STANDARD OPERATING PROCEDURES MANUAL

FOR THE

DEPARTMENT OF ENVIRONMENTAL QUALITY

WATER QUALITY MONITORING

Commonwealth of Virginia
Department of Environmental Quality
Water Quality Monitoring and Assessment
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This document describes the routine operations and quality control activities performed by the Department of Environmental Quality (DEQ) in most of its ongoing data generating programs. Outlining procedures for sampling and field testing activities helps ensure that these procedures are standardized geographically across the state and between monitoring programs. The procedures described in this manual also help ensure that sampling precision, accuracy, representativeness, comparability and completeness of the data are obtained and documented. The sample collection procedures described in this document must be followed for all Water Quality Monitoring Programs unless the program is specifically covered under another SOP and/or Quality Assurance Project Plan that has been approved by WQMA QA Coordinator.

Many of the DEQ water quality monitoring programs have similar sample collection, field testing activities, and quality assurance requirements. Data generated from these programs must meet the needs of the data users. Comparability of data between DEQ's sampling programs and regional offices is an important quality objective

CHAPTER 1: PREPARATION

Before going out, it is recommended make a checklist of all routine material and equipment needed on the sample run to help gather all the items needed. Make separate checklists for specialized sampling such as clean metals, sediment and boat sampling.

At a minimum, the checklist should include the following items:

1. Field data sheets printed from CEDS for the scheduled run including sites where Quality Assurance (QA) samples are collected

2. List of sampling containers needed, preservatives, and labels, including QC samples, plus extra containers and labels.

3. Equipment for field measurements, sampling devices, coolers, and ice.

4. Topographic or similar map of the monitoring run and GPS unit to confirm site locations.

5. Safety gear relevant to the monitoring activity being conducted. Refer to Chapter 7 of this document for suggested equipment.

6. Cell phone, STARS radio or other form of emergency communication.

7. Verify all sampling equipment is clean, in good working condition and any equipment batteries are charged.

Before leaving on a sample run, inform the designated contact or supervisor the location of the run, expected return time and contact method if you are overdue.

Calibrate all field probe instruments according to manufacturer guidelines outlined in Chapter 3 of this document and enter the calibration information into the calibration log sheet. A template for the calibration log sheet is available in Appendix A.

Chapter 2: Cleaning and Preparation of Sampling Equipment

2.1. Sampling Equipment

A variety of handling and cleaning methods are used to maintain sampling equipment depending on use and application requirements. For example, if collecting metals or similar samples, the use of non-metallic materials like plastic (or Teflon) is needed. For the collection of organic samples, non-organic or inert materials, such as stainless steel or Teflon, are needed.

2.1.1. General Grab Sampling Equipment Storage and Transport

Most sites are sampled using buckets or similar type of grab sampling equipment. Sample equipment must be clean and well maintained to ensure accurate sample results. Below are several general tips to follow when using grab sampling equipment.

- Never store or carry equipment such as the sampling spool in the sample bucket. Doing so can contaminate the equipment and cause nicks and scratches to the bucket.
- Examine equipment for dirt, rust, or scratches (clean or replace as necessary). Dirty or scratched equipment can allow residue and bacteria to remain after cleaning.
- Dispose and replace sample bottles or containers if cracks or contamination are present.
- Bulk shipments of plastic sample containers often have lids shipped separately. When storing containers, keep opened boxes covered or closed to reduce dust and other contaminants from entering exposed container openings or lids.
- When assembling plastic sample containers, wash hands or wear powder free gloves to reduce potential contamination from entering the container.

2.1.2. General Water Grab Sampling Equipment

This is a list of general water grab sampling equipment you will need while conducting tests out in the field.

- Sampling rope on spool.
- Stainless steel bucket with a fitting for mounting bacteria sample bottle on the outside, or other suitable sampling device (Van Dorn, Kemmerer, pump and hose, etc.).
- Clean sample bottles and/or cubitainers suitable for the samples being collected.
- Syringe or vacuum pump, filter paper, filter holder etc. for samples requiring filtering.

2.1.3. Sediment Grab Sampling Equipment

This is a list of sediment grab sampling equipment you will need while conducting tests out in the field.

- Sampling rope on spool.
- Certified pre-cleaned glass jar(s) with Teflon-lined lid
- Teflon coated or plastic spoon, and stainless steel spoon.
- Sample dredge (such as Petite Ponar) depending on sediment type and depth of water.
- Appropriately sized stainless steel pans.

2.2. Sampling Equipment Preparation and Cleaning

The subsections below cover routine sampling encountered by field teams. If sampling for compounds is not covered in this section, please contact the Quality Assurance Coordinator.

2.2.1. Water Sampling Equipment Cleaning and Maintenance

This complete guide is a schedule of tasks to maintain the cleanliness of your equipment.

At the End of Each Sampling Day:

1. Rinse grab sample equipment with lab grade water and air dry.
    a. If buildup remains, clean as outlined in the weekly or monthly schedule.
2. For pump and hose apparatus, refer to the Equipment Maintenance section of the Chesapeake Bay Program Tidal SOP manual at: http://deqnet/documents/index.asp?path=%2Fdocs%2Fwater%2FWater%5Fquality%2Fmonitoring%5Fand%5Fassessments%5Fprogram/CBP
3. If using another depth sampling device like a Kemmerer, or Alpha Bottle, follow the manufacturer's recommendations for cleaning.

Weekly Maintenance:

1. Wash stainless steel water sampling equipment (buckets, bacteria samplers, etc.) with lab grade soap (Liquinox™ or Alconox™) using a brush to remove all surface deposits.
2. Rinse thoroughly with tap water, then lab grade water, and allow to air dry at room temperature.
3. If rust or other deposits remain, scour clean along the steel grain using a soft brush, or clean cloth, and baking soda and water paste. ***After cleaning, repeat steps 1 and 2***.

Monthly Maintenance:

1. For the pump and hose apparatus, refer to the Equipment Maintenance section of the *Chesapeake Bay Program Tidal SOP manual* in *Section 2.2.1*.

Annual Maintenance:

1. Inspect rope used to lower sampling equipment for fraying and replace as needed.
2. Inspect rubber tubing to hold bacteria sample bottles for wear and replace as needed.
3. For the pump and hose apparatus, refer to the Equipment Maintenance section of the *Chesapeake Bay Program Tidal SOP manual* in *Section 2.2.*

2.2.2. Sediment Sampling Equipment Cleaning and Maintenance

At the end of each sampling day or prior to sampling:

1. Wash equipment using clean scrub brushes and Alconox™ or Liquinox™ detergent and rinse with lab grade water until all residues are removed.
2. Repeat washing and rinsing procedure using Citranox™ and lab grade water.
3. Rinse with pesticide grade ethanol or methanol to remove organic compounds.
4. Rinse thoroughly with lab grade water until all ethanol or methanol is removed.
5. Dry equipment at room temperature, away from potential sources of contamination.
6. Visually inspect equipment for any contamination prior to storage. Such contamination would include water spots, dust or sediment, rust and similar substances.
7. Cover cleaned equipment with clean aluminum foil until use.

2.3. General Sample Containe+Handling and Preservation

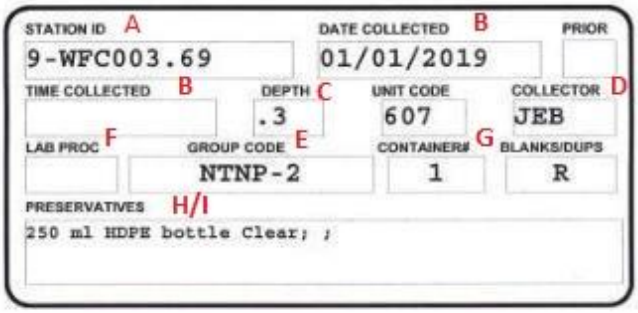
Proper sample containers and sample preservation are essential to sample integrity. Refer to the Division of Consolidated Laboratory Services (DCLS) laboratory catalog in Common Education Data Standards (CEDS) for the appropriate preservation procedures. Samples not preserved properly may be rejected by DCLS.

