STANDARD OPERATING PROCEDURES MANUAL

FOR THE

DEPARTMENT OF ENVIRONMENTAL QUALITY

WATER MONITORING AND ASSESSMENT PROGRAM

Commonwealth of Virginia
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CHAPTER 1: PREPARATION **7**

CHAPTER 2: CLEANING AND PREPARATION OF SAMPLING EQUIPMENT **8**

2.1. SAMPLING EQUIPMENT 8
2.1.1. ***General Sampling Equipment Storage and Transport*** ***8***
2.1.2. ***General Water Sampling Equipment*** ***8***
2.1.3. ***Sediment Sampling Equipment*** ***9***
2.2. SAMPLING EQUIPMENT PREPARATION AND CLEANING 9
2.2.1. ***Water Sampling Equipment Cleaning and Maintenance*** ***9***
2.2.2. ***Sediment Sampling Equipment Cleaning and Maintenance*** ***10***
2.3. GENERAL SAMPLE CONTAINER HANDLING AND PRESERVATION 10
2.4. SAMPLE IDENTIFICATION 11
2.5. MUFFLING SAMPLE FILTERS 12
2.6. CHEMICAL PRESERVATIVES AND REAGENTS 13
2.6.1. ***Chemical Preservatives and Reagents Disposal*** ***13***
2.7. LABORATORY GLASSWARE CLEANING 15
2.8. LAB REAGENT PREPARATION 16
2.8.1. ***General*** ***16***
2.8.2. ***Stock Solution Preparation Using Powder Reagents*** ***16***
2.8.3. ***Lab Solution Preparation Using Liquid Reagents and Flask*** ***17***
2.8.4. ***Lab Solution Preparation Using Liquid Reagents and Pipette*** ***17***
2.8.5. ***Proper Filling and Mixing Using Volumetric Flasks*** ***18***

CHAPTER 3: CALIBRATION AND MAINTENANCE OF FIELD MULTIPROBES **19**

3.1. GENERAL CALIBRATION ITEMS 19
3.1.1. ***Controlled Environment*** ***19***
3.1.2. ***General Calibration Guidelines and QA limits*** ***19***
3.1.3. ***General Transport of Probeware*** ***20***
3.2. QUALITY ASSURANCE OF FIELD PROBES 20
3.2.1. ***Midday DO Calibration Confirmation*** ***20***
3.2.2. ***End of Day Checks of Probes*** ***21***
3.2.3. ***Annual Probe Performance Check*** ***23***
3.2.4. ***Annual Temperature Check*** ***23***
3.3. ACCUMENT AP AND ORION SERIES HANDHELD pH/mV/ION METER 25
3.3.1. ***General procedures*** ***25***
3.3.2. ***Instrument setup*** ***25***
3.3.3. ***Calibration*** ***25***
3.3.4. ***Field measurement procedures*** ***26***
3.3.5. ***Maintenance*** ***27***
3.4. YSI MODEL 58 DISSOLVED OXYGEN AND TEMPERATURE METER 28
3.4.1. ***Calibration*** ***28***
3.4.2. ***Field Measurement Procedures*** ***29***
3.4.3. ***Maintenance*** ***29***
3.5. YSI 55 HANDHELD OXYGEN AND TEMPERATURE METER 30
3.5.1. ***Calibration*** ***30***
3.5.2. ***Taking Measurements*** ***30***
3.5.3. ***Maintenance*** ***30***
3.6. YSI 85 HANDHELD OXYGEN, CONDUCTIVITY, SALINITY AND TEMPERATURE METER 31
3.6.1. ***Calibration*** ***31***
3.6.2. ***Taking Measurements*** ***32***
3.6.3. ***Maintenance*** ***32***
3.7. HYDROLAB DATASONDE AND MINISONDE WITH SURVEYOR 34
3.7.1. ***Calibration*** ***34***
3.7.2. ***Preparation for use*** ***38***

3.7.3. Data Recording *38*
3.7.4. Data Logging *38*
3.7.5. File transfer from Sonde to PC *39*
3.7.6. Storing data in clipboard *40*
3.7.6.1. Reviewing clipboard scans *40*
3.7.6.2. Deleting clipboard scans *40*
3.7.7. Maintenance *40*
3.8. HYDROLAB H2O WATER QUALITY MULTIPROBE WITH DISPLAY 41
3.8.1. System components *41*
3.8.2. Calibration *41*
3.8.3. Transport to the Field *44*
3.8.4. Field set-up and operation *45*
3.8.5. Maintenance *45*
3.9. HYDROLAB QUANTA 46
3.9.1. Calibration *46*
3.9.2. Data Recording *47*
3.9.3. Maintenance *47*
3.10. YSI 6-SERIES MULTIPROBE SONDE 50
3.10.1. Calibration *50*
3.10.2. Data Recording *55*
3.10.3. Logging Data *55*
3.10.4. Discrete sample measurement logging to MDS 650 ..... *55*
3.10.5. Deploying Sonde for Unattended Logging *56*
3.10.6. Post Sampling Verification and Data Evaluation *57*
3.10.7. Data and Records Management *58*
3.10.8. Maintenance *58*
3.11. IN-SITU MULTIPROBE TROLL 9500 WITH RUGGED READER 61
3.11.1. Calibration *61*
3.11.3. Logging profiler data *64*
3.11.4. Maintenance *64*

CHAPTER 4: FIELD SAMPLING PROCEDURES **67**

4.1. USE OF PROTECTIVE GLOVES 67
4.2. EQUIPMENT RINSE 67
4.3. SURFACE WATER SAMPLING 67
4.3.1. General *67*
4.4. SAMPLING FROM A BRIDGE 69
4.4.1. General *69*
4.4.2. Bacteria Sampling Using a Modified Stainless Steel Bucket *69*
4.5. STREAMBANK AND INSTREAM SAMPLING 70
4.5.1. General *70*
4.5.2. Instream Sample Collection *71*
4.5.3. Streambank Sample Collection *71*
4.6. SAMPLING FROM A BOAT 72
4.6.1. General *72*
4.6.2. Collection of Samples With a Pump and Hose *73*
4.6.3. Secchi Disk Measurements *74*
4.6.4. Light Attenuation *74*
4.7. VACUUM FILTERING METHOD (IN-LINE FILTERING) 78
4.7.1. Supplies Needed *78*
4.7.2. Filtration of Samples *81*
4.8. CHLOROPHYLL A COLLECTION USING SYRINGE FILTRATION 86
4.9. SEDIMENT SAMPLING 87
4.9.1. Sampling Methodology *87*
4.9.2. Sampling Location and Substrate Selection *87*
4.9.3. Collecting Sediment with a 'Dredge' Type Sampling Device *87*

