel to each other
   - A stable stream bed free of large rocks, weeds, and protruding obstructions such as piers, which would create turbulence
   - A flat stream bed profile to eliminate vertical components of velocity.

- Wait 15 minutes after moving rocks and vegetation prior to beginning flow measurements to allow stabilization of the stream flow.

d. Establish a stage reference point (RP) (procedure described in following pages).

e. Measure and record the starting stage.

f. Install the current meter on the wading rod, attach the headphones - try a spin test (USGS publication, Smoot & Novak, 1968). Do the headphones click once for every revolution of the current meter? Adjust as necessary.

g. Record pertinent information on the flow sheet. Include: stream name, date, time start, stage start, location of RP, person doing flow, person recording information, stream conditions.

h. Determine the width of the stream. String a measuring tape across the stream perpendicular to the direction of the flow. Secure the tape on each bank with the chaining pins.

i. Determine how many measurements are necessary to give an accurate total discharge. Measuring velocity at 20-30 equidistant points across the width of the stream is recommended. More measurement points should be chosen in areas of significant depth or velocity change. Example: More measurements should be made in the area where the flow hugs one stream bank. A rule of thumb is that no area (point) being measured should contain more than 5% of the total flow of the stream.

j. Looking upstream, record from which bank (right or left) the measurements are starting. Record the location on the tape of the starting bank. Example: The left bank starts at 1.5 feet on the tape.

Sampling Techniques

a. Stand downstream and to the side so as not to obstruct the flow of the water to meter.

b. Record the tape measure reading of the point.

c. Measure the depth by placing the wading rod in the stream so that the base plate rests on the streambed. The depth is read from the graduated main rod and is estimated to the hundredth of a foot. Record depth readings on the flow sheet.

d. Use the upper scale of the top setting rod to set the depth of the current meter. At depths of 1.5 feet and less, the average velocity is best measured

at a point 0.6 of the depth from the water surface. Using the scale at the top of the wading rod automatically sets the current meter at the desired depth. To set the depth, press the rubber button on the flow rod. This releases the smaller rod. Move the smaller rod until the foot mark on it matches the appropriate tenth marker on the zero to ten scale at the top of the larger rod.

e. At depths greater than 1.5 feet, measure the velocity at 0.2 and 0.8 of the water depth (the average of these two velocities will later be recorded as a single value). The topsetting flow rod makes setting these two depths easy. Adjust the top scale readings to one half of the actual depth (this is the 0.8 reading) and to double the actual depth for the 0.2 reading.

f. Start the stopwatch and count the number of revolutions (clicks on the headphones) for at least 40 seconds. Start the stopwatch on count number 0 and stop the watch exactly on a count, not a certain number of seconds.

g. The pygmy meter is rated so that one revolution per second equals to one fps velocity. The Price meter rating is found in the top of the meter box. To use the Price meter table - count a certain number of revolutions: 1, 3, 5, 7, 10, 15, 20, 25, 30, 40 & 50. Compare to the time interval and read the velocity from the rating table. The Price meter also has a connection for measuring very high velocities where a signal is emitted from the meter for every 5 rotations instead of every single rotation.

h. Record the number of revolutions and the time (to the nearest second) on the flow sheet.

i. Move to the next point and repeat steps “a”-“f “until velocities in at least 20 cross sections have been measured.

j. After the final measurement has been made, record the tape reading of the finishing bank.

k. Measure and record the ending stage.

l. Record the finishing clock time.

m. Replace the current meter pivot with the traveling pin and return meter to box to prevent damage while traveling.

5. BRIDGE BOARD METHOD

Bridge Board Sampling Techniques and Supplies

Equipment Needs

- Clipboard with flow sheet and pencil
- Measuring tapes
- Duct tape
- Stage tape with weight
- Traffic safety cones and vests
- Bridge board assembly
- Price AA current meter (with tailpiece)
- Torpedo sounding weights (15 lb., 30 lb.)
- Headphones
- Stopwatch

Bridge Pre-Sampling Setup

(Refer to Buchanan & Somers, 1969, USGS publication)

a. Assemble bridge board equipment. This involves attaching the assembled Price AA current meter and the appropriate torpedo sounding weight to the hanger end of the winch cable. The current meter's position on the hanger is dependent upon which torpedo weight is used.
b. Attach the headphone jacks to the output terminals of the winch.
c. Do a spin test to ensure that current meter is working properly.
d. Determine the width of the stream and secure the measuring tape to the upstream handrail of the bridge with duct tape. More than one 100-foot tape may be necessary.
e. Determine the distance interval of the 20-30 points necessary to make an accurate measurement. Example: Measure velocity every 2 feet on a 50-foot wide stream. Be ready to change the interval if velocity or depth changes significantly. Remember, no more than 5% of the flow in any one interval.
f. Fill out flow sheet information (refer to step in section 4.1.1- “h”) of the current meter procedure).

Bridge Sampling Techniques

a. Measure the tape down from the reference point.

b. Record the tape reading from the starting stream bank.
c. Move to the first point to measure velocity. The bridge board rests on the handrail (guardrail) of the bridge.
d. Zero the winch depth indicator by lowering the current meter until the cups of the meter are half in the water. Pull the zeroing armature out and rotate until the depth indicator reads zero. Because most bridge handrails are not level, make sure to zero the winch depth indicator at each point that a velocity measurement is made.
e. Measure the depth of the water. To do this, lower the current meter until the cable goes slack. This indicates that the torpedo weight has hit something. Raise and lower the meter a couple of times to get a consistent depth reading for the bottom.
f. Add 0.5 foot to the reading to get the actual depth of the water. This accounts for the torpedo weight that hangs 0.5 foot below the current meter (which was zeroed at the cups). Record the actual depth.
g. At depths less than 2.5 feet, measure the velocity at 0.6 of the water depth (measured from the surface).
h. At depths greater than 2.5 feet, measure the velocities at 0.2 and 0.8 of the water depth.
i. Lower the current meter to the calculated depth.
j. Measure the velocity by counting clicks (revolutions) for at least 40 seconds. Refer to velocity measurements using the Price AA current meter.
k. Move to the next point and repeat steps “d”-“k”.
l. After the last velocity is measured, record the measurement of the finishing stream bank.

m. Record the finish time.
n. Measure and record the ending stage.
o. Store all flow equipment properly.
p. Compute the total discharge.