- Containers used by the Department of Environmental Quality (DEQ) are parameter and program specific to meet agency and DCLS sample size, purity, construction, and material requirements. Do not substitute sample containers listed in CEDS without first consulting the WQM Quality Assurance Coordinator or DCLS.
- Mark boxed or packaged sample containers with the date of receipt and stocked on shelves with the oldest dated box/packages used first. Keep sample container boxes closed while in storage to prevent dust or foreign material from entering.
- Discard sample containers that are damaged or have foreign material inside the container.
- After collecting the sample, tightly secure lids to prevent water leakage.
- Sample containers and coolers should be stored with the tops securely fastened. Replace caps or tape loose cooler fasteners to prevent loss of sample contents.
- Include a temperature bottle in each cooler so DCLS can determine sample temperature.

- Unless specified, pack sample coolers with collected samples as follows:
    1. Place all samples in an ice filled cooler immediately after collection
    2. ***Always place sample containers upright***.
    3. If possible, cover containers with ice so container openings are just above the ice.
    4. Filtered pad samples such as for chlorophyll a, go in a suitable sized Ziploc® style bag resting on top of the ice in the cooler. To prevent water from entering the bag, keep the sealed bag zipper opening outside the cooler. The best method is to place the zipper opening between cooler and lid on hinge side of the cooler.
    5. Bacteria sample bottles must be placed in mesh bags in coolers and surrounded with wet ice.
- To minimize breakage, wrap glass sample containers in bubble wrap or similar materials.
- Prior to shipping, drain melted ice from the cooler and top off the cooler with fresh ice to the level of necks of sample bottles.

2.4. Sample Identification

At a minimum, each sample container must be identified with the following information:



A. Station ID
B. Sample date and time (in military time)
C. Sample depth,
D. Collector initials
E. Parameter group code
F. Lab processes code (if applicable)
G. Container number (see below)
H. Preservative used (if any)
I. Volume filtered (if any, write in preservatives section)

For most sample containers, use a laser printer to print sample tag information on an adhesive Avery® style label, and apply directly to the container exterior. For cubitaners and bottles where the label does not stick well, attach the label to a wire tag; then, secure the wire tag by tying the wire to the container below the bottom lid lip next to the bottle shoulder.

Record any hand written information such as time collected on the sample tag using indelible ink. Sharpie® permanent markers with a fine or ultrafine tip work well for labeling.

Information on the sample tag must match exactly what is scheduled in CEDS field data screen. DCLS will reject samples where information on the tag does not match exactly what is entered into CEDS (including the sample time). If more than one container is needed for a group code, each container collected for that group code must have a label with identical information in addition to an indication of 1 of 3, 2 of 3, etc., as required.

The sample time must exactly match what is entered into CEDS. For routine and other non-compliance samples, it is acceptable to record sample time to the nearest 15 minutes as long as the sample time exactly matches the time entered into CEDS. Compliance or chain of custody sample should record the exact time of sampling.

Only print labels from CEDS using a laser printer to avoid the labels from smudging when wet. If a label is needed where a station is not yet established in CEDS, or if CEDS is down, the wire tags contain a blank sample tag that can be written on and attached to the bottle.

Note: Always check the labeling information against the actual site. Samples not labeled properly may be rejected by the laboratory.

Some sample types may require specific labeling. If so, the requirements are usually detailed with the sampling guidelines provided upon receipt of the sample bottles.

2.5. Muffling Sample Filters

Most samples covered under this SOP manual do not use muffled glass fiber filters that remove trace contaminants like carbon. If samples require muffled filters, the WQM program uses the muffling procedure found in the *Chesapeake Bay Program Tidal SOP manual* at http://deqnet/documents/index.asp?path=%2Fdocs%2Fwater%2FWater%5Fquality%2Fmonitoring%5Fand%5Fassessments%5Fprogram/CBP

2.6. Chemical preservatives and reagents

Each regional office is responsible for maintaining an adequate supply of chemical reagents to preserve samples and clean equipment. Expired or tainted chemicals can result in inadequate preservation and sample contamination.

Note: While the information below is helpful, please refer to the DEQ Chemical Hygiene Plan at http://deqnet/programs/healthsafety/policiesprograms.asp , or speak with the regional Chemical Hygiene Officer for more information.

- American Chemical Society (ACS) reagents (or similar) grade preservatives are required for sample preservation.
- When receiving new chemical reagents, staff should note the expiration date provided on the bottle. If no date is listed, record the date the chemical was received on the container label using indelible ink. *Table 2.1* contains a list of the typical shelf life for routinely used chemicals covered in this SOP.
- Write the date when then bottles were first opened to ensure they are consumed quickly.
- When not in use, store chemicals in the appropriate storage cabinet. ***Do not store clear or amber colored reagent bottles in the open, as light may degrade the chemical***. Chemicals in opaque bottles may be stored on a shelf or laboratory cabinet if they are not overly toxic or harmful. Otherwise, store reagents in the proper chemical storage cabinet
- Whenever handling chemicals of any kind always follow safe laboratory techniques including the use of eye protection, gloves, and aprons. Always wash hands after handling chemicals. *Chapter 7* contains general information on handling routinely used reagents.

2.6.1. Chemical Preservatives and Reagents Disposal

- Soap solutions and waste tap or lab grade water can be poured down the drain.
- Diluted solvents and acids used in cleaning may be poured down the drain after neutralization and additional dilution with tap water.
- High strength solvent and acid waste is to be handled as hazardous waste and must be properly collected and disposed of according to federal, state, and local regulations. Consult the regional Chemical Hygiene Officer for guidance on handling and disposing high strength chemicals or waste products.

Table 2.1: Typical Shelf Life of Reagents*

Compound Type	Name of Reagent	Shelf Life	Recommended Storage Area	Discard prior to expiration if:
Acid	Concentrated acetic acid	3 years	Acid cabinet	Cloudy and/or solids are observed in the container.
	Conc. hydrochloric acid			
	Conc. sulfuric acid			
	Conc. nitric acid		Oxidizer or acid cabinet. **DO NOT** store with organics.	Nitric acid has urine-like yellow/orange color.
Base	Conc. sodium hydroxide or pellets.	5 years	Base (Alkaline) cabinet. Pellets may be stored in a closed container on a laboratory shelf.	Liquid is cloudy/ discolored or solids are observed. Pellets clump together and cannot break apart easily
Solvents/ Organics	Acetone	5 years	Flammables cabinet	Solution turns cloudy or solids observed in the container
	Conc. ethanol or methanol			
	Formalin (formaldehyde)	3 years		
Solid Chemicals	Potassium chloride crystals	5 years	Laboratory shelf away from light (if bottle is not opaque)	Powder clumps do not break apart from shaking container
	Sodium thiosulfate crystals			
	Magnesium carbonate			
	Sodium Chloride			
Buffers	pH Buffer	12-18 months	Laboratory shelf or storeroom	Cloudy or suspended solids
Dye	Rhodamine WT	1 year	Laboratory shelf away from light	Discoloration or cloudy
Diluted Reagents	Any of the above compounds which is diluted <50%	1 month to 1 year	Laboratory shelf away from light (if bottle is not opaque)	Discoloration, suspended solids, insufficient strength

Note: *The DEQ Safety page http://deqnet/programs/healthsafety/policiesprograms.asp contains a list of chemicals and storage locations for each region.

2.7. Laboratory Glassware Cleaning

After use, all glassware used to prepare or handle chemicals must be cleaned using lab grade soap and water. Precise volumetric flasks and pipette glassware may require additional acid wash cleaning. After adding lab grade water, acid washing should be performed if water droplets are seen inside the volumetric glassware.

Note: When handling high strength acids, consult the regional Chemical Hygiene Officer.