4.9.4. Collecting Sediment with the 'Scoop and Pan' Method *90*
4.10. POLLUTION RESPONSE PROGRAM SAMPLING PROCEDURES (PREP) 91
4.10.1. Cyanide *91*
4.10.2. Sulfide *91*
4.10.3. Pesticides and Herbicides *91*
4.10.4. Purgeable Organic Compounds (Volatiles) *92*
4.10.5. Volatile Aromatic Hydrocarbons Including BTEX in Water *92*
4.10.6. Base/neutrals and Acid Extractables (Semivolatiles) *93*
4.10.7. Petroleum Identification and Quantification in Water Samples *93*
4.10.8. Total Petroleum Hydrocarbon (TPH) in Water Samples *93*
4.10.9. Sampling when Petroleum Product is Unknown *94*
4.10.10. Toxicity Sampling *94*
4.10.11. Total Petroleum Hydrocarbon and BETX Identification in Soil Samples *95*
4.10.12. Sample Packing and Shipping *95*
4.11. COLLECTION OF TRACE ELEMENT SAMPLES (CLEAN METALS) 95
4.11.1. Scope *95*
4.11.2. Applicability *95*
4.11.3. Summary *96*
4.11.4. Significance and Use *96*
4.11.5. Equipment Preparation and WQM Scheduling of Sample Kits *97*
4.11.6. Equipment and Supplies *98*
4.11.7. Collection Protocol for Freshwater and Saltwater Using the Bridge Bottle *99*
4.11.8. Effluent Sample Collection Protocol *104*
4.11.9. Sample labeling *105*
4.11.10. Sample Shipping *105*
4.11.11. Quality Control *107*
4.11.12. Clean Metals Quick Reference Guide *108*
4.11.13. Referenced Documents *109*
4.12. CHAIN OF CUSTODY PROCEDURES AND COMPLETING COC RECORD 115
4.12.1. Transferring COC of samples from person to person *115*
4.12.2. Transferring COC of samples via courier *115*
4.12.3. Priority Codes *115*
4.12.4. Preparing the COCR Form *115*
4.12.5. COCR Fields *115*
4.12.6. Using CEDS for COCR Forms *118*
4.12.7. Preparing Samples for Shipment *119*
4.12.8. Possible COCR Problems and Solutions *119*
4.12.9. The Personal Field Log *120*
4.12.10. Call List for Sample Related Issues *121*
4.12.11. Directions to DCLS *123*

CHAPTER 5: QUALITY ASSURANCE AND QUALITY CONTROL **125**

5.1. QUALITY ASSURANCE OF FIELD PARAMETERS 125
5.2. QUALITY CONTROL SAMPLES FOR AMBIENT SAMPLES 125
5.2.1. General *125*
5.2.2. Equipment Blanks for General Water Quality Parameters *125*
5.2.3. Field Split Samples for General Water Quality Parameters *126*
5.3. QUALITY CONTROL SAMPLES FOR SEDIMENTS 127
5.3.2. Field split sediment samples *128*

CHAPTER 6: SAMPLE IDENTIFICATION AND CORRECTIVE ACTION **129**

6.1. FIELD DATA SHEET 129
6.2. SAMPLE LABEL AND TAG 129
6.3. CORRECTIVE ACTION 129

CHAPTER 7: SAFETY **131**

7.1. **BASIC SAFETY PREPARATION** 131
7.2. **GENERAL LABORATORY AND WAREHOUSE SAFETY** 133
7.3. **REAGENT CHEMICAL SAFETY** 133
7.3.1. ***General Safety Procedures for Handling Acids*** ***134***
7.4. **WADING** 135
7.5. **WORKING FROM BRIDGES** 135
7.5.1. ***Selecting Bridges for Sampling*** ***135***
7.5.2. ***Vehicle Parking Procedures*** ***136***
7.6. **WORKING FROM BOATS** 136
7.6.1. ***Personal Flotation Devices*** ***140***
7.7. **COLLECTING FISH** 142
7.7.1. ***Electrofishing*** ***142***
7.7.2. ***Handling Fish*** ***143***
7.8. **CONTAMINATED WATER** 143
7.9. **WEATHER** 143
7.9.1. ***Lightning Safety*** ***143***
7.9.2. ***Temperature Exposure*** ***143***
7.10. **HAZARDOUS PLANTS AND ANIMALS** 145
7.11. **REFERENCES** 147

APPENDIX A: CALIBRATION LOGSHEET FOR MULTIPROBES **148**

APPENDIX B: THEORETICAL DISSOLVED OXYGEN CHART **154**

APPENDIX C: CORRECTIVE ACTION REQUEST FORM **157**

APPENDIX D: ENTERING QA/QC INTO CEDS **160**

APPENDIX E: DISSOLVED OXYGEN USING MODIFIED WINKLER METHOD **163**

Introduction to Water Quality Assessments
Operating Procedures Manual

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, make a checklist of all routine material and equipment you will need for sampling to help you 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. A 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 as outlined in Chapter 7
6. Cell phone or other form of emergency communication.
7. Verify that all sampling equipment is clean, in good working condition and the batteries are charged.

Before leaving on a sample run, let your immediate supervisor or designated contact in your office know where you will be, when you are expected to return and how to contact you if you are overdue.

Calibrate all field 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

Different sampling equipment requires different cleaning methods based on its use in the field. Non-metallic materials, such as plastic or Teflon, are used whenever possible for the collection of samples for metals. For the collection of organic samples, non-organic or inert materials, such as stainless steel or Teflon, are used.

2.1.1. General Sampling Equipment Storage and Transport

The majority of sample sites are sampled using grab sampling equipment. To ensure the highest quality of data results, this equipment must be well maintained. Below are several general items to follow when transporting grab sampling equipment out into the field.

1. 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

2. Examine the equipment for obvious signs of dirt, rust or scratches, and replace when necessary. Dirt, rust, and scratches can contaminate samples by allowing dirt and bacteria to survive cleaning or allow residue to contaminate sensitive samples.

3. Look at sample containers to see if cracks or contamination is present. If so, dispose the container in question and obtain a replacement.