6. BOAT FLOW MEASUREMENT METHOD

Due to the danger because of boat traffic, extreme care should be taken when setting up for boat measurements. Locations where boat traffic is minimal should be chosen and any boats in the area should be warned off.

Boat Flow Sampling Techniques and Supplies

Equipment Needs

- Boat
- Rope (> width of stream)
- Bridge board assembly
- Headphones
- Stopwatch
- Clipboard with flow sheet and pencil
- Hand calculator
- Measuring tape - 100 foot
- Duct tape
- Stage tape with weight
- Cross piece assembly

Boat Flow Pre-Sampling Setup

(Refer to Buchanan & Somers, 1969, USGS publication Stations for more detailed instructions)

a. Assemble bridge board equipment.
b. Make a spin test.
c. Prepare the boat for work. It takes a minimum of two people in the boat, one to operate the bridge board and one to calculate the meter depths and record the flow information. The cross piece attaches to the bow and holds the boat in position on the rope.
d. Stretch and secure the rope across the stream channel, just over the water surface and perpendicular to the flow direction. Use duct tape to attach the measuring tape to the rope (an alternative is to use a rope marked at regular intervals).
e. Fill out flow sheet information.
f. Determine the interval of sampling points.

Boat Flow Sampling Techniques

a. Record the tape measure reading at the starting stream bank.
b. Measure the velocity at first point using the following steps.

The bridge board rests on the gunwale of the boat.

- Zero the winch depth indicator.
- Lower the current meter until slack in the cable indicates the bottom.
- Add 0.5 foot to the reading (to correct for the weight that hangs 0.5 foot below the meter).
- Calculate the depths to set the current meter at 0.2, 0.8 (see step section in 4.1.1- “h”) of current meter procedure).
- Set the depth indicator at the proper depth.
- Record the number of revolutions and the time interval at each depth.
- Move boat to next point.
- Repeat the steps under 6.9 until the entire stream has been measured.

c. Record the tape measure reading of the finishing stream bank.

d. Record the finish time.

e. Compute the total discharge.

7. V-NOTCH WEIR METHOD

V-Notch Sampling Techniques and Supplies

Equipment Needs

- V-notch weir (> stream channel width)
- Vertical staff gage
- Carpenter's level
- Hammers (3 lb., 8 lb. sledge)
- Straight edge
- Graduated container
- Stopwatch

V-Notch Flow Pre-Sampling Setup

a. Determine a good location for the weir plate. Avoid hard rock or loose sandy stream bottoms. Also avoid riffle areas where faster velocities erode the weir plate.

b. Set up the vertical staff gage. The gage should be located in the upstream pool formed behind the weir. Use the carpenter's level to make sure that all faces of the gage are level. Because of the effect of drawdown, the gage should not be located too close to the weir plate.

c. Use the sledgehammer to pound the weir plate into both banks so that the plate dams up all flow in the channel. Use the carpenter's level on all faces of the plate, it must be level. Make sure also that there is no water flowing around or under the weir plate.

V-Notch Flow Sampling Procedures

a. Determine the zero point of the weir (very important reading):

- Use the straight edge and level to measure a level line from the base of the angle on the weir plate back to the staff gage. Or if the distance from the top of the weir plate to the base of the angle is predetermined; then a level line is measured from the top of the plate to the gage and the distance subtracted.
- Read the water level on the staff gage just at the point where the water starts to flow through the notch in the plate.
- Let the water flowing through the V-notch stabilize.

b. Read the staff gage.

c. Determine the difference between the zero point on the staff gage and the water level flowing through the weir plate (read as the water level on the staff gage). This difference is known as head.
d. Look at the flow table for the V-notch weir. Look up the corresponding flow for the particular head height. An alternative to using the flow table is the volumetric method (procedure described in following pages).
e. Periodically clean the weir to prevent the buildup of sediments or solids around the notch. This buildup will affect the accuracy of the weir. Leaves are a problem to a V-notch weir. A leaf stuck at the base of the weir angle can cause a significant rise in water level.

8. VOLUMETRIC METHOD

Volumetric Sampling Techniques and Supplies

Equipment Needs

- Graduated container
- Stopwatch

Volumetric Flow Procedure.

a. Mark the container to a known volume (examples: 1 gallon, 1 liter).
b. Place the container under the discharge, collecting all flow.
c. Time the interval needed to fill container to the volume mark.
d. Empty the container.
e. Repeat steps “b”-“d” several times.
f. Average timed results.
g. Calculate the flow rate as flow volume/time. Example: 1 gallon in 15 seconds.
h. Convert flow to cfs or MGD.

9. FLOW SHEET CALCULATIONS

Data Form

Data to be Recorded on Data Sheet

- Distance from the initial point
- Depth
- Time (in seconds)
- Revolution

Calculations

a. Columns titled, velocity (mean in vertical), area, width, and discharge are calculated values.
b. Calculate the width of each cross section. The width of the section is the sum of one-half the distance from the point of measurement to each adjacent point. Example: In the first section the width is one-half the distance to the adjacent point plus one half the distance to the stream bank.
c. Calculate the velocity in each cross section. In depths of greater than 2.5 feet, two velocity measurements are taken in each cross section (0.2, 0.8). Average the two readings and record in column. This depends on the current meter in use:
    - The Price pygmy meter - the number of revolutions divided by the seconds.
    - The Price AA meter - The velocity is taken directly from the meter rating table. Not all Price AA meters use the same rating table-be sure that you have the correct table for the meter you are using.
d. Calculate the cross-sectional area. Multiply the width times the depth.
e. Calculate the discharge of each cross section. Multiply the cross-sectional area by its velocity.
f. Calculate the total discharge of the stream. Add all the cross-sectional discharges together.
g. Record the average velocity of the stream. Divide the total discharge by the total area.

11. OPEN CHANNEL FLOW MEASUREMENT METHOD

Introduction

The following section provides a brief overview of methods for determining flow in an open channel. For more detailed information regarding open channel flow measurements, refer to the references section.