1. Before starting the acid washing process, clean the glassware.
    a. Add a small amount of phosphate free, lab soap such as Liquinox® and lab grade water into the glassware. Use a clean bottle brush or cap the opening with a stopper or gloved hand, wet the entire inner surface with the soap and water mixture. Pipettes can be cleaned by placing in a pipette cleaner or by hand washing.
    b. Dump the soapy water and rinse with lab grade water six times.
2. ***For non-volumetric glassware, skip to step 9***. If cleaning volumetric glassware, look to see if water droplets form on the inner surface. ***If no droplets are seen, proceed to step 9. If droplets are seen, proceed to step 3.***
3. Using the fume hood, pour a small amount (usually 10 to 20 ml) of 50% or concentrated sulfuric acid into a beaker. Use the beaker to pour into the flask or draw up into a pipette.
4. Allow the acid to slowly coat the glassware inner surface by capping and rotating/inverting flasks to wet the entire surface. For pipettes, fill to at least 1 inch above the volumetric line.
5. The acid is finished cleaning when the liquid coating the surface flows smoothly (no ripples). ***Repeat steps 3 to 5 until the acid coating the bottle flows smoothly***.
6. Slowly pour out the acid wash into a beaker to reuse for a second piece of glassware. Discard acid washes in an acid waste container or neutralize in a bucket containing a solution of water and baking soda (sodium hydroxide). If neutralizing, add excess baking soda until bubbles stop forming, or check the pH using litmus paper. ***Do not dump contents down the sink until neutralized (no bubbles) or pH is above 4.00.***
7. Rinse the glassware using three successive rinses with 20-50 ml of lab grade water. Pour the rinses into the waste container or neutralizing bucket. For pipettes, use a clean squirt bottle with lab grade water and do three rinses through the top hole of the pipette.
8. Observe the glassware to see ***if water spots form; if so, repeat steps 3-7***.
9. Allow the glassware to dry completely. For volumetric flasks, cap using a plastic flask cap or foil to prevent dust from entering the cleaned container while drying and in storage. ***Do not cap flasks with ground glass stoppers as they can become stuck.*** Pipettes can be wrapped in foil. Store glassware in the glassware cabinet or pipette drawer until needed.

2.8. Analytical Scale Calibration

Analytical scales that are used to measure reagents must be properly calibrated as follows:

1. Turn the scale on and allow it to warm up for at least 15 minutes. While warming up, use a soft brush to clean the weighing pan and close all scale doors.
2. Level the scale by adjusting the feet and using the provided level bubble indicator.
3. If calibration weights have dust or foreign material present, gently clean them using a soft cloth or lint free tissue. ***Do not pick up or handle weights with bare hands.***
4. Once the scale has warmed up, tare the scale so the readout displays 0.0000 grams.
5. Following the scale manufacturer manual, set the scale to calibration mode. In calibration mode, the scale will usually display the needed weight to calibrate.
6. Using a lab tissue, or weight tongs, place the necessary weight(s) on the center of the scale weighing pan and gently close the door.
7. The scale will indicate calibration is complete and automatically exit calibration mode.
8. Remove weights and allow the scale to report a stable reading. Tare the scale to 0.0000.
9. To ensure the calibration accuracy. Place the calibration weight(s) back onto the scale to confirm readings are within 0.0005 grams of the weight(s). ***If not, repeat steps 6-9***.

2.9. Lab Standard Preparation

Depending on the equipment used and parameter monitored, it may be necessary to prepare a working set of reagents. To ensure accurate preparation, use the following steps while handling reagents.

2.9.1. General Guidelines

The following measures should be taken when using reagents.

- Refer to *Table 2.1* to confirm reagents are not expired and in good condition,
- If using unexpired but clumpy powder reagent, it must be dried to ensure accuracy through the following steps:
    1. Place an approximate amount of reagent into a clean weighing pan or crucible.
    2. Place the pan into a drying oven set at 100-110° C and heat for at least 3 hours.
    3. Remove the pan from the oven and place in a desiccator that has activated desiccant (usually blue color indicates activated desiccant) until ready for weighing.
    4. Just before weighing, use a clean lab spatula to confirm powder no longer clumps.
- Only use lab grade water to prepare reagents. Contact the Quality Assurance Coordinator if there may be problems with the regional office water purification system.
- Use analytical scales and Class A volumetric glassware to measure stock reagents.

Table 2.2 lists commonly used standards, amount of stock regent, and dilution water needed.

Table 2.2: Preparation of Commonly Used Stock Solutions per Liter

Solution	Uses for	Powder Reagent	Liquid Reagent	Dilute To:
50% H_2SO_4	Preservative, cleaning agent	N/A	500.0 ml concentrated H_2SO_4. ***First add 300 ml water to volumetric flask.***	1.0 L with lab grade water
10% HCl	Cleaning agent	N/A	100.0 ml concentrated HCl. ***First add 300 ml water to volumetric flask.***	
1 M KCl	Stock solution	74.551 g KCl	N/A	
0.1 M KCl	12,880 uS/cm standard	7.455 g KCl	100.0 ml 1 M KCl	
0.01 M KCl	1,413 uS/cm standard	0.7455 g KCl	100.0 ml 0.1 M KCl or 10.0 ml of 1 M KCl	
0.001 M KCl	147 uS/cm standard	N/A	100.0 ml 0.01 M KCl or 10 ml of 0.1 M KCl	
10 g/L MgCl	Chlorophyll a filter preservative	10.0 g MgCl	N/A	
Rhodamine WT 125 mg/l Stock	Stock solution	N/A	5.0 ml 2.5% Rhodamine WT	
625 ug/L Rhodamine WT	Chlorophyll and blue green standard	N/A	5.0 ml of Rhodamine WT stock	
1000 mg/l NaCl	Chloride sensor	1.655 g NaCl and 0.5g $MgSO_4$	N/A	
10 mg/L NaCl	Chloride sensor	0.5g $MgSO_4$	10.0 ml 1000 mg/L NaCl	

2.9.2. Standard Preparation Using Powder Reagents

If using an analytical scale to measure powder reagents, use the following procedure:

1. Turn on and calibrate the scale as outlined in *Section 2.8* of this manual
2. After verifying the accuracy of the scale, place a clean, empty weighing dish or crucible onto the scale and close the scale door.
3. After the scale stabilizes, press the "**Tare**" button. The display should show a reading of 0.0000 g. Remove the dish after zeroing the scale.
4. Using a clean beaker, measure out an approximate amount of powdered reagent needed.
5. With a clean spatula, transfer the reagent from the beaker into the weighing dish. Place the dish back onto the scale and allow the reading to stabilize. Add or remove reagent to ensure accuracy by arriving at the desired weight; then, close the scale door. ***Do not pour unused reagents back into the reagent bottle.***
6. When the weight is correct, remove the weighing dish containing the reagent and carefully pour into a clean volumetric flask using a clean funnel. Rinse all residues from the pan and funnel into the flask using lab grade water. Discard any unused reagent. ***Do not dump unused reagent back into the reagent container***.
7. Proceed to *Section 2.9.5* for mixing the reagent using a volumetric flask

2.9.3. Standard Preparation Using Liquid Reagents and Flask

If using volumetric glassware to measure liquid reagents, use the following procedure:

1. See if the volumetric flask being used is clean by pouring ~10 ml of lab grade water to see if spotting on the stem of the flask occurs. Discard this rinse down the drain.
    a. If spotting is seen, use new flask or go to *Section 2.7* to acid wash your current flask.
2. Fill the flask with the reagent so the bottom of the meniscus (U shaped depression) rests on the etched line of the flask stem. Discard any excess down the drain using a clean pipette.
3. Drain the entire flask contents into the larger preparation volumetric flask. Rinse the smaller flask with three rinses of 10-20 ml of lab grade water and drain into the larger flask. Ensure that rinses cover the entire inner surface of the smaller flask.
4. Proceed to *Section 2.9.5* for mixing the reagent using a volumetric flask

2.9.4. Standard Preparation Using Liquid Reagents and Pipette

If using a volumetric pipette to measure reagents, use the following procedure:

1. Be sure the pipette is clean by filling to the pipette printed measurement line with the reagent. Drain the contents into a waste container or sink and check for droplets on the inside of the pipette. If it is dirty, obtain a new pipette or clean following *Section 2.7*.
2. If the pipette is clean, fill again with the stock solution to the printed measurement line and drain into the volumetric flask which will be used to prepare the lab working solution.
3. If the pipette has a To Contain (TC) mark, blow out any residue into the flask and rinse the inside with lab grade water into the flask. If using a To Dispense (TD) pipette, ***Do not blow out contents or rinse***.
4. Proceed to *Section 2.9.5* for mixing the reagent using a volumetric flask

2.9.5. Proper Filling and Mixing Using Volumetric Flasks

Follow these procedures when using volumetric flasks for filling and mixing.