4. Bulk shipments of plastic sample containers often have lids separate from the container. While in storage, it is best to keep any open boxes covered or closed to reduce dust and other contaminants from entering the exposed containers or lids.

5. When assembling plastic sample containers, the use of clean hands or powder free gloves will reduce potential contamination from entering the bottle.

2.1.2. General Water Sampling Equipment

1. Rope on spool

2. An appropriately sized stainless steel bucket with a fitting for the bacteria sample bottle mounted on the inside, or a suitable water sampling device (Van Dorn, Kemmerer, Labline, pump and hose or HDPE Nalgene bottle etc.)

3. Clean sample bottles and/or cubitainers suitable for the samples being collected.

4. Syringe, filter paper, filter holder etc. for samples requiring filtering.

2.1.3. Sediment Sampling Equipment

1. Rope on spool

2. Certified pre-cleaned glass jar(s) with Teflon-lined lid

3. Teflon coated or plastic spoon, and stainless steel spoon

4. Appropriate dredge (such as Petite Ponar) depending on sediment type and depth of water

5. Appropriately sized stainless steel pan

2.2. Sampling Equipment Preparation and Cleaning

Items outlined below cover routine sampling encountered by field teams. If sampling for compounds or water matrix not covered in this section, please contact the Quality Assurance Coordinator.

2.2.1. Water Sampling Equipment Cleaning and Maintenance

At the end of each sampling day:

1. Rinse sampling buckets at the end of the sampling day with DI water and allow to air dry at room temperature.
    a. If rinsing the bucket does not remove buildup before the weekly cleaning schedule, clean bucket with lab grade soap as directed in the weekly schedule.

2. If a pump and hose apparatus is used, pump 5 gallons of DI water through the pump and hose system and completely drain.

3. If using a Kemmerer or Alpha Bottle sampling device follow the manufacturer's recommendations for cleaning those sampling devices using DI water.

Weekly maintenance:

1. Wash sampling buckets with lab grade soap (Liquinox or Alconox) at the end of each week using a brush to ensure the removal of all particulate matter or surface film.

2. Rinse thoroughly with tap water, then DI water, and allow to air dry at room temperature.

3. If sampling buckets have rust stains or other hardened deposits, use a paste made of baking soda and water to scour the deposits using a soft brush or clean cloth. Scour in the direction of the grain of the steel. After cleaning, repeat steps 1 and 2.

Monthly maintenance:

1. Pump 5 gallons of a 5% vinegar solution (5% solution consists of 1 quart of concentrated acetic acid mixed with a 4 ¾ gallons of water) through the hose and pump apparatus.

2. Pump 5 gallons of DI water through the hose and pump apparatus and completely drain.

3. If hose appears worn, discolored, or has biological growth inside, replace immediately.

Annual maintenance:

1. Replace hoses of pump and hose sampling devices.

2. Perform routine maintenance on pump as specified in the manufacturer's manual.

2.2.2. Sediment Sampling Equipment Cleaning and Maintenance

At the end of each sampling day:

1. Wash equipment thoroughly with clean scrub brushes using Alconox™ powdered or Liquinox™ liquid detergent.

2. Rinse with DI water until all residues are removed.

3. Repeat washing procedure using Citranox™.

4. Rinse with DI water until all residues are removed.

5. Rinse with pesticide grade ethanol or methanol to remove organic compounds.

6. Rinse thoroughly with DI water until all ethanol or methanol is removed.

7. Dry equipment at room temperature away from potential sources of contamination.

8. Visually inspect equipment for any contamination prior to storage. Such contamination would include water spots, dust or sediment, rust and similar substances.

9. Cover the clean equipment with clean aluminum foil until use.

2.3. General Sample Container Handling and Preservation

Proper sample containers and sample preservation are essential to sample integrity. Refer to the DCLS laboratory catalog in CEDS for the appropriate preservation procedures. Samples not preserved properly may be rejected by DCLS.

- Containers purchased by DEQ are parameter and program specific to meet agency and DCLS requirements for sample size and purity of container construction and material.

- 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.

- Inspect sample containers prior to use for tears, punctures, cracks, or foreign material inside the container and discard if present.

- After collecting the sample, make sure the lids are secured tightly to prevent water from seeping in or out of the container.

- Sample containers and coolers should be stored with the tops securely fastened. Replace or tape containers with loose fasteners to prevent loss of sample containers.

- DCLS provides temperature bottles that they use to determine sample temperature upon arrival at DCLS. Make sure that every cooler used to ship samples to DCLS contains one of these bottles.

- In the field, unless specified otherwise, place all samples in an ice filled cooler immediately after collection. To ensure samples do not exceed the 4°C holding temperature, place sample containers upright and if possible, covered with ice in such a manner that the container openings are above the level of ice. Transport chlorophyll a filter pad samples in an appropriately sized Ziploc bag and placed on top of the layer of ice. Ziploc bags containing filters should either be double bagged or oriented so that the sealed opening of the Ziploc bag hangs outside the cooler lid when the lid is closed. Bacteria sample bottles should be stored in mesh bags, placed in coolers and surrounded with wet ice.

- It is recommended to wrap glass sample containers using bubble wrap or other waterproof protective materials to minimize accidental breakage.

- Prior to shipping, drain any ice water from the cooler and refill 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:

1. Station ID
2. Sample date and time (in military time)
3. Sample depth,
4. Collector initials
5. Parameter group code
6. Lab processes code (if applicable)
7. Container number (see below)
8. Preservative used (if any)
9. Volume filtered (if applicable).

For most sample containers, it is acceptable to use a laser printer to print sample identification information on an adhesive Avery® label and applied directly to the exterior of the container. For cubitaners and similar sample containers where an Avery label will not stick, the label may be affixed to a wire tag. Fasten the tag to the container using the provided wire in such a way to prevent it from coming off. The best location to wire tags is between the lower lip and shoulder the sample container

Record all hand written information on sample tags using indelible ink to prevent running if the tag gets wet. Sharpie® permanent markers with a fine or ultra fine tip provide an excellent water resistant mark for sample labels.

Information on the sample tag must match exactly what is scheduled in CEDS field data screen. Samples will not be analyzed if information posted on the sample bottle does not exactly match what is entered into CEDS. 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, 3 of 3, etc., as required.

The sample time must exactly match what is entered into CEDS. Failure to do so will result in DCLS invalidating the sample. 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.