Open channel flow can be defined as flow in any channel in which liquid flows with a free surface. Open channels are generally used in moving fluids at most municipal treatment facilities, industrial waste treatment operations and in most irrigation applications. An open channel can also be a stream or a ditch. Open channel flow is typically measured by the use of a calibrated restriction device placed in the channel that affects the surface level of the liquid as it moves past the restriction. This type of open channel measuring device is referred to as a "primary" device. The known dimensions and physical characteristics of the restriction device are used to correlate a relationship between water surface level and flow. After the water level/flow relationship has been established, the flow in the open channel can be easily measured by manually sighting the height of the liquid's surface level against a calibrated scale (staff) and then referring to the appropriate rating curve or table.

The following are the most commonly used types of "primary" open channel flow measuring devices, (restriction devices):

Weir: a dam constructed across an open channel, over which liquid flows through an opening or notch. The most commonly used types are rectangular, trapezoidal and triangular.

Flume: a specially shaped open channel, designed to change the channel area or slope, resulting in an increase velocity and surface level of the liquid flowing through it. The most commonly used types are: Parshall and Palmer-Bowlus

Flow Meter

a. A flow meter is a mechanical device used to measure the liquid level in the channel and convert the level into a corresponding flow rate
b. A stage recorder is a mechanical device used to record the surface level of the liquid over a period of time

Note: Measuring flow in an open channel by means of a weir or flume is a simple function of surface level and is the most basic and inexpensive method available. However, if continuous stage or flow recording is required, then the use of a stage recorder and/or a flow meter in conjunction with the primary device may be necessary. Some of the more commonly used methods employed by these devices to determine the surface level of a liquid are floats, dipping probes, ultrasonic sensors, and bubblers.

APPENDIX 12: Sediment Oxygen Demand

1. GENERAL DESCRIPTION OF SOD TEST

Sediment Oxygen Demand (SOD) is one of the more significant variables in water quality modeling evaluations for determining stream assimilative capacity. SOD data are primarily used for waste-load allocation purposes in the evaluation of receiving waters.

The SOD test involves placing an SOD chamber on the bottom sediment, securing it to prevent water infiltration and monitoring oxygen change within the chamber. A dissolved oxygen sensor inside the chamber measures the rate of decrease in oxygen that is used by organic materials in the bottom sediments over a given period of time. A standard SOD test includes seven SOD chambers of which two are water column control (blank) chambers and five are replicate SOD chambers (Figure 43). The blank chambers, used to determine water column respiration rate, have bottom plates that prevent bottom sediment from contacting the water in the chamber. The SOD replicate chambers have open bottoms allowing the internal water to circulate over the bottom sediment. The rate of oxygen change in the replicate SOD chambers minus the water column respiration of the blank chambers equals the SOD rate.

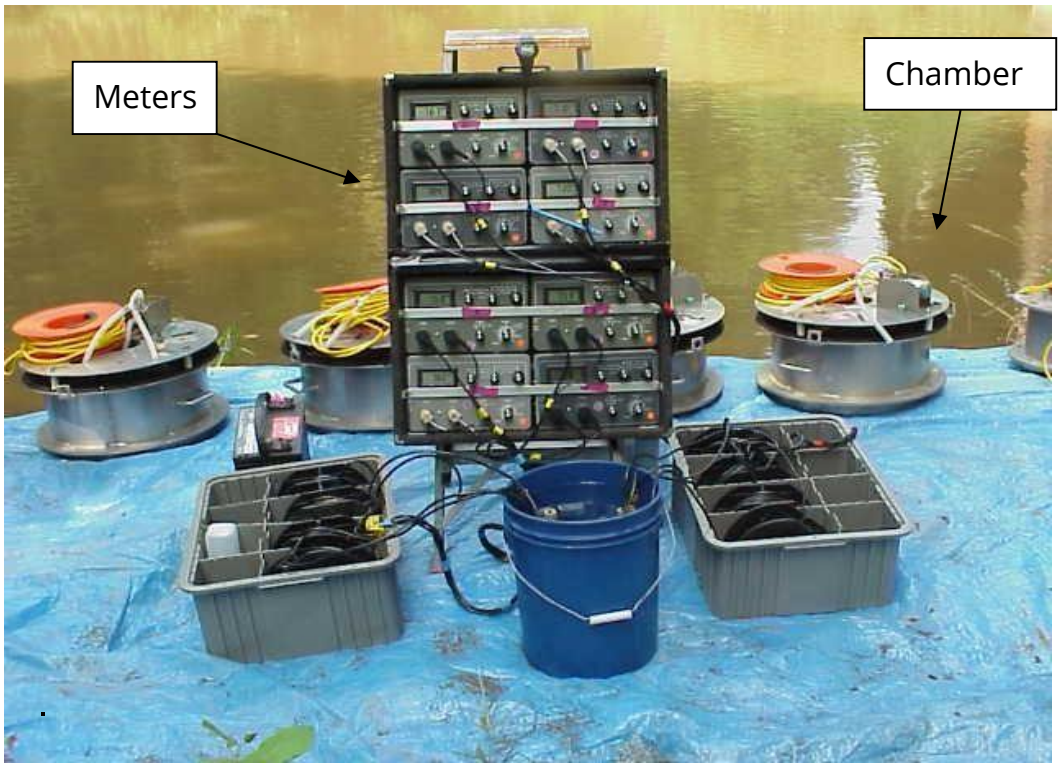


Figure 44. SOD Equipment

SOD Rate Formula:

The SOD rate for any study location is then calculated by using the SOD rate formula:

ß x (K x V) ÷ A = gr $0_2/m^2/hr.$ where: ß = rate of

change in D.O. as mg 0_2/L/min.

V = chamber volume in liters

A = chamber area in meters square

K = 0.06 (constant) converts liters to square meters

SOD rates are dependent on benthic metabolic processes, sediment particle size, stream velocity and other factors. SOD rates from 77 in-situ tests performed at locations with various substrate compositions are presented in Sediment Oxygen Demand, Processes, Modeling and Measurement (Murphy and Hicks, 1986, p. 318). An example SOD Excel Worksheet is provided at the end of this section for reference.