1. Fill the volumetric flask containing the reagent and rinses to the neck with lab grade water.
2. Tightly cap the flask using a cap and invert ten times or until the powder/crystals dissolve.

Note: Magnesium carbonate will not fully dissolve.

3. Remove the cap and using a squirt bottle, add additional lab grade water to the etched line on the flask. The bottom of the meniscus (U shaped depression) should rest on the etched line. If the flask is overfilled, discard the solution as it is now more diluted than required.
4. Cap and invert the flask a least 20 times to ensure proper mixing. Label the container that will hold the standard with the standard strength, date of preparation, and preparer initials.

Chapter 3: Field Probe Calibration and Maintenance

3.1. General Calibration Items

This chapter covers the calibration and maintenance of electronic field equipment. Although different regions and programs use different field probes, the same general procedure applies.

3.1.1. General Transport and Storage of Probeware

To ensure sensor accuracy, transport and store probes in the following manner:

1. Attach the calibration cup or other container that has a small amount (~10 ml) of fresh pH 4.0 buffer or tap water. ***Do not submerge the sensors in the solution.***
2. Turn off the probe and display to conserve battery life while not in use.
3. If possible, keep equipment in a temperature controlled environment such as the vehicle's passenger compartment while traveling and store inside the regional office when not in use.

3.2. Quality Assurance of Field Probes and Related Equipment

Read these sections when prepping field probes for use, calibration, and storage.

3.2.1. Controlled Environment

When possible, calibrate sensors in a temperature controlled environment. Allow the probe to stabilize before calibrating. A probe is considered stable if the readout indicates it is stable or does not significantly change (±0.01 units) within ten seconds.

3.2.2. General Calibration Guidelines and QA limits

Calibrate handheld probes before going into the field. Always use unexpired standards or within six months of the date of opening/date of preparation.

3.2.2.1. Temperature Verification

Periodically compare the field probe temperature reading to a laboratory thermometer of known accuracy. Record both readings on the log sheet. ***Notify the QA Coordinator if the temperature difference is greater than 0.5 °C.***

3.2.2.2. Probe Calibration

Calibrate sensors using probe specific procedures listed in this chapter.
Table 3.1 outlines calibration tolerances and key calibration notes.

Table 3.1 Probe Calibration Tolerances

Parameter	Calibration Tolerance	Calibration Notes
Conductivity	2.0% of standard	Use standards closest to expected field values.
Clark DO	0.20 mg/L of theoretical level	Calculate theoretical Dissolved Oxygen (DO) values using Appendix B
Optical DO	0.10 mg/L of theoretical level	Calculate theoretical DO values using Appendix B
pH	0.10 SU of reference	Use standards closest to expected field values. Probes using a 2-point calibration must use a third Point (4.0 or 10.0) in the end of the day check if any field readings were outside the calibrated range.

3.2.3. Midday DO Calibration Confirmation

When sampling Dissolved Oxygen (DO) at multiple sites during the day, perform a DO confirmation check in the middle of the run. This check confirms the DO sensor is still accurate and reduces the need to flag an entire sample-run worth of readings. To perform the midday check:

1. Place the DO sensor in a 100% air/water saturated environment. Either wrap in a wet towel or in the storage cap with small amount water and away from direct sunlight.
2. Allow the readings to stabilize (<0.05 unit change in 10 seconds).
3. Record the DO reading in mg/L or % saturation (***see the Note below***), temperature, and barometric pressure on the field data sheet and enter it to the comment field of CEDS.
4. The reading should be within 0.49 mg/L of the Appendix B theoretical DO value.

Note: 95 to 105% saturation is an acceptable shortcut check up to 1,000 feet elevation. Over 1,000 feet, this shortcut may not work due to lower barometric pressure. Field packets should contain *Appendix B* in the event recalibration is needed or to check theoretical DO values.

If the midday probe check fails, recalibrate the probe in the field and flag the morning data. ***Do not enter the run dissolved oxygen data collected prior to a failed mid-day check in CEDS.***

3.2.4. End of Day Checks of Probes

When sampling multiple sites during the day, teams must verify field probe accuracy by performing an end of day check. ***The end of day check is not a calibration*****.** The probe is verified by checking against standards in a controlled environment. ***If the end of day check exceeds the values outlined in Table 3.2, do not enter associated field data into CEDS.***

Note: A probe at room temperature will stabilize faster and reduce error during the end of day check. If probe temperature drifts strongly (>0.20° C in 10 seconds) during a check, set up a room temperature water bath and submerge the probe for at least 15 minutes

3.2.4.1. Dissolved Oxygen End of Day Check

This is an end of day check for any changes in DO you may encounter as you make your rounds; follow these steps:

1. Rinse and place the oxygen sensor in the calibration cup to allow the temperature and DO readings to stabilize ($\pm$0.01 unit change per 10 seconds). This may take several minutes.
2. While adjusting, record the probe or lab barometer pressure on the calibration log sheet. If the lab barometer is not in service, follow NWS procedures outlined in *Appendix B*.
3. Once the probe temperature and DO readings are stable, record the values on the end of day check portion of the calibration log sheet.
4. Using the table in *Appendix B*, determine the theoretical dissolved oxygen saturation value. Record this on the calibration log sheet in the appropriate section.
5. ***Do not enter DO data into CEDS if the reported end of day DO value is greater than or equal ($\geq$) 0.50 mg/L of theoretical levels***, Service DO sensors whenever end of day check is greater than 0.30 mg/L (0.20 mg/L Optical DO sensors) of theoretical DO levels.

3.2.4.2. Conductivity End of Day Check

This is an end of the day check for conductivity found in the water samples. For an accurate result, follow these steps:

1. Rinse the conductivity probe with lab grade water and blot dry. Ensure the reading is 0 uS/cm (or 0 mS/cm). This verifies the sensor is not giving a false positive reading.
2. Rinse the sensor and calibration cup with conductivity solution of the same strength used to calibrate the probe. Refill the cup with fresh conductivity solution. Dislodge any air bubbles on the sensor by shaking or gently swirling the cup.
3. Allow the conductivity readings to stabilize. This may take up to one minute or two.
4. Record the reading in the appropriate section of the calibration log sheet.
5. ***Do not enter conductivity values in CEDS if the value is off by more than 10% for 147 uS/cm standard or 5% of higher strength conductivity solutions.*** Service sensors if the end of day check fails.

Note: Table 3.2 lists the acceptable range of readings on commonly used conductivity solutions.

Table 3.2 Conductivity End of Day Check Acceptable Ranges

Conductivity Solution Used	Acceptable range
12,880 uS/cm (12.9 mS/cm)	12,236 – 13,524 uS/cm (12.2 – 13.5 mS/cm)
1,413 uS/cm	1,342 uS/cm – 1,484 uS/cm
147 uS/cm	132.3 uS/cm – 161.7 uS/cm

3.2.4.3. pH End of Day Check

1. Rinse the pH probe with tap or lab grade water followed by fresh or used 7.0 buffer.
2. Fill the calibration cup (or appropriate container) with fresh 7.0 buffer.
3. Allow the pH and temperature readings to stabilize (±0.01 units in 10 seconds).
4. Record the reading in the appropriate section of the calibration log sheet. If the probe can display millivolt (mV) readings, record this as well.
5. ***Repeat steps 1 through 3 for pH 4.0 and/or 10.0 buffer.*** Use the same strength standards used during the morning calibration and any covering the values encountered in the field.
6. ***Do not enter pH data into CEDS if the pH probe reading is greater than (>) 0.20 S.U. different from buffers used in the end of day check.*** Service pH sensors if the end of day check is greater than 0.10 S.U. of a pH buffer standard.