Labels for established sampling sites should be printed from CEDS using a laser printer. Ink jet printers often result in the tags smudging when wet. If the site is not established, the needed tag information must be hand printed with indelible ink on a blank adhesive label and then affixed to the container tag as necessary.

It is absolutely imperative that the actual sampling site match the labeling information. **Always check the labeling information against the actual site.**

Some sample types may have specific labeling requirements. Those requirements are detailed with the sampling guidelines usually provided upon receipt of the sample bottles.

Samples not labeled properly may be rejected by the laboratory.

2.5. Muffling Sample Filters

Certain samples, such as Particulate Nitrogen and Carbon (PNC), require muffling of glass fiber paper before use. This muffling is to remove any contaminants present on the filter paper due to dust or during the manufacturing process.

1. Set the muffle furnace to 500-550°C. Do not set above 550°C as the filter will likely become fragile and easily tear during sample collection.

2. Place the required number of filters on an aluminum foil tray or in a porcelain crucible. One filter is required for each sample. In addition, prepare sufficient extra dry muffled filters necessary for DCLS to determine background contaminants and to have spares in the event of filters tearing while in the field.

3. Once the furnace reaches 500-550°C, place the tray or crucible containing the filters in the muffle furnace, close and latch the door.

4. Bake the filters for one hour.

5. After one hour, remove the filters from the furnace and allow them to cool in a desiccator with activated desiccant until they are cool.

6. Place the filters in a plastic snap-lid box with activated desiccant.

7. Replace/recharge desiccant as needed. Desiccant should have color indicators to show that it is of sufficient strength to remove water vapor. Typically, this indicator will be a blue color that will slowly fade to clear or pale blue, purple, or pink.

   a. Recharge spent desiccant by evenly spreading spent gel on a tray and placing it in a drying oven set at 100-120°C for at least an hour. Large quantities of desiccant or the "rock" type desiccant may require overnight drying. Place new or recharged desiccant in a tightly sealed container to prolong shelf life.

2.6. Chemical preservatives and reagents

Each regional office is responsible for maintaining an adequate supply of chemical reagents to preserve samples and clean sampling equipment. The use of expired or contaminated reagents will result in inadequate preservation or contamination of samples. Regions should follow the guidelines below to prevent this from occurring.

- ACS regent grade preservatives are required for sample preservation.

- Upon receipt of fresh preservative or reagents, regional staff should note the expiration date provided on the bottle. If no date is listed, regions should record the date the chemical was received on the bottle label using indelible ink. Table 2.1 contains a list of typical shelf life for reagents routinely used by regional staff.

- Bottles should be dated when first opened to ensure they are consumed quickly.

- When not in use, reagent bottles should be stored in the appropriate safety cabinet. Reagents should never be stored in the open as the risk of contamination or light attenuation will degrade the quality of the reagent. Bottles containing solid reagents in opaque bottles may be stored on a shelf or laboratory cabinet.

- Whenever handling chemicals of any kind, always follow safe laboratory techniques including the use of eye protection, gloves and aprons as appropriate and wash your hands afterwards. Chapter 7 contains additional information on handing routinely used reagents by field staff.

2.6.1. Chemical Preservatives and Reagents Disposal

- Segregate and dispose of laboratory wastes according to federal, state, and local regulations.

- Soap solutions and waste tap/DI/analyte-free water can be poured down the drain.

- Diluted solvents and weak acids used in cleaning may be poured down the drain after neutralization and additional dilution with tap water.

- High strength solvent and acid waste should be handled as hazardous waste and must be collected and disposed of properly according to federal, state, and local regulations. Refer to Chapter 7 if there are problems with proper disposal of high strength or other waste generated by activities covered by this document.

Table 2.1: Typical Shelf Life of Reagents

Compound Type	Name of Reagent	Shelf Life	Recommended Storage Area	Discard if observe the following
Acid	Concentrated Acetic acid	3 years	Acid cabinet	Color other than clear (typically yellow) and/or solids observed in the container
	Conc. hydrochloric acid			
	Conc. sulfuric acid			
	Conc. nitric acid		Oxidizer or acid cabinet. Do not store with organics.	
Base	Conc. sodium hydroxide or pellets	5 years	Base (Alkaline) cabinet. Pellets may be stored on a closed container on a laboratory shelf	Liquid becomes cloudy/ discolored or solids observed in the container Pellets clump together and cannot break apart easily
Solvents/ Organics	Acetone	5 years	Flammable cabinet	Color other than clear or solids observed in the container
	Conc. ethanol or methanol			
	Formalin (formaldehyde)	3 years		
Inert	Potassium chloride crystals	5 years	Laboratory shelf away from light (if bottle is not opaque)	Powder clumps and cannot break apart by shaking
	Sodium thiosulfate crystals			
	Magnesium carbonate			
	pH buffer	12-18 months	Laboratory shelf or storeroom	Cloudy or suspended solids
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

2.7. Laboratory Glassware Cleaning

From time to time, laboratory glassware (specifically volumetric flasks and glass graduated cylinders) will become dirty despite routine cleaning methods. This 'dirty' glassware will show spots of water along the inner wall of the glassware when lab grade water is applied. The only way to ensure proper cleaning of such glassware is to use high strength acids and flush with lab grade water. Follow safety procedures outlined in Chapter 7 of this manual for handling concentrated acids and the instructions below to clean glassware safely and effectively.

1. Before starting the acid washing process, clean the glassware.
    a. Pour a small quantity of lab grade, phosphate free, soap such as Liquinox® and tap water into the glassware. Using a brush or by capping the opening with a stopper or gloved hand and allow the soap to thoroughly contact the entire inner glass surface. Pipettes can be cleaned by placing in a pipette cleaner or by hand washing.
    b. Dump the soapy water and use tap water and rinse with tap water three times.
    c. Rinse the glassware three times using DI or lab grade water.

2. After washing, note if water spots form. If not, the glassware is clean and does not need acid washing. If you still observe water spots, proceed with the next step.

3. Using the fume hood, pour 10-30 ml of 50% or concentrated sulfuric acid into the flask or draw up an amount to coat the inner surface of the pipette. Sulfuric acid is better than nitric or hydrochloric acids, as it does not produce as much toxic fumes which could overpower the technician.

4. Allow the acid to coat the inner surface by slowly rotating the glassware. Be sure to cap volumetric flasks so that the acid can coat the entire inner surface.