SOD Equipment List – Due to the amount of gear and equipment necessary to successfully complete SOD tests, a checklist is recommended when preparing for testing.

Site Evaluation – Each site should be visited and checked out to determine if sediment is suitable in the area under investigation. Figure 45 is the SOD Site Evaluation Form that should be completed for each location.

2. FIELD CALIBRATION DISSOLVED OXYGEN METERS

An initial calibration is performed on the YSI 58 meters prior to the SOD test and a terminal calibration is performed on the meters after the test is completed. All calibration data is recorded on the SOD Calibration Forms (Figure 46). The need for accuracy is paramount for SOD evaluations due to the extremely small increments of change in D.O. measured during the test (+/- 0.01 mg/L). Because of the number of meters being calibrated on site and the extreme accuracy required for SOD testing, initial and terminal calibration procedures in this section vary from other D.O. meter calibration methods in this document. SOD meters are calibrated using the Modified Winkler Azide method as opposed to saturated air calibration methods.

SOD Equipment List

CHAMBERS:
___ FIVE REP CHAMBERS ALUM
___ TWO BLANK CHAMBERS ALUM
___ ONE CLEAR BLANK CHAMBER
___ TUBES ON CHAMBERS
___ FLOW RESTRICTORS IN TUBES
___ SPACERS ON CHAMBER
___ BATTERY CLIPS ON DC LEADS
___ RUBBER SEALS OK
___ SILICON SEALS OK
___ TEST PUMPS
___ CHAMBER COLLARS
___ 3 BATTERIES MINIMUM (CHARGED)
___ CHAMBER HARNESS AND FLOATS
___ STOPPERS -#1 & #11½

METERS:
___ DO METERS
___ NEW MEMBRANES ON PROBES
___ CONDO METER
___ MEMBRANES & ELECTROLITE KIT
___ 100′ CABLES WITH PROBES
___ EXTRA 50′ CABLE AND PROBE
___ CALIBRATION & SOD FIELD SHEETS
___ COPPER BATTERY BARS
___ STAND FOR METERS (BOAT OR BANK)
___ "C" CLAMPS (LARGE) - BOAT METER STAND
___ BUNGIES FOR METER STAND
___ BOARD FOR METER STAND MOUNT

WINKLER
___ WINKLER KIT (CHECKED OUT)
___ EXTRA CHEMICALS
___ BURET AND GLASSWARE
___ EXTRA BURET AND GLASSWARE
___ BUCKET FOR CALIBRATION
___ WATER CALIBRATION
____ BURET STAND
____ BURET WIRE
____ STARCH

PERSONAL EQUIPMENT
____ RAIN GEAR
____ BOOTS
____ WATCH
____ COOLER AND ICE
____ WASH WATER/SOAP
____ INSECT REPELLANT
____ SUN SCREEN
____ HAT
____ SUN GLASSES
____ FOOD/DRINKS/WATER

MISC. EQUIPMENT
____ CAMERA AND ACCESSORIES
____ SEDIMENT JARS AND TAGS
____ SOD TOOL BOX
____ MAPS
____ CALCULATOR/PENCILS
____ TARPS
____ FIRST AID KIT
____ MACH, SHOVEL
____ CHAIRS, BOX
____ ROPES FOR BANK OR BOAT
____ CELL PHONE
____ COLORED TAPE
____ BATTERY TESTER
____ "C" CLAMPS (SMALL) FOR REP LIDS
____ FIELD LOG FOR SOD TEST
____ PLASTIC CRATES

SOD SITE EVALUATION

Site Location - _____

_____

Date: _____ Time: _____

Site Description - _____

_____

_____

_____

Topo Map # _____ % From Right Bank (facing US) _____

Weather - _____

_____

Bank Description - _____

_____

Depth - _____ Velocity (fps) - _____

Sediment Description - _____

_____

Bottom Topography - _____

Water Description (turbid, clear, etc.) _____

_____

Site Schematic:

Figure 45. SOD Site Evaluation Form

All meters are to be air calibrated prior to field operations (and on-site calibration) to assure all meters are functioning properly and stabilized.

The procedures are as follows:

Calibration Procedures

INITIAL CALIBRATION Method

a. Turn off all electronics (cellular phones, depth finders, etc.) prior to reading and calibrating meters.
b. Connect the D.O. probe to the probe receptacle of the YSI 58 meter and screw the retaining ring finger tight.
c. Connect the D.O. stirrer to the stirrer receptacle of the YSI 58 meter and screw the retaining ring finger tight. Check the stirrer battery condition by turning the stirrer switch to its spring-loaded battery check position. The warning LOBAT will indicate when approximately 5 hours of battery life remain.
d. Zero the instrument. Set the function switch to ZERO and adjust the display to read 0.00 with the O2 ZERO control.
e. Switch to the 0.01 mg/l position and wait at least 60 minutes for the probe to polarize. Allowing additional time to repolarize the probe is necessary whenever the meter has been turned off or the probe has been disconnected.
f. After the 60 minute wait, turn the function switch to ZERO and readjust the O2 ZERO control to 0.00 if necessary. The meter is now ready to calibrate.
g. Calibration - Meters are calibrated using the Winkler azide method as described in this SOP in Section III.
h. The D.O. probes are placed in a container of tap water with a relatively stable temperature. A minimum of four Winkler tests are then performed on the tap water. Three of the four resulting Winkler values must be within a 0.1 mg/l range. If the values are not in the 0.1 mg/l range, the Winkler tests should be repeated until the values are within the + or - 0.1 range. The three Winkler values are then averaged to provide an initial calibration value.

i. After the probes have stabilized in the container of tap water, the function switch is set to 0.01 mg/l, the meters are then adjusted to the initial calibration value by turning the O2 CALIB control. The meters are now calibrated.

j. Leave the instrument on throughout the test to avoid repolarizing the probe. Reactivate the stirrer approximately 2 minutes before each reading and turned off after the reading.

k. Obtain a bottom salinity reading using a YSI Model 33 S-C-T Meter. If salinity is present, the SALINITY knob on the YSI Model 58 D.O. Meter is adjusted accordingly.

l. Upon completion of the SOD test, perform a terminal calibration on all YSI 58 D.O. meters used. All terminal calibration data is recorded on the SOD terminal calibration form (Figure 46).