Table 3.3 summarizes the maximum allowable error range for the end of day check to enter run data into CEDS and the range when probes must be serviced.

Table 3.3 Maximum Error Limits and Servicing Limits for End of Day Check

Parameter	Maximum Allowable Error	Servicing of Sensor Required
DO- Clark cell	> 0.49 mg/L of theoretical	>0.30 mg/L of theoretical
DO- optical sensor	> 0.49 mg/L of theoretical	>0.20 mg/L of theoretical
Conductivity 147 µS/cm	> 10% of standard	> 10% of standard
Conductivity >147 uS/cm	> 5% of standard	> 5% of standard
pH	> 0.20 S.U. of buffer	>0.10 S.U. of buffer

3.2.5. Periodic Field Probe and Equipment Performance Verification

Electronic equipment will degrade due to age or improper maintenance. To limit data loss due to faulty readings, field and central office staff perform the following verification checks.

3.2.3.1 Field Probe Equipment Verification

At least once every six months, the following procedure is performed to verify the accuracy and performance of identified key electronic equipment.

1. Calibrate at least three probes for the same parameter(s).
2. Place the calibrated probes(s) in a container of warm (~30° C) tap water so that at least all sensors are submerged. Using a cooler as the container is helpful.
3. Turn on the handheld display(s) and probe circulator(s) if necessary.

Note: For the most accurate readings, circulate water with a large spoon or similar method.

4. Allow the readings to stabilize.
5. Once stabilized, Record the dissolved oxygen, pH, and temperature reading(s) from the displays to the verification sheet found in *Appendix F***.**
6. Calculate the average from all of the probe readouts and compare to individual readings.
7. Once the verification form is completed, submit it to the QA Coordinator for review.

Note: An optional verification step involves checking DO readings by Winkler titration and using a reference pH solution. *Appendix E* contains procedures to conduct such checks.

Table 3.4 lists the acceptable range for the above checks and necessary corrective actions.

Table 3.4: Periodic check, variance from average or reference value, and corrective action

DO	pH	Temperature	Corrective Action Required
<0.30	<0.10	<0.50	No corrective action needed
0.30 to 0.60	0.10 - 0.20	N/A	Service affected probes by replacing membrane, electrolyte and/or cleaning sensor
>0.60	>0.20	>0.50	Contact QA Coordinator as data may need flagging. Tag unit out of service until serviced and checked.

3.2.3.2 Lab Barometer Verification

At least quarterly, regions should check the laboratory reference barometer to the nearest National Weather Service (NWS) station. The most accurate NWS stations are at major airports.

1. Using *Appendix B*, locate the nearest NWS station and adjust the reading based on the elevation of where the reference barometer is stored.
2. Record the NWS and reference barometer reading on the calibration log sheet.
3. If readings between the NWS and lab barometer are greater than 5 mmHg, contact the QA Coordinator

3.2.3.3 Probe and Reference Thermometer Verification

At least annually, Central Office personnel will check water quality sensors and reference thermometers using a National Institute of Standards and Technology (NIST) validated master thermometer.

1. Three water baths are made of varied temperatures
    a. Ice Bath (0-5° C)
    b. Room Temperature (18-23° C)
    c. Warm Water (30-35° C)
2. Regional probes and reference thermometers are placed in each water bath and readings are compared to the NIST reference thermometer.
3. If readings are greater than 0.5°C, affected units are flagged and data collected by the associated units will be evaluated for possible quality assurance failure and flagging.

Note: Muffle furnaces are checked at their set operating temperature against a master thermistor unit rated for the operating temperature. Muffle furnaces readings should be 500 +/- 5^{o} C.

3.2.3.4 Analytical Scale and Weight Verification

At least annually, Central Office personnel will verify the accuracy of analytical scales and calibration weights used by the Regional Offices. This check uses NIST verified weights to check the scale at three reference points across the expected weighing range and the regional calibration weight(s). For analytical scales and weights typically used to weigh out chemical reagents covered in this SOP manual, the maximum allowable error is +/-0.0005 gram. The verification procedure is as follows:

1. As outlined in *section 2.8* of this document, the scale is cleaned of dust, allowed to warm up, balanced, and tare to zero grams (0.0000 g).
2. The scale is calibrated using the NIST validated weights maintained by Central Office.
3. The scale accuracy is verified using the three NIST verified weights maintained by Central Office, Readings must not be greater than 0.0005 g of the verified weight value.
4. Scale is recalibrated ***if it fails verification using steps 1-3.***
5. ***If the scale again fails verification, it is reset to factory default settings, recalibrated, and checked using steps 1-4***.
6. If scale still fails, it is tagged out of service until serviced by a professional company.
7. If scale passes verification, Regional Office calibration weight(s) are checked. If the calibration weight(s) are off by more than 0.0005 g, they will not be used until evaluated by a professional company or replaced with an NIST verified set.

3.3. Handheld pH Meter

Applies to Accumet, Orion, and Oakton model series hand held meters.

3.3.1 General procedures

Follow these steps when checking the pH of water.

1. Rinse electrode thoroughly with water after each sample or standard. Blot sensor dry. Make sure the pH buffer is not expired or appears contaminated before calibrating.
2. Calibrate with at least two pH buffers that most closely bracket the expected sample water pH. The buffers used for calibration are 7.0, 4.0 and/or 10.0.
    a. If sample pH was outside calibrated buffers used, check the accuracy by using the buffer that brackets the observed reading to see if recalibration is necessary.
3. If the probe is off more than ±0.20 SU of the buffer value, recalibrate and resample if possible. If not, exclude the data.*****
4. Between readings, rinse pH probe with water and blot dry.

Note: *The pH probe generally has a one-year service and shelf life.

3.3.2 Instrument setup

Be sure to follow these directions when setting up your pH meter for testing.

1. Connect sensor cable(s) to the appropriate meter jack(s) using any required adaptors.
2. Cover any unused meter AC power jacks or ATC jacks, to maintain a waterproof state.
3. Turn the meter on by pressing the power button. When the unit finishes the startup checks, press the **pH**, **SETUP**, or **MODE** button to display pH values.

3.3.3 Calibration

Follow these steps to properly calibrate your pH meter ***before*** field testing.

1. Press the **CAL**, **SETUP**, or **MODE** button to enter into calibration mode. The display will indicate calibrate mode by a flashing number or the word **CAL** on the display.
2. Remove the electrode protective cap and rinse with water. Shake or blot the sensor dry.
3. Immerse the electrode in the first buffer solution (usually 7.0) and slowly stir the electrode to ensure the sensor is properly reading the solution.
4. The display should recognize the pH buffer by displaying a steady value and/or beep when stabilized and locked. Some units require pressing the **CAL** button to lock readings. Record calibrated pH value on the log sheet.
5. ***Repeat steps 2-4 with a second or third buffer.*** The unit should then exit the calibration mode. ***If the meter failed to calibrate or has an error message, do not use until the sensor is serviced or replaced.*** Refer to manufacturer manual if a pH slope value is needed.
6. Place the plastic protective cap over the probe when not in use. Be sure the cap has some fresh pH 4.00 buffer or electrode storage solution and placed in the bottom of the cap.