5. When the acid has coated the surface sufficiently you will note the liquid will appear to have a smooth sheen. If you see ripples in the coating, the acid has not completely digested the contaminants.

6. Once coating the inner surface sufficiently, slowly pour out the contents into an acid waste container or neutralize in a bucket containing sodium hydroxide and water or baking soda and water. If neutralizing, periodically check pH using litmus paper. If the pH is below 4.00, slowly add more sodium hydroxide or baking soda to raise pH.

7. Rinse the glassware using three, 20-50 ml successive rinses 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 down from the top hole of the pipette.

8. Observe the glassware to see if water spots form. If so, repeat steps 3-7. Otherwise, allow the glassware to dry completely and cap using a flask cap or aluminum foil to prevent dust from entering the cleaned container. Store glassware in the cabinet until needed.

2.8. Lab Reagent Preparation

Depending on the equipment used and parameter monitored, it is necessary to prepare a working set of reagents. To ensure accurate preparation, it is necessary to follow the following steps.

2.8.1. General

1. If a liquid standard is purchased from a supplier, be sure to check that the expiration date and condition of the standard is still good prior to using it. If using a powder standard, be sure that it is within the printed expiration date and is free flowing.

2. If the powder reagent is clumpy and is within the expiration date, it will require drying to ensure accuracy.
   a. Place an approximate amount of powder reagent needed to prepare the reagent solution in a clean aluminum pan or ceramic crucible.
   b. Place this pan into a drying oven set at 100-110 C and leave in for at least 3 hours.
   c. Remove the pan from the oven and place in a desecrator which contains activated desiccant (usually blue silica gel) until ready for use
   d. Just before use, check that the powder no longer clumps by using a clean lab spatula

3. The dilution water should be lab grade water which is produced onsite. Contact the Quality Assurance Coordinator if you suspect problems with the water purification system.

4. Table 2.2 lists commonly used reagent solutions used by the regions and the amount of stock reagents and dilution water needed. When weighing or measuring stock reagents, the use of calibrated analytical balances or class A volumetric glassware is necessary.

2.8.2. Stock Solution Preparation Using Powder Reagents

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

1. Turn on the electric scale and allow 10 minutes to warm up.

2. Verify the scale is balanced and on a vibration resistant mounting such as a slab table. Most scales have a bubble balance to allow you to adjust to a level surface.

3. Verify the accuracy of the scale using a certified weight. Ideally, the certified weight should be similar in weight to the standard being measured. The scale must measure the certified weight to within +/- 0.001 g of the weight value. Calibrate if necessary by following manufacturer instructions.

4. After verifying the accuracy of the scale, place a clean, empty weighing dish or crucible onto the scale and close the scale door.

5. After the scale stabilizes, press the 'Tare' or 'Zero' button. The unit will display 0.0000 g. Remove the dish after zeroing the scale.

6. Using a clean beaker, measure out an approximate amount of powdered reagent needed.

7. Using 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. When you are close to the desired weight, close the scale door to ensure accuracy.

8. When the weight is correct, remove the pan containing the reagent powder and carefully pour into a clean volumetric flask using a clean funnel and rinse all residue 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.

9. Proceed to 2.8.5

2.8.3. Lab Solution Preparation Using Liquid Reagents and Flask

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

1. Ensure the volumetric flask you will measure the stock liquid reagent is clean by pouring ~10 ml of reagent to observe any spotting on the stem of the flask. Discard this rinse.
    a. If spotting is observed, use new clean volumetric glassware or clean the current volumetric glassware using the procedures outlined in section 2.7

2. If the flask is clean, fill with reagent so that the bottom of the meniscus (U shaped depression) rests on the etched line of the flask stem. If the meniscus is above the line, drain a small portion or use a clean pipette to withdraw the amount and refill as necessary. Do not place discarded reagents back in the reagent bottle.

3. Drain the entire reagent contents of the flask into the larger preparation volumetric flask and rinse the smaller flask with at three, 10-20 ml rinses of lab grade water. Allow rinses to flow into the larger flask. Ensure that rinses cover the entire inner surface of the flask.

4. Proceed to 2.8.5

2.8.4. Lab Solution Preparation Using Liquid Reagents and Pipette

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

1. Ensure the pipette is clean by filling to the printed measurement line of the pipette with the stock solution. Drain the contents into a waste container or sink and check that the pipette does not hold water droplets. If it is dirty, obtain a new pipette or clean the pipette 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 is marked with TC (To Contain), blow out all contents of the pipette. If the pipette is marked with TD (To Dispense), do not blow out.
    a. If using a TC pipette, rinse pipette by flushing with lab grade water.
        i. Flush using a rinse bottle with a narrow nozzle through the hole on top of the pipette where the pipette bulb was attached.

ii. Rinse with 3 squirts of DI water and allow rinses to drain into the flask.

b. If using TD pipette, DO NOT rinse the pipette.

4. Proceed to 2.8.5

2.8.5. Proper Filling and Mixing Using Volumetric Flasks

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 you overfill, discard the solution as it is now more diluted than required.

4. Cap and invert the flask a minimum of 20 times to ensure proper mixing. Label the flask or container which will hold the standard with the strength of the prepared solution. Include the initials of the technician and the date the solution was made.

Table 2.2: Preparation of Commonly Used Stock Solutions per Liter

Stock Solution	Uses for	Powder Reagent Needed or	Liquid Reagent Needed	DI Water Volume
50% Sulfuric acid	Sample preservative, cleaning agent	N/A	500.0 ml concentrated sulfuric acid (Add 300 ml water to the dilution flask first!)	Bring to 1.0 L volume
10% Hydrochloric acid	Cleaning agent	N/A	100.0 ml concentrated hydrochloric acid. (Add 300 ml water to the dilution flask first!)	
1 M KCl	Stock solution for standards	74.551 grams dried KCl powder	N/A	
0.5 M KCl	58,670 uS/cm standard	37.276 grams dried KCl powder	500.0 ml of 1 M KCl	
0.1 M KCl	12,880 uS/cm standard	N/A. Too difficult to weigh accurately	100.0 ml 1 M KCl	
0.05 M KCl	6,670 uS/cm standard	N/A. Too difficult to weigh accurately	100.0 ml 0.5 M KCL or 50.0 ml 1 M KCl	
0.01 M KCl	1,413 uS/cm standard	N/A. Too difficult to weigh accurately	100.0 ml 0.1 M KCl or 10.0 ml of 1 M KCl	
0.005 M KCl	718 uS/cm standard	N/A. Too difficult to weigh accurately	100.0 ml 0.05 M KCl or 10.0 ml of 0.5 M KCl	
0.001 M KCl	147 uS/cm standard	N/A. Too difficult to weigh accurately	100.0 ml 0.01 M KCl or 10 ml of 0.1 M KCl	
10 g/L Magnesium carbonate	Chlorophyll a preservative	10 grams magnesium carbonate	N/A	

CHAPTER 3: CALIBRATION AND MAINTENANCE OF FIELD MULTIPROBES

3.1. General Calibration Items

This chapter covers the calibration and use of electronic field parameter equipment. Although the use of equipment varies by region and program, all units should perform the same general calibration and end of day check procedures.