Note: If SOD tests are performed in coastal areas where tidal influence may cause salinity values to fluctuate during the test, salinity readings should be taken frequently and salinity adjustments made to the YSI 58 D.O. meters.

TERMINAL CALIBRATION Method

a. The D.O. probes are placed in a container of tap water with a relatively stable temperature and allowed to stabilize. A minimum of 4 Winkler tests are then performed on the tap water. The resulting Winkler values must be within a 0.1 mg/l range. If three of the four values are not in the 0.1 mg/l range, the Winkler tests should be repeated until the values are within the range. The Winkler values are then averaged to provide a terminal calibration value.

b. After the Winkler bottles have been filled with the tap water, turn on the stirrers, wait one minute and then record the D.O. and temperature readings.

c. Each D.O. reading should be within a 0.1 mg/l range from the average Winkler calibration value.

3. QUALITY ASSURANCE

Procedure

a. Complete the Pre-Sampling Calibration, Post-Sampling Calibration Check, and SOD Worksheets on-site during each SOD test.

b. Perform Winkler tests per this SOP – Section III – azide modification.

c. Perform a minimum of three Winkler titrations for Initial Calibration and Terminal Calibration.

d. Winkler values must be within a 0.1 mg/l range. If any value is outside the 0.1 mg/l range, then additional Winkler tests are performed until the values are within the range.

e. The terminal YSI 58 D.O. Meter reading should be within a 0.1 mg/l range from the average terminal Winkler calibration value.

f. A minimum ambient bottom D.O. of 2.0 mg/l is required to perform an SOD test (Murphy and Hicks, 1986).

g. Chamber velocities must be in a 0.08 to 0.12 ft/sec. range (Howard, 1988).

h. Take detailed field notes during the SOD test including a site description.

i. Conduct a pre-check to each SOD study to provide information on the study feasibility and station characteristics. During the -check sediment samples are generally collected to determine bottom characteristics.

ALL METERS ZERO PRIOR TO CALIBRATION (YES NO)	CALIBRATION NOTES:
MEMBRANES VISUALLY CHECKED PRIOR TO CALIBRATION (YES NO)	
MEMBRANES LAST REPLACED	
BATTERIES LAST REPLACED	
CALIBRATOIN METHOD (SATURATED AIR WINKLER)	
CALIBRATION PERFORMED BY	
SALINITY	

INITIAL CALIBRATION

TIME OF INITIAL CALIBRATION
WINKLER READINGS: (A) (B) (C)
(AVERAGE)

METER	AMB	BLANK O	BLANK OO	CLEAR	1	2	3	4	5	A	B	C
READINGS BEFORE CAL												
WINKLER												
DIFFERENCE												
ADJUSTED												

TERMINAL CALIBRATION

TIME OF INITIAL CALIBRATION
WINKLER READINGS: (A) (B) (C)
(AVERAGE)

METER	AMB	BLANK O	BLANK OO	CLEAR	1	2	3	4	5	A	B	C
READINGS BEFORE CAL												
WINKLER												
DIFFERENCE												

Figure 46. Sediment Oxygen Demand Calibration Worksheet

4. CHAMBER DEPLOYMENT

After the D.O. meter calibration procedure is complete, the SOD chambers are prepared for the test. All chambers are prepared as follows prior to being placed into the water (Lawhorn, 1988).

Setting up Chambers

Chamber Preparation

a. Place the lids on replicate chambers in the up position with spacers located between the lid and lid companion ring. Wing nuts should be tight enough to hold the spacers in place but not so tight as to hamper removal after the chamber has been set in place on the bottom.
b. Insert the water sampling port stoppers on each chamber lid (size #1, two on each lid).
c. Open the monitoring probe port on all chamber lids (no stoppers).
d. Inspect the replicate chamber lid gaskets for damage or debris that could prevent a watertight seal.
e. Inspect the seals on the blank chambers for damage.
f. Clip the harness ropes to each chamber.
g. Open the water intake ports located on the bottom of the blank chambers (no stoppers).
h. Disconnect the return pump tubing from the chamber lid male connectors.

Boat operation only:

a. Hang the chambers in sequential order along the gunwale with the chamber lids several inches below the surface of the water.
b. Tie the chamber harness to a gunwale cleat.
c. Situate the boat over the bottom where the chambers will be placed.
d. Do not allow the chambers to disturb the bottom sediment.

Land operation:

Chambers are placed on the stream bank in the order that they will be deployed. This will prevent harness ropes, pump cables and probe cables from becoming tangled during the chamber deployment and the SOD test.

Chamber deployment:

a. Blank chambers are deployed first because sufficient time is required to replace surface water trapped inside the chamber with ambient bottom water prior to initiating the SOD test.
b. When deploying blank chambers in soft sediment, place the chambers in an area away from the area that the replicate chambers will be deployed in order to avoid stirred up sediments from being drawn into the chamber through the open probe port.
c. One clear polycarbonate and acrylic blank chamber is used in addition to the conventional aluminum blank chambers to provide an indication of whether or not photosynthesis is occurring in the water column. The mechanical functions and the deployment procedure for the clear blank chamber are identical to that of the aluminum blank chambers. In cases of high flow a weighted band should be placed around the clear chamber to prevent it from being washed away.
d. Each blank chamber must be filled with surface water that enters through the two filling ports located on the bottom plate of the chamber.
e. After the blank chamber is filled at the surface and prior to lowering the chamber, two #11½ stoppers must be inserted into the filling ports. Surface water is used to fill the blank chamber to create negative buoyancy so the chamber can be lowered to the bottom.
f. After the filling port stoppers are in place, the chamber is agitated to dislodge any air that is trapped under the lid. The trapped air will exit through the probe port.
g. The chamber is then lowered to the bottom.
h. When the blank chamber is on the bottom, the pump is turned on. Unlike the replicate chambers, the lid and bottom of the blank chambers are permanently sealed thus no water exchange occurs when the chamber is lowered to the bottom. Surface water must be purged from the chambers by operating the pump with the tubing disconnected from the male adapters on the lid while the chamber is on the bottom. Bottom water is drawn into the chamber through the open probe port while the surface water is purged through the disconnected return tubing. With the two return pump tubes disconnected, the chamber will purge surface water and draw in bottom water.

i. A light tapping on the pump housing and tubing will dislodge air bubbles trapped in the pump system.

j. The pump is then allowed to run while the other chambers are being deployed.

k. This procedure is repeated for each blank chamber.