3.3.4 Field measurement procedures

1. Turn the unit on when ready to sample and make sure it is in pH measurement mode.
2. Immerse probe in the sample solution and stir at a moderate pace.
3. When the meter senses that the reading is stable, "**STABLE**" may appear under the measurement reading. Record this value on the field sheet. Enter pH data into CEDS to the hundredth place.
4. If "**AUTO**" is not displayed on the screen, the auto read function is not active, and the meter will continuously monitor the pH value of the sample.
5. If "**AUTO**" is displayed on the screen, the meter will fix the stabilized pH value on the screen. "**AUTO**" will flash on the display until a stable reading is obtained.
6. Rinse the probe with water and place back into the plastic protective cap which contains a moist tissue of pH storage solution (pH 4.0).

3.3.5 Maintenance

See *Table 3.5*

Table 3.5 Maintenance: Accumet, Beckman, Oakton, Orion, and similar series pH/ISE meter

Sensor Type	Item	Procedure
pH sensor	**Slow pH response or sensor is returned from long-term storage** **returned from long term storage.**	**Note:** If using a liquid filled sensor, make sure to fill the fluid chamber with the proper electrolyte and to the proper level. Soak sensor in warm water with mild detergent for 10-30 minutes. If ineffective, clean the glass sensor with mild soap and a soaked scrub. If still ineffective, or the sensor shows a ***slow response,*** soak sensor for a few minutes in water with 10% HCI, or several hours with a pH 4 Buffer (for pH sensor only). After cleaning, rinse with tap water and blot dry. Place storage solution for at least two hours before calibrating
	Short term storage of pH/ISE electrode (<1 month)	1. Place electrode tip in container provided or other watertight container containing electrode storage soulation or pH 4.0 buffer. 2. If using a liquid-filled pH sensor, leave vent open while measuring. Close vent while in storage. Refill probe solution if below recommended levels.
	Long term storage of pH/ISE electrode (>1 month)	1. Disconnect sensor from meter. 2. Rinse sensor tip and blot dry. a. For liquid filled probes. drain all liquid and rinse with lab grade water b. Using the vent hole. Drain water and allow to air dry. 3. Remove batteries to avoid damage to the unit while in storage. 4. Place electrode in a protective container. Store in a cool and dry location.
General Maintenance	**Replacing batteries and/or using AC power Supply**	1. Remove the battery cover from the back of the meter and old battery. 2. Install the new battery of the same type and voltage. Make certain any battery wires. ***Do not to interfere closing of the battery cover***. If using an AC adaptor, connect the adaptor to the AC jack and power source. The meter is not waterproof when the AC adaptor is connected.

3.4 Handheld Dissolved Oxygen Meter

This section applies to YSI 55, 85, 95, and PRO series handheld meters.

Note: YSI 58 users, refer to the manufacturer manual for calibration and operation of the unit.

3.4.1 Calibration

Follow this procedure to properly calibrate your DO meter for field use.

1. Ensure that the calibration cup sponge is wet. Insert the probe into the calibration cup.
2. Turn the instrument on and wait for stable dissolved oxygen and temperature readings which may take 10 minutes to an hour. Optical units stabilize in less than 5 minutes.
3. Enter the calibration menu. Depending on the model used, this either involves pressing the **UP ARROW** and **DOWN ARROW** buttons at the same time (YSI 55, 85, 95), or press the **CAL** button (YSI Pro)
4. The display may either prompt the user to enter the local altitude in hundreds of feet or barometric pressure of the calibration site. Use the **ARROW KEYS** to increase or decrease the displayed value to match current conditions. When the proper reading is displayed, press the **ENTER** button once.
5. The instrument should now display "**CAL**" in the display. The calibration value may be displayed with the % saturation reading. Make sure readings remain stable.
6. Press the **ENTER** button to lock the saved reading and exit the calibration menu. The display should no longer say "**CAL**" and return to the normal readout display.
7. Record the calibrated DO and temperature readings on the log sheet.
8. If the probe can test for other parameters such as the YSI 85 sensor to check conductivity, calibrate the unit following above steps and calibration and end of day check steps outlined in *Section 3.2 and 3.3* of this document.

3.4.2 Taking Measurements

When arriving to the field, turn on the unit. It should display the current temperature and DO reading after a few seconds. An error message will display if an internal problem is detected.

If the probe has a Clark Cell sensor (clear plastic over the sensor tip), make sure the probe moves through the sample water at the rate of at least 1 foot per second to provide adequate stirring and representative results. Optical DO sensors (black plastic over the sensor tip) do not need moving water to get an accurate reading.

Record field parameter data and enter into CEDS based on the hundredth unit of the display.

3.4.3 Maintenance

See Table 3.6

Table 3.6. YSI 55, 58, 85, 95, Pro Series General Maintenance

Sensor Type	Item	Procedure
Clark Cell DO Sensor	**Changing DO membrane.** **Every 2-4 weeks or if membrane is torn, wrinkled, has air bubbles, or sensor readings are erratic or slow.**	1. Discard old membrane or cap. Rinse electrode with lab grade water. Blot dry. 2. Fill electrode well or cap with KCl solution provided in the membrane kit. **a. If using a membrane cap:** i. Fill cap ¾ full with KCl solution and remove any bubbles by tapping the side. ii. Lower the electrode into the membrane cap and tighten to finger tightness. iii. If bubbles or wrinkles are seen on the membrane, try again. **b. Using membrane film** i. With the electrode facing up, fill with enough KCl to form a positive meniscus. ii. Remove air bubbles by tapping the side of the sensor with a pencil. iii. Lay a sheet of membrane film so the KCl solution grabs the sheet. If bubbles are trapped under the sheet, remove and add more KCL solution. iv. Replace O-ring if older than 3 months or is damaged. ***Do not grease the O-ring.*** v. Secure the membrane with the O-ring by pushing down with both thumbs. vi. Note any air bubbles or wrinkles. If so, discard the membrane and try again. vii. If the membrane is satisfactory, trim excess film using scissors or a razor blade. 3. Allow the sensor to equalize (4 to 24 hours) before calibrating and using in the field**.**
	Cleaning silver anode. **Check for majority of anode is dark dull gray to black when changing DO membrane.**	1. Immerse the electrode in household strength ammonia solution (~3% ammonia hydroxide) for up to 8 hours or use 14% ammonia solution and soak 2-3 minutes. **Note:** 14% ammonia solution will destroy the sensor if left in too long. 2. If still tarnished, gently buff with moist 400 or finer grit sandpaper. 3. Triple rinse sensor with lab grade water and blot dry. 4. Add a new membrane using steps listed above.
	Cleaning gold cathode. **Check for tarnished or dull gold color when changing membrane**	1. Wipe the cathode with a clean lent-free cloth, pencil eraser or hard paper. The proper color should be gold with a matte finish. 2. If still tarnished, gently buff with moist 400 or finer grit sandpaper. 3. Triple rinse sensor with lab grade water and blot dry. 4. Add a new membrane using steps listed above.
Optical DO Sensor	**Cleaning sensor** **Whenever dirt, algae, or mold on the sensor housing or membrane.**	1. Flush the entire sensor with clean, fresh water. 2. Use a lint free tissue and gently wipe away any foreign material on the sensor cap. If the above fails, use a 15 minute white vinegar soak. Rinse with lab grade water. ***Do not clean with alcohol or organic solvents, as it will destroy the membrane.*** ***Do not remove the membrane cap unless replacing with a new cap.***
	Replacing membrane **Yearly, or damaged membrane or LED light seen through scratches (>1mm)**	1. Unscrew and discard the old membrane cap, old O-ring, and cap seal. 2. Place the new O-ring and cap seal at the appropriate locations of the optical sensor. ***Do not add grease to the O-ring or cap seal***. 3. Attach new membrane cap to the sensor. Tighten to finger tightness.***** **Note:*_Do not_** touch the outer or inner surface of membrane tip with ungloved fingers. 5. Add the new calibration constants as outlined in the replacement sensor package.
	Rehydrate membrane **If sensor dried out after 2 hours not in a 100% humid environment**	1. If possible, remove the optical sensor from the probe housing. 2. Place 400 mL of room temperature water in a 600 mL beaker. 3. Place the sensor with the membrane in the room temperature water for 24 hours. 4. Store the sensor in either water or water-saturated air at room temperature prior to calibration and deployment.
Conductivity	**Cleaning conductivity**	1. Using a paper towel or cotton swab, clean the sensor of hardened foreign

Sensor (YSI 85, PRO series only)	**sensor.** **As needed**	material using warm water and mild detergent. 2. Rinse sensor with lab grade water and blot dry. 3. If needed, repeat using a soft brush. Hard scouring will damage the sensor.
General care	**Short term storage (<1 month)**	Place DO sensor in calibration cup or bottle with a clean, moist sponge. Check to see the sponge is still moist every week when the unit is not in use.
	Long term storage (>1 month)	1. ***Clark sensor units only***- remove membrane or cap and rinse electrode with lab grade water and blot dry. Attach a dry membrane or cap. Store in a cool, dry place. 2. ***Optical DO units***- Add ~10 ml lab grade water to keep probe hydrated. 3. Remove batteries.