3.1.1. Controlled Environment

Calibrate sensors in a controlled environment such as in the designated field preparation room. Avoid calibrating units in the field since it can introduce error. If calibration must be performed in the field, it should be conducted indoors or in an area that is close to room temperature. Allow the probe to stabilize before calibrating. A probe is considered stable if the readout does not significantly change ($\leq$0.1 units) within ten seconds.

3.1.2. General Calibration Guidelines and QA limits

Calibrate using standards that are within the printed expiration date or within six months of the date of opening/date of preparation if no expiration date is printed. Calibrate probes each day the units go into the field. Table 3.1 outlines calibration tolerances to reference standards.

3.1.2.1. Temperature verification

Once every three calibrations of the field probe, compare the temperature readout to a laboratory thermometer which was checked by the DEQ master thermometer within the past year. Record the both temperature measurements on the log sheet. If the temperature difference is greater than 0.5 C, notify the QA Coordinator.

3.1.2.2. Conductivity Calibration

For specific conductance, staff should calibrate using a conductivity solution that is close to the expected values encountered in the field. Be sure to rinse the probe with DI water and then the conductivity solution before calibrating with fresh conductivity solution. After calibration, the probe should be within 1.0% of the buffer value used.

3.1.2.3. Clark Cell Dissolved Oxygen Calibration

For dissolved oxygen calibration, calibrate or verify probes using procedures outlined in this chapter based on the probe make and model. After calibration, the readout must be within ± 0.2 of the theoretical dissolved oxygen saturation calculated using the table provided in Appendix B.

3.1.2.4. Optical Dissolved Oxygen Calibration

Daily calibration of optical dissolved oxygen sensors is necessary, as they are prone to damage if not properly stored. After calibration, the probe must be within $\pm$0.1 mg/l of the theoretical dissolved oxygen saturation calculated using the table provided in Appendix B.

3.1.2.5. pH Calibration

For pH calibration, use freshly prepared pH 7.0 buffer and pH 4.0 and/or pH 10.0 buffers depending on the expected values encountered in the field. For example, when performing saltwater monitoring, pH values are assumed to be above 7.0 so calibrating with 7.0 and 10.0 buffer is acceptable.

If the expected pH is unknown, whenever possible, sample teams should calibrate pH probes with 4.0, 7.0, and 10.0 buffers if the instrument is capable of such calibration. Probe readouts must be within ± 0.2 S.U. of the buffer value.

At least once per month it is important to verify the calibration of a two buffer calibrated probe (4 and 7 or 7 and 10) by immersing the probe in the third buffer not used to calibrate the sensor to determine if the calibration curve would accurately display pH at this range. If the probe cannot accurately read this value, servicing is necessary.

Table 3.1 Error Limits for Initial Daily Calibration

Parameter	Value
Conductivity	± 1.0% of calibrated standard
Clark Cell DO	± 0.2 mg/L of theoretical level
Optical DO	± 0.1 mg/L of theoretical level
pH	± 0.2 S.U. of calibration buffer

3.1.3. General Transport of Probeware

Many of the sensors, such as those reading pH and dissolved oxygen, can easily dry out during transport resulting in inaccurate readings and damaged equipment. To prevent this, transport sensors in a humid environment. Ideally, attaching the sensor storage cup provided with the probeware which includes a moist sponge or 10-20 ml of pH 4 buffer will ensure probes do not dry out during transport. The 4 buffer will also reduce microbial growth on the sensors and ensure the pH sensor is not compromised, as would be the case with tap or lab grade water. If using water in the storage cup, avoid letting the water touch the sensors as much as possible. Remove the storage cup and affix the sensor guard (if provided) before deploying at the site.

Transport calibrated instruments, such as multiprobe equipment, in a controlled environment most associated with the conditions the unit was calibrated in. Typically, on hot (air temp >30 ^{O}C) or cold (air temp <10 ^{O}C) sample days, it is recommended to transport the multiprobe in the passenger compartment of the vehicle. This will ensure faster response of the unit in the field and post check verification.

3.2. Quality Assurance of Field Probes

3.2.1. Midday DO Calibration Confirmation

For multiprobe units with a DO sensor, it is useful to perform a DO % saturation confirmation in the middle of run. This check will determine if the calibration of the DO sensor is still accurate to reduce the risk of invalidating the entire sample run worth of dissolved oxygen readings. To perform the midday check, place the DO sensor in a 100% air/water saturation environment such as a wet towel or in the storage cap with small amount water in it. Allow the probe to equilibrate,

record the DO % saturation on the field data sheet then transfer it to CEDS in the comment field. The reading should be 95 to 105% saturation. If DO % saturation is out of the specified range, the unit will need to be recalibrated in the field.

3.2.2. End of Day Checks of Probes

When returning from the field, teams must verify the accuracy of the field probe equipment by performing an end of day check. The end of day check is not a calibration, but a method where the probeware is verified by checking against standards in a controlled environment. If the check exceeds criteria outlined in Table 3.2, do not enter associated field data into CEDS.

3.2.2.1. Conductivity Check

Upon returning from the field, turn on the probe and allow the temperature to stabilize to room temperature. If temperature is slow to adjust due to from returning from extreme hot or cold environments, you may place the sensor(s) in a room temperature water bath for faster adjustment. Remove from the bath before proceeding with the end of day check.

1. Rinse the conductivity probe with DI water and blot dry. Ensure the reading is 0 uS/cm.

2. Immerse the probe in conductivity solution of the same strength used during calibration.

3. Allow the conductivity probe to stabilize. This may take several minutes.

4. Record the reading in the appropriate section of the calibration log sheet.

5. If the conductivity value is off more than 5% (conductivity solution <1000 uS/cm) or 10% when using higher strength conductivity solutions, conductivity readings collected during the run are considered invalid and should not be entered into CEDS.