Replicate Chamber Deployment

a. After the blank chambers are deployed, each replicate chamber is slowly lowered to the bottom substrate prior to setting the chamber.

b. Set the replicate chambers out in downstream to upstream order to prevent sediment disturbance and any silt that may have been disturbed from settling on areas where other chambers will be placed.

c. If the chamber location is unsatisfactory because of debris, or other bottom characteristics that would prevent the chamber from sealing then the chamber is carefully relocated. In addition, if the chamber location is atypical of the general stream area, the chamber or possibly the station should be relocated.

d. After the replicate chamber is placed in a satisfactory location on the bottom, carefully examine the sediment/flange seal and the sediment/inner core seal to assure that ambient water infiltration will not occur during the test.

e. The replicate chamber lid is then lowered by loosening the four wing nuts and removing the PVC spacers. The lid must be lowered very slowly as not to create a pressure wave and stir up silt inside the chamber. If silting occurs in the chamber, initial D.O. readings will be erratic and a longer period will be required for SOD rate stabilization (see: Section 5. Procedure for Recording SOD Data).

f. Replace the spacers between the companion ring and stainless steel washers. The wing nuts are then tightened and the gasket forms a watertight seal.

g. Activate the pump and lightly tap the pump housing and tubing to dislodge air trapped in the pump system.

h. Turn off the pump and allow any silt that may have been suspended to resettle before starting the test.

i. Reconnect the return tubing.

j. Repeat this process until all replicate chambers are deployed.

Once all replicate chambers are in place, insert DO probes into the probe ports beginning with the first blank chamber deployed and ending with the final replicate chamber.

During the D.O. probe installation, replicate chamber pumps can be turned on and a final check of the chamber and pump tubing can be performed.

In addition to the D.O. probes located inside the SOD chambers, one D.O. probe is placed on the outside of a chamber to record ambient D.O. values.

When an SOD test has been completed, chambers can usually be lifted from the bottom using the harness ropes.

5. RECORDING SOD FIELD DATA.

After SOD Chambers and Probes are installed

Readings

a. Stirrers are activated approximately 2 minutes prior to reading meters and turned off after the data is recorded.

b. All meters (including the ambient meter) are read at 15 minute intervals. For each chamber, D.O., temperature, and the change in D.O. per 15 minute time period is recorded on the SOD field sheet form (Figure 47).

c. D.O. readings from the replicate chambers will usually decrease at a relatively similar rate. Typically, if relatively uniform decreases in D.O. are observed in the replicate chambers after stabilization, a sufficient SOD rate can be calculated from approximately 2 hours of testing (Murphy and Hicks, 1986).

d. A minimum oxygen reduction of 0.4 mg/l is required before an SOD test should be terminated. This situation is not typically encountered and would provide an extremely low SOD rate indicating little organic content in the sediment.

e. SOD tests with very slow oxygen uptake rates may be less reliable due to an extremely small amount of oxygen depletion over a greater period of time. Since longer tests are necessary when slow oxygen uptake is occurring, the potential for meter calibration drift increases.

f. See Figure 48 for an example of completed SOD worksheet.

Recording Errors

Erratic D.O. Readings Troubleshooting

If observed in replicate chambers the following are possible problems:

a. Initial D.O. readings may be erratic if sediment was disturbed during chamber placement on the bottom. This problem occurs often at stations where sediment consists of soft mud or a silt-like composition and is usually observed in all of the replicate chambers. For this reason, several of the initial D.O. readings may be omitted from the SOD rate calculations. The readings will usually stabilize as the suspended particles in the chamber settle out, (generally about 15 to 30 minutes, 1 to 2 readings).

b. If D.O. readings from all chambers do not stabilize after 30 minutes, it may indicate that the chambers are sinking into the soft sediment causing the circulation diffusers to become close to the sediment and continually disturbing the silt. If this occurs, the chambers must be reset on the bottom and a chamber collar must be placed around the bottom chamber flange to prevent the chambers from sinking. Chamber collars are flat, thin pieces of material, that increases the surface area of the chamber flange and prevent the chamber from sinking into soft sediment.

c. If D.O. readings from a replicate chamber do not stabilize and begin to decrease after the other chambers have stabilized, it may indicate that the chamber was not initially sealed and ambient bottom water is leaking into the chamber via the ports, gasket seal, pump tubes or the sediment flange seal. The chamber must be reset and the seal integrity reconfirmed.

d. On occasion, ambient water will begin leaking into a chamber. Chamber leaks (blowouts) are the most frequent problem encountered in SOD tests. This problem is easily recognized when D.O. values in a chamber that have been steadily decreasing suddenly begin to rise rapidly. However, if the chamber leak is small, the rate of decrease in D.O. may only be slowed, resulting in an unrealistically low rate for the chamber. For these reasons, the rate of D.O. change in each chamber must be

carefully evaluated and recorded for each 15 minute time period during the SOD test.

If a chamber leak is detected the following options may be considered:

- Stop the leak and restart the test for that chamber; or
- Delete the data from that chamber from the SOD test; or
- Terminate entire test, if sufficient data has been recorded to establish a reliable linear regression.

e. If the D.O. in a chamber falls much more rapidly than in the other chambers, it may indicate that the chamber has been inadvertently placed on organic debris such as decaying leaves or other organically rich deposits that may be uncharacteristic of the area. The chambers must be placed on sediment that is typical for the station area. If this problem is encountered, the chamber should be relocated or the data deleted from the SOD test.

   The validity of SOD test data is dependent on locating the test site at an area that is typical of the water body being studied. If the chamber location is atypical of the general stream area then the chamber or possibly the station should be relocated.

f. When other obvious D.O. or temperature problems occur during the SOD test, it is usually the result of meter or probe malfunction and can be detected by the terminal calibration results.