3.5 Multiprobe Calibration With Handheld Display

Applies to Hydrolab series 5 and HL4, In-Situ SmarTROLL, YSI series 6 and EXO

Note: The below procedures are written for users who are familiar with the basic operation of multiprobe displays including accessing the calibration and diagnostic menus. Refer to the multiprobe manual to troubleshoot or accomplish tasks not outlined below.

3.5.1 Calibration and Verification

General procedures:

Always

- Use fresh, unexpired standards to calibrate/verify. Old standards may be used to rinse probes.
- Calibrate/verify using standards closest to expected field values.
- Record calibration, end of day checks, and maintenance notes the calibration log book.
- When calibrating/verifying, have the sensors facing towards the ceiling. When handling the sonde, hold near the cable connection to minimize temperature changes.

Never

- Accept calibrations/verification if a warning or probe failure message is displayed. Service and recalibrate the unit before taking out to collect readings.
- Take a unit out on a run if the internal battery display drops below 7.2 volts (**Surveyor 4**) or battery indicator is less than ¼ full. Recharge or replace low batteries.
- Turn off the display prior to completing calibrations or results may not be saved.

Note: For the fastest and most accurate calibration, recommend calibrate in the following order.

A. Multiprobe Barometric Pressure Calibration/Verification

1. Turn on the display and go to the main readout screen
2. Compare the barometer reading to the laboratory barometer or use **Appendix B** to calculate local barometer reading from nearest National Weather Service (NWS) station.
3. If the two readings are off by 10 mmHg or more, calibrate the multiprobe barometer.

Calibration - When sonde barometer is +/-10 mmHg from lab barometer or NWS reading.

Note: Hydrolab HL4, In-Situ SmarTROLL, and **YSI EXO. *Do not*** allow users to calibrate the multiprobe barometer. Barometer units must be returned to manufacturer for adjustment**.**

1. Enter the calibration menu
2. Select **BP:User Cal**, **Barometer,** or similar option
3. Use the **keypad** to enter the mmHg reference barometer value and press **Done/OK**. Record the entered value in the log sheet.

B. Multiprobe Conductivity Calibration

Note: Hydrolab HL4 and **In-Situ SmarTROLL** require users to perform a zero calibration. Hydrolab Series 5 and YSI EXO should also verify a zero reading for conductivity using similar procedures but will not zero calibrate.

Zero Calibration- Calibration required for HL4 and SmarTROLL

1. Rinse the conductivity sensor twice with lab grade water and dry sensor gap with a tissue.
2. Use the **keypad** to enter the **Calibration** screen and select **Conductivity.** If prompted select **Specific Conductivity 2 point**. Enter **0** as the value **keypad** and select **Done/OK**. Confirm the probe is reading 0 and press **OK** and follow screen prompts.

Slope Calibration- All Sondes

1. Replace the storage cup and rinse with fresh or used conductivity standard that is closest to expected field values (e.g. 147 uS/cm for freshwater, 12,880 uS/cm for sea water)
2. Fill the calibration cup with fresh standard until the thermistor and conductivity sensor are submerged. Tap the side of the cup if bubbles are inside the conductivity sensor gap.
3. Allow the temperature and conductivity readings to stabilize. A stable reading is when the smallest displayed digit does not change more than one unit in ten seconds. Usually this will take a minute. Record the conductivity reading on the log sheet.
4. Go to the calibration menu and select **Specific Conductivity.**
5. Use the keypad to enter the calibration standard used and press **Done/OK**. Follow screen prompts to complete calibration and record displayed SpCond value on the log sheet.
6. The SpCond value must be within ±2.0% of the standard used.

Note: ***YSI Series 6 and EXO*** units report a conductivity cell constant. Use the **arrow keys** and **Enter** to select "Cal Constants" followed by "Advanced Menu". ***The acceptable cell constant is 5.0 ± 0.45.*** If values are outside the range, retry calibration with fresh standard and if it fails again, the conductivity sensor may need cleaning and/or replacement.

C. Multiprobe Dissolved Oxygen Calibration- Clark Cell

Note: It is best to store water used to calibrate dissolved oxygen sensors in a clean carboy at room temperature. This will reduce temperature equilibrium time.

1. Inspect the DO electrode and membrane for tears, nicks, discolored electrodes, and air bubbles. If present, follow maintenance procedures found in Table 3.7 of this document.

Note: If replacing a membrane, do not calibrate or use the sensor for at least four hours to allow for membrane stretching. Otherwise it will result in inaccurate results.

Note: For YSI Series 6, go to the "Report Menu" and record the DO Charge. The value must be between 25 and 75. If not, replace the DO sensor membrane.

2. If the unit has a circulator, turn it off while calibrating.
3. Fill the calibration cup with water to just below the O-ring of the oxygen probe.
4. Using a lint free tissue, gently remove water droplets from the oxygen probe membrane.
5. Loosely cover the storage cup and allow DO and temperature readings to stabilize (+0.01 units in 10 seconds). Record the initial DO and temperature in the calibration log book.
6. Calculate the theoretical oxygen value using Appendix B of this document. Record the theoretical value in the calibration log sheet.
7. Enter the calibration menu and select DO mg/L. Enter the current barometric pressure of the reference barometer using the **keypad**. Follow screen prompts.
8. Record the barometric pressure, sensor temperature, calibrated oxygen value and theoretical oxygen value on the log sheet in the appropriate place.
9. The DO (mg/l) reading must be within ±0.20 mg/l of the calculated theoretical value.

Note: For YSI Series 6 , use the **arrow keys** to go to "Advanced Menu", "Cal Constants". Record the "DO Gain". The gain must be between 0.7 and 1.4. Service the probe if out of range.

C. Multiprobe Dissolved Oxygen Calibration- Optical Sensor

Note: It is best to store water used to calibrate dissolved oxygen sensors in a clean carboy at room temperature. This will reduce temperature equilibrium time.

1. Using a cupped hand or cardboard tube will allow the user to see flashes of blue light from the oxygen sensor membrane. If blue light >1 mm in size is seen at the membrane tip (not the edges), replace the membrane using the procedure found in Table 3.6 of this manual.
2. Rinse sensors and calibration cup twice with lab grade water.
3. Vigorously shake a half filled container with room temperature water for at least 30 seconds. Add the water to the calibration cup until all the sensors are submerged leaving a small air gap at the end of the cup. Place a cap loosely on top of the calibration cup
4. Allow temperature and DO readings to stabilize. A stable reading is when the smallest displayed digit does not change more than one unit in ten seconds. Record this initial DO reading and temperature in the calibration log book.
5. Calculate the theoretical DO value using the Dissolved Oxygen Calibration Table in Appendix B of this document. Record theoretical value in the calibration log sheet.
6. Use the **keypad** to enter into the calibration menu. And select **Optic Dissolved Oxygen % saturation** (or LDO % sat, RDO % sat, or similar) Confirm the barometric pressure is accurate or enter the correct value using the **keypad**. Follow screen prompts.
7. After the confirming the DO and temperature values are still stable, follow the screen prompts to calibrate.
8. The optical DO (mg/l) reading must be within 00.10 mg/l of calculated theoretical value. Record the calibrated DO mg/L value in the log sheet

Note: For YSI Series 6 and EXO, when the calibration is completed, go to the Sonde **Advanced Menu** and then to the **Cal Constants** and record the **DO Gain**. The gain should be between 0.85 and 1.15. If the DO gain is out of range, perform a factory reset as outlined in the YSI manual to see if the reset corrects the DO Gain. ***If not, the probe needs servicing***.