3.2.2.2. Dissolved Oxygen Check

Upon returning from the field, turn on the probe and allow the temperature to stabilize to room temperature. If temperature is slow to adjust due to from returning from extreme hot or cold environments, you may place the sensor(s) in a room temperature water bath for faster adjustment. Remove from the bath before proceeding with the end of day check.

1. Once the temperature is stabilized, place the oxygen sensor in the calibration chamber following the instructions provided by the particular probe manufacturer or as outlined later in this chapter.

2. Allow the probe dissolved oxygen reading to stabilize. A stable reading is one that does not significantly change ($\leq$0.1 units) for ten seconds. This may take several minutes depending on the age of the sensor.

3. While the probe is adjusting, use a scrap piece of paper to record the barometric pressure reading using the calibrated barometer in the room. If the barometer is out of service, you may use the nearest weather station pressure reading following the procedures outlined in Appendix B.

4. When the probe temperature and dissolved oxygen readings are stable, record the values on the end of day check portion of the calibration log sheet.

5. Using the table in Appendix B, determine the theoretical dissolved oxygen saturation value. Record this on the calibration log sheet in the appropriate section.

6. If the difference between the observed reading and theoretical value greater than or equal to 0.5 mg/L, dissolved oxygen data collected during the day is considered invalid and should not be entered into CEDS. Service DO sensors if the end of day check is greater than 0.3 mg/L or optical DO sensors are greater than 0.2 mg/L of theoretical DO levels.

Note: Using pre-calibration dissolved oxygen values of the following day as an end day check for the proceeding day is not encouraged. Probes that would have exceed QA values at the end of day check and meeting the following day may indicate insufficient time to allow the probe to adjust properly. In addition, this additional time can mask slow performance of a poorly performing sensor.

3.2.2.3. pH Check

Upon returning from the field, turn on the probe and allow the temperature to stabilize to room temperature. If temperature is slow to adjust due to from returning from extreme hot or cold environments, you may place the sensor(s) in a room temperature water bath for faster adjustment. Remove from the bath before proceeding with the end of day check.

1. Rinse the pH probe with freshly prepared 7.0 buffer or buffer used during the morning calibration. Fill the calibration chamber (or appropriate container) with freshly prepared 7.0 buffer or clean 7.0 buffer used during the morning calibration.

2. Allow the probe pH reading to stabilize. A stable reading is one that does not significantly change ($\leq$0.1 units) for ten seconds. This may take several minutes depending on the age of the sensor.

3. Record the reading in the appropriate section of the calibration log sheet. If the probe can display millivolt (mV), record this as well.

4. Repeat steps 1 through 3 for pH 4.0 and/or 10.0 buffer. Use the same strength standards used during the morning calibration.
    a. If field pH readings were outside the standard two buffer calibration curve, verify the accuracy of the pH sensor by immersing in the buffer (4.0 or 10.0) which would have bracketed the observed field readings.

5. If the pH values for any of the calibrated buffers exceed 0.2 S.U., pH data collected during the run is considered invalid and should not be entered into CEDS.

Note: Using pre-calibration pH values of the following day as an end of day check for the preceding day is not encouraged. Probes that would exceed QA values at the end of day check and meet the following day indicates insufficient time to allow the probe to adjust

properly. In addition, this additional time can mask slow performance of a poorly performing sensor.

Table 3.2 Maximum Error Limits for End of Day Check

Parameter	Value
Dissolved Oxygen	± 0.4 mg/L of theoretical level
pH	± 0.2 S.U. of calibration buffer
Conductivity < 1000 µS/cm	± 5.0% of calibration standard
Conductivity > 1000 uS/cm	± 10.0% of calibration standard

3.2.3. Annual Probe Performance Check

Each year, the QA Coordinator or designee will perform a comparison check of field multiprobes to identify sondes that may be inaccurate in the field while passing standard calibration and end of day checks. This verification check will use the following procedure:

1. Calibrate all probes being tested using the normal calibration procedures.

2. Fill a 10 to 20 gallon container with cold or warm tap water. A clean medium garbage can could be used for this purpose.

3. Add enough white vinegar or banking soda to alter the pH to mimic field conditions (usually a pH between 6.0 or 8.0 S.U.). Usually a couple of ounces are sufficient. Use litmus strips to confirm pH. Adding the vinegar or baking soda while filling the container with water will help with mixing.

4. Put on the multiprobe sonde guard and place sondes in the water so that they are resting vertically on the bottom. Suspending all the units at the same time and depth is ideal if you have the room and capability to do so.
   a. For units with a Clark Cell DO sensor, be sure the stirrer is working properly.

5. Once the readouts are stable, record the measurements from each probe.

6. Satisfactory performing units will have parameter results that are within the maximum error limit outlined in Table 3.2 compared to the average measurement of the same parameter displayed by all tested sondes.

If upon inspection one or more sondes show unacceptable readings while the end of day check indicates the unit is operating within acceptable limits, the unit will be taken out of service for repair/replacement. Regions may need to perform additional checks on their own based on the frequency of affected sondes or the severity of the error.

3.2.4. Annual Temperature Check

At a minimum, Central Office personnel will conduct annual verification of temperature sensing field equipment. This check will consist of comparing probe readouts to a NIST certified thermometer at three reference points covering the expected temperature range encountered in water quality monitoring. The maximum acceptable error of field temperature equipment is +/- 0.5 of the NIST reference thermometer.

The procedure used to check temperature-sensing probeware with the Central Office NIST certified thermometer is below.

1. Three containers are prepared consisting of well mixed water baths. The baths are:
   a. Ice/water mixture (0-4°C)
   b. Room temperature (20-25°C)
   c. Warm tap water approximating maximum expected ambient temperature (30-35°C).

2. The thermistor and a NIST verified thermometer that is within 1 year of the date of verification are lowered side by side into each bath. Water in each bath is agitated with a mixing bar or similar device to ensure the bath is of uniform temperature.

3. Record temperature readings from both instruments once readings stabilize. A stable reading is one that does not change more than 0.1^{o}C for ten seconds.

4. If the difference between the NIST verified thermometer and probe thermistor values exceed 0.5^{o}C in any bath, immediately replace the probe thermister. If immediate repair is not possible, tag the unit out of service until it is repaired or replaced.