6. METER AND PROBE PREPARATION

Procedure

a. Check all D.O. meters, cables and probes to assure proper functioning **before** the survey.

b. Evaluate the YSI 58 instrument batteries and replaced if necessary. Stirrer batteries should be checked to assure that batteries are adequate to complete SOD test.

c. Replace all D.O. probe membranes prior to each SOD survey. After the membrane has been changed, a minimum of 24 hours should be allowed for the probe to equilibrate before it is used for an SOD test. YSI Standard Membranes should be used.

7. SOD CHAMBER VELOCITY TEST

SOD rates are directly related to the sediment/water interface velocity, therefore specific and consistent velocities must be maintained in all chambers for accurate SOD testing. EPA recommends a constant chamber velocity of 0.1 ft/sec and an acceptable range of 0.08 to 0.12 ft/sec (Howard 1988). To maintain this velocity range, DWR uses a flow restrictor placed in the chamber pump tubing to reduce pumping velocity. The restrictor is 1" long, made from brass stock, and has a 7/64" opening in the center to allow a desired velocity of water.

All SOD chambers are periodically tested in the lab to ensure that velocities remain constant after repeated field use and pump wear. Velocity tests are performed using a Marsh McBirney Magnetic Flow Meter Model 201. The Marsh McBirney meter is factory calibrated. Chamber velocity tests procedures are as follows:

Velocity test procedures for replicate chambers:

a. Insert a # 11½ stopper in the monitoring probe port and two # 1 stoppers in water sampling ports. All pump tubing should be connected and the chamber lid must be tight against the chamber companion ring.
b. Place the chamber upside-down on a support in a manner that will allow access to the monitoring probe port. The support should not touch the pump tubing or alter the pump flow in any manner. The chamber and support should be located over a sink or other acceptable area where the test water can be easily drained.
c. Fill the chamber with water to the cutting ring flange (normal water/sediment interface).
d. Turn the pump on. It may be necessary to add more water to fill pump and pump tubing after the pump is turned on and to tap the pump and tubing to dislodge trapped air. Place the Marsh McBirney probe 2 inches below the surface of the water halfway between the outer and inner chamber wall. Allow the water circulation in the chamber to reach the maximum velocity (approximately 15 minutes).
e. Read the Marsh McBirney Meter. The velocity in the chamber should be within a range of 0.08 to 0.12 ft/sec.
f. If the velocity is not constant or out of the acceptable range, check the following:

- Probe orientation or placement in the chamber.
- Restrictions in pump tubing (7/64" brass restrictor may be blocked).
- Air bubbles could be locking the pump or altering flow.
- Pump may be damaged and not pumping maximum flow. • Check pump battery voltage output (12 volt) • Check velocity meter calibration.

Velocity Tests for Blank Chambers:

a. Insert two # 11½ stoppers into the filling ports on the bottom of the blank chamber. All pump tubing should be connected.
b. Place chamber right side up on a support in a manner that will allow access to the filling ports.
c. Fill the chamber completely with water.
d. Turn the pump on . It will be necessary to add more water to fill pump and pump tubing after the pump is turned on and to tap the pump and tubing to dislodge trapped air. Place the Marsh McBirney probe through the D.O. probe monitoring port at a depth of 2 inches. Allow the water circulation in the chamber to reach the maximum velocity (approximately 15 minutes).
e. Read the Marsh McBirney Meter.
f. Use the same trouble shooting procedures as with the replicate chambers if problems are encountered.

8. LEAK TEST FOR SOD CHAMBERS

SOD chambers must remain watertight during the SOD test to prevent ambient bottom water from entering the chamber and invalidating the test. The exchange of ambient bottom water and chamber water can occur by two means, by leaking between the sediment and chamber cutting edge or by leaking through any of the normally sealed chamber gaskets, stoppers, fittings and tube connections. Chamber leaks at the sediment/chamber interface generally occur as a result of sediment or sand washing out from around the chamber due to scouring and are usually detected during the test. Leaks through chamber seals, other than the sediment/chamber interface can be detected during the Chamber Velocity Test (Section 7). Note: leak test is under worst case conditions because chamber water

(inside/outside) is equalized during the test. Procedures for leak testing SOD chambers are as follows:

1. Insert a # 11½ stopper in the monitoring probe port and # 1 stoppers in water sampling ports. All pump tubing should be connected and the chamber lid must be tight against the chamber companion ring.
2. Place the chamber upside-down on a support in a manner that will allow access to the monitoring probe port. The support should not touch the pump tubing or alter the pump flow in any manner. The chamber and support should be located over a sink or other acceptable area where the test water can be easily drained.
3. Fill the chamber with water to the cutting ring flange (normal water/sediment interface for replicate chambers and to bottom plate on blank chambers).
4. Turn the pump on.
5. If water leaks out, repair or replace the seal and repeat the leak test.

9. THREE POINT ANCHOR TECHNIQUE

If a boat operation is necessary to perform a SOD test, care must be taken to provide maximum stability and minimize wave action and horizontal swing over the bottom. Movement of the boat by wave action or swing on a single anchor line will result in chambers being lifted and the SOD test terminated. This problem can be avoided by using the following three-point anchor technique:

1. After the boat is on station, align the bow into the current.
2. Set bow anchor on SOD boat allowing a minimum scope of 3 times depth. More scope may be necessary if strong current or winds are present.
3. Use support boat to set aft port and aft starboard anchors (minimum scope 3 times depth).
4. Anchors should be oriented in a 3-point (tripod like) pattern with the SOD boat in the center.

5. After all anchors are set, the lines should be tightened as much as possible and cleated to provide maximum stability and minimize horizontal movement of the SOD boat.
6. While anchoring, care should be taken not to disturb the sediment where the SOD test is to be performed.
7. It is potentially dangerous to anchor with the stern of the boat facing upstream if current, waves or bad weather exists. The 3-point anchor method should not be used in areas affected by strong tidal current unless the test can be completed prior to the turning of the tide.