D. Multiprobe pH Calibration

Note: When calibrating/verifying pH sensors, it is best to calibrate with the 7, 10, and 4 buffers but staff can just use the two pH buffers that most closely bracket the expected field values. After calibrating/verifying the unit, add ~10 ml of pH 4 buffer or tap water in the storage cup to keep the sensors moist. ***Do not allow the liquid to contact the sensors for an extended period.***

Note: Hydrolab and **YSI** series units require calibrating with 7 buffer first. **In-Situ** requires calibrating the slope buffer (4 or 10) before 7 buffer.

1. Rinse the calibration cup and sensors twice with lab grade water.
2. Rinse twice with a small amount of pH 7.0 buffer saved from previous calibrations to remove any residual liquid. Discard rinses down the drain.
3. Fill the calibration cup with fresh pH 7.0 buffer sufficient to cover the pH sensor.
4. Allow temperature and pH to stabilize.
5. Enter the calibration menu and select **pH 2 point** (or 3 point, or similar).
6. Using the **keypad**, enter the value of the standard being used and select **Start/Done/OK**. Follow screen prompts to calibrate. Record the calibration pH value in the log sheet.
7. Pour the used buffer into a storage bottle for use as a future rinse or end of day check.
8. ***Thoroughly flush the calibration cup and sensors twice with lab grade water.***
9. Rinse the cup and sensors twice with used pH buffer for the second standard.
10. Fill the calibration cup with fresh buffer that is the same type used to just rinse the sensor.
11. Once readings stabilize, use the **keypad** to enter the new value and press **Start/Done/OK**.
12. Record the displayed pre-calibration pH and mV values displayed in the log sheet and follow screen prompts to calibrate the buffer value.
13. ***If calibrating with the third buffer repeat steps 4-9***.

E. Multiprobe Turbidity Calibration

Optical DO Probe Note: Remove the Optical DO probe or central wiper prior to calibrating if the turbidity standard contains formalin or sodium sulfide. ***Do not remove any dedicated wiper that is on the turbidity sensor.***

1. Check to make sure the turbidity probe and wiper are clean and free from any material.
2. Use a lint free tissue to remove any deposits on the sensors and in the calibration cup.
3. Rinse the calibration cup and sensors three times with lab water to remove any debris.
4. Active the wiper to make sure it is wiping and parking correctly.
5. Slowly fill the calibration cup ⅓ to ½ full with the 0 NTU standard (lab grade water or 0 NTU standard). Avoid adding bubbles while filling. Immerse the sonde in the water with the sensors pointing down with one turn on the cup threads to allow air passage**.**
6. Make sure the sensor does not have bubbles trapped next to the wiper. If so, use the **keypad** to activate the wiper to remove any bubbles.
7. Go to the calibration menu and select **Turbidity 2-point calibration**
8. Once readings are stable (no changes in 10 seconds) input the value 0 NTU and press **Enter/OK** to calibrate. Record pre and calibrated vales in the log book
9. Remove the sensor and dump out the 0 standard. Rinse the sensors and calibration cup three times with used turbidity standard.
10. For two minutes slowly swirl to mix fresh turbidity standard bottle. **DO NOT shake.**
11. Slowly fill the calibration cup 1/3 to ½ full with fresh standard. Avoid adding bubbles while filling. Immerse the sonde in the water with the sensors pointing down with one turn on the cup threads to allow air passage**.**
12. Make sure the sensor does not have bubbles trapped next to the wiper. If so, use the **keypad** to activate the wiper to remove any bubbles.
13. Once readings are stable (no changes in 10 seconds) input the turbidity standard value press **Enter/OK** to calibrate. Record pre and calibrated values in the log book.
14. Dump the second standard in the rinse container and rinse the probes and calibration cup three times with tap water. Replace removed optical DO or central wipers.

F. Multiprobe Chlorophyll Calibration

Note: Below instructions apply only to YSI EXO Total Algae sensors**.**

The EXO Total Algae sensor contains two sensors. One measures Chlorophyll and the other Chlorophyll RFU (blue green).

Calibration Note: YSI recommends performing a 1 point (zero) calibration using lab grade water. If calibration using Rhodamine dye is required, proceed as outlined below.

Chlorophyll ug/L- Used for spot sampling to estimate presence and concentration of chlorophyll levels such as determining presence of algae blooms.

1. Rinse the calibration chamber three times with lab grade water.
2. Fill the calibration chamber with lab grade water. Check to make sure no air bubbles are present on the sensor.
3. In the **Calibrate** menu, select **BGA-PC/Chlor**, then select **Chl ug/L**. Select **2 point** (blank and slope calibration).
4. Enter **0** for the lab water blank and **66** for the second standard
5. Select **Start Calibration** and observe readings under **Current** and **Pending** data points.
6. After readings stabilize (~40 seconds) click **Apply** to accept the blank calibration.
7. Remove the sensors and drain out the lab grade water. Rinse twice with 625 ug/L Rhodamine WT standard and fill to the first line of the calibration chamber.
8. Insert the sensor and remove any air bubbles. Click **Proceed** on the display and observe readings. When readings are stable (~40 seconds) click **Apply** to accept the calibration.
9. Click **Complete** and view the calibration summary screen and QC score. Record readings on the log sheet.
10. Click **Exit** and use the arrow keys to return to the main calibration menu. Rinse the sensor and chamber three times with lab grade water.

Temperature affects the intensity of Rhodamine dye used to calibrate chlorophyll sensors. Use the chart below to verify chlorophyll calibration and end of day checks.

Temp	Chl ug/l	Temp	Chl ug/l	Temp	Chl ug/l
30	56.5	22	66	14	76
28	58.7	20	68.4	12	78.6
26	61.3	18	70.8	10	81.2
24	63.5	16	73.5	8	83.8

Blue-green Algae Phycocyanin- Used for spot sampling to estimate presence and concentration of blue-green algae blooms.

Calibration Note: YSI recommends performing a 1 point (zero) calibration using lab grade water. If calibration using Rhodamine dye is required, proceed as outlined below.

1. Rinse the calibration chamber three times with lab grade water.
2. Fill the calibration chamber with lab grade water. Check to make sure no air bubbles are present on the sensor.
3. In the **Calibrate** menu, select **BGA-PC/Chlor**, then select **BGA ug/L**. Select 2 point (blank and slope calibration).
4. **Enter 0** for the lab water blank and **16** for the second standard
5. Select **Start Calibration** and observe readings under **Current** and **Pending** data points.
6. After readings stabilize (~40 seconds) click **Apply** to accept the blank calibration.
7. Remove the sensors and drain out the lab grade water. Rinse twice with 625 ug/L Rhodamine WT standard and fill to the first line of the calibration chamber.
8. Insert the sensor and remove any air bubbles. Click **Proceed** on the display and observe readings. When readings are stable (~40 seconds) click **Apply** to accept the calibration.
9. Click **Complete** and view the **Calibration Summary** screen and **QC Score**. Record readings on the log sheet.
10. Click **Exit** and use the arrow keys to return to the main calibration menu. Rinse the sensor and chamber three times with lab grade water.

Temperature affects the intensity of Rhodamine dye used to calibrate blue-green sensors. Use the chart below to verify chlorophyll calibration and end of day checks.

Temp	Chl ug/l	Temp	Chl ug/l	Temp	Chl ug/l
30	11.4	22	16.0	14	20.1
28	13.1	20	17.1	12	21.2
26	14.1	18	17.5	10	22.2
24	15.0	16	19.1	8	22.6