STUDY AREA											
STATION LOCATION											
DATE	SEDIMENT TYPE	DEPTH	CHAMBERS DIVER DEPLOYED? YES/NO	VELOCITY FT/SEC (at bottom)							
PERSON(S) READING METERS	STAFF ON SITE	BOAT SOD/BANK SOD									

TIME	MIN.	AMB DO TEMP	BLANK O DO TEMP	BLANK OO DO TEMP	BLANK CLEAR DO TEMP	REP 1 DO TEMP	REP 2 DO TEMP	REP 3 DO TEMP	REP 4 DO TEMP	REP 5 DO TEMP	BACK-UP DO TEMP
	START	|	|	|	|	|	|	|	|	|	|
	15	|	|	|	|	|	|	|	|	|	|
	30	|	|	|	|	|	|	|	|	|	|
	45	|	|	|	|	|	|	|	|	|	|
	60	|	|	|	|	|	|	|	|	|	|
	75	|	|	|	|	|	|	|	|	|	|
	90	|	|	|	|	|	|	|	|	|	|
	105	|	|	|	|	|	|	|	|	|	|
	120	|	|	|	|	|	|	|	|	|	|
	135	|	|	|	|	|	|	|	|	|	|
	150	|	|	|	|	|	|	|	|	|	|
	165	|	|	|	|	|	|	|	|	|	|
	180	|	|	|	|	|	|	|	|	|	|
	195	|	|	|	|	|	|	|	|	|	|
	210	|	|	|	|	|	|	|	|	|	|
	225	|	|	|	|	|	|	|	|	|	|
	240	|	|	|	|	|	|	|	|	|	|
	255	|	|	|	|	|	|	|	|	|	|
	270	|	|	|	|	|	|	|	|	|	|

TIME	MIN.	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO	Change in DO
	START										
	15										
	30										
	45										
	60										
	75										
	90										
	105										
	120										
	135										
	150										
	165										
	180										
	195										
	210										
	225										
	240										
	255										

Figure 47. SOD Field Sheet

Rocky River near Aquadale
STATION LOCATION: Rocky River at Hwy 52
SUB-BASIN 03 07 13 COUNTY - Stanley

STAFF ON SITE: QUIDLEY, WILLIAMS, FISHER, MEDLIN
DEPTH AT SITE: 2 feet
BOTTOM SEDIMENT: SAND

TEST CONDUCTED FROM RIGHT BANK
AVERAGE AMBIENT WATER TEMP 24.4°C
SOD TEST FOR SOD CALIBRATION

		BLANK CHAMBERS		REPLICATE CHAMBERS					
	AMB	BLANK 0	BLANK OO	REP 1	REP 2	REP 3	REP 4	REP 5	Extra Chamber
NOTES									not used
AVERAGE T°C	24.3	24.3	24.4	24.4	24.3	24.4	24.3	24.4	
D.O. Range	7.81 - 8.02	7.79 - 7.86	7.87 - 7.97	6.34 - 7.80	6.61 - 7.63	6.78 - 7.80	6.69 - 7.60	6.61 - 7.47	
Test Time in min.									
0	7.99	7.86	7.97	7.80	7.63	7.80	7.60	7.47	
15	8.02	7.85	7.96	7.63	7.49	7.68	7.48	7.35	
30	8.00	7.84	7.95	7.47	7.37	7.56	7.37	7.23	
45	7.94	7.83	7.94	7.30	7.24	7.45	7.26	7.10	
60	7.83	7.82	7.93	7.14	7.12	7.34	7.17	7.02	
75	7.81	7.82	7.93	6.98	7.02	7.23	7.07	6.95	
90	7.90	7.81	7.91	6.83	6.91	7.12	6.98	6.87	
105	7.91	7.80	7.89	6.67	6.81	7.01	6.88	6.77	
120	7.93	7.80	7.88	6.50	6.71	6.89	6.78	6.68	
135	7.90	7.79	7.87	6.34	6.61	6.78	6.69	6.61	
RATE OF WATER COL (ß) mg/L/min		-0.0005	-0.0007						
RATE OF REPLICATE (ß) mg/L/min				-0.0108	-0.0073	-0.0075	-0.0067	-0.0063	
		-0.0006	AVG. RATE OF WATER COL (ß) mg/L/min	-0.0006	-0.0006	-0.0006	-0.0006	-0.0006	
RATE CALCULATION			REPLICATE RATE (-) AVG WATER COL RATE mg/L/min	-0.0102	-0.0067	-0.0069	-0.0061	-0.0057	
ß x (K x V) ÷ A = gr O2/m2/hr			SOD RATE gr/m2/hr	-0.1113	-0.0734	-0.0756	-0.0664	-0.0620	
ß = Rate of change in D.O. as mg O2/L/min			gr/m2/day	-2.6713	-1.7624	-1.8147	-1.5937	-1.4884	
v = Chamber volume in liters									
A = Chamber area in meters square									
K = .06 (Constant)									

AVERAGE SEDIMENT OXYGEN DEMAND (rate calculated for 3 hours)

-0.0778 gr/m2/hr
-1.8661 gr/m2/day AT AMB. TEMP 24.4°C
-1.4145 CORRECTED TO 20°C (COEF 1.065)
-0.0072 gr/ft2/hr
-0.1734 gr/ft2/day

FIELD STAFF :

CALCULATIONS PERFORMED BY:
Harold Quidley

CALCULATIONS CHECKED BY:
Ed Williams

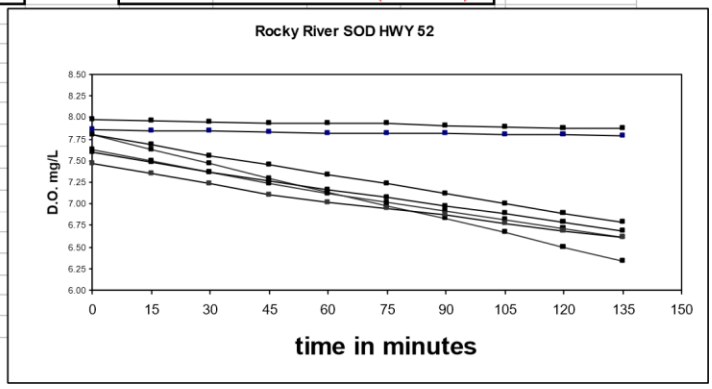


Figure 48. Example of SOD Excel Worksheet for Determining Average SOD Rates.