3.3. Accument AP and Orion series handheld pH/mV/Ion meter

3.3.1. General procedures

1. Rinse the electrodes thoroughly with DI or distilled water after each sample or standard. Shake off excess water and blot dry.

2. Calibrate the meter each day before use with a minimum of two fresh standard buffer solutions that most closely bracket the expected pH of the samples to be tested. The buffers used for calibration are 7.0, 4.0 and/or 10.0.

3. If the sample pH reading is outside the two buffers you calibrated with, check the meter using the buffer that brackets the observed reading to determine if recalibration is necessary. If the probe reads the pH value of the buffer within +/-0.2 S.U. of the listed buffer value, the probe and data are in compliance. If not, recalibrate the probe with the two buffers that best brackets the observed value and resample. If this is not possible or practicable, exclude the data.

4. After reading highly acidic or alkaline samples, rinse electrodes thoroughly with DI or distilled water and allow the probe to equalize in provided pH probe storage solution. If slow responses are observed in measurements, allow additional time for equilibration.

5. Be sure to check the expiration dates on the pH buffers prior to using them for calibration/post-calibration.

6. The pH probe generally has a one-year shelf life.

3.3.2. Instrument setup

1. For pH, mV, and ion measurements, attach the sensor connections to the appropriate jack on the meter. Adaptors may be necessary for non standard sensors. Some sensors require a separate connection for automatic temperature compensation (ATC). If ATC measurements are not necessary, cover the ATC jack to maintain a waterproof state.

2. Some Accument and Orion meters provide an AC power connection for use indoors. If not used, cover this connection to maintain a waterproof state. If an AC adapter is connected, the meter is not waterproof.

3. Press the on/off button to turn on the meter. Press **Setup** twice and then **ENTER** to clear the memory.

4. With the electrodes immersed in storage or buffer solution, press pH to enter into pH mode.

3.3.3. Calibration

1. For the first use of the day, press the **Setup** key twice and then hit **ENTER** to clear the previous calibration data.

2. Press the **MODE** key until the display indicates the instrument is in pH mode. Remove the protective cap from electrode, rinse with tap water and blot excess water with a soft tissue.

3. Immerse electrode in the first buffer solution and slowly stir the electrode to remove bubbles from the electrode surface and ensure homogenous mixing of the buffer solution.

4. Press the **STD** key and continue stirring. Be sure that the ATC probe is also immersed in the buffer solution. The standard 1 symbol and value should flash. When a stable reading is achieved, the auto symbol will stop flashing and the standard buffer value is displayed.

5. Press **STD** button to access the Standardize screen. The buffer group used by the meter will briefly be displayed, and when prompted, press **STD** to standardize.

6. Press **STD** again to initiate standardization. The meter will automatically recognize the buffer used, and display the value on the screen. Standardize will flash until the buffer is accepted, and the meter returns to the Measure screen. The accepted buffer value remains displayed on the screen.

7. Repeat steps 2-4 with a second and subsequent buffers. When the meter accepts the second buffer, it will briefly display the efficiency (as percent slope) associated with the electrode's performance prior to returning to the Measure mode. If the percent slope is outside the range of 90-102, the meter will display ELECTRODE ERROR and will not return to the Measure screen until you press **ENTER**. The message ELECTRODE ERROR will remain until an acceptable slope is attained after standardization.

8. Record all pH values onto the logsheet.

9. Place the plastic protective cap over the probe for transport. Make sure to saturate the cotton ball with pH 4.00 buffer or electrode storage solution and placed in the bottom of the cap.

3.3.4. Field measurement procedures

1. If the instrument has been checked and calibrated, press the on/off key to switch unit on.

2. Immerse the electrode into the sample solution and stir at a moderate pace. Note: Make sure the meter is in pH mode.

3. When the meter senses that the reading is stable, STABLE will appear under the measurement reading. You can record this value on your field sheet. Enter pH data into CEDS to the tenth place (do not round values based on the hundredth place).

4. If AUTO is not displayed on the screen, the autoread function is not active, and the meter will continuously monitor the pH value of the sample, resulting on changing values.

5. If AUTO is displayed on the screen, the meter will fix the measured pH value on the screen when it is stable. 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 4.00 or pH storage solution.

3.3.5. Maintenance

See Table 3.3

Table 3.3 Preventative Maintenance: Accument AP and Orion series pH/ISE meter

Sensor Type	Item	Procedure
pH sensor	**Slow pH response or reconditioning a pH sensor which was in 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. 1. Soak sensor in warm water with mild detergent for 10-30 minutes. 2. If ineffective, clean the glass bulb sensor with a cotton ball/swab or lint free cloth soaked with mild soap and water. 3. If ineffective or the sensor shows slow response, soak a few minutes with 10% HCl or several hours with pH 4 buffer (pH sensor only). 4. After cleaning, rinse with tap water and blot dry. Place sensor in 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 water tight container containing KCl electrode storage solution or pH 4.0 buffer. 2. If using a liquid filled pH sensor. Leave the vent open while measuring. Close vent while in storage. Refill electrode 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. If sensor is liquid filled, drain all liquid and rinse with DI water using the vent hole. Drain DI water and allow to dry. 3. Place electrode tip in a protective container and 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. 2. Remove the old battery. Connect a new battery of the same type and voltage. 3. Place the installed battery in battery compartment. Make certain the battery wires do not to interfere with the closing of the battery cover. 4. Replace the battery cover. 5. If using an AC adaptor, connect the adapter to the top connector AC power jack and to a power source. Note that the meter is not waterproof when the AC adapter is connected

3.4. YSI Model 58 Dissolved Oxygen and Temperature Meter

3.4.1. Calibration

Calibrate the meter daily when in use. Daily calibration is required as the probe is sensitive to physical shock, disturbing or fouling of the sensing membrane or the drying out of the electrolyte solution.

3.4.1.1. Air calibration (% Saturation)

Air calibration is the quickest and simplest calibration technique

1. Turn the instrument function switch to the % saturation setting

2. Place a moist sponge or a piece of cloth in the plastic calibration bottle. Loosen the bottle lid about ½ turn and slip the bottle over the probe guard up to the bottle. Place the probe in a protected location at room temperature.

3. Set the function switch to **ZERO** and readjust the display to read 0.00. Switch back to **% AIR SAT** mode.

4. After the display reading stabilizes, unlock the O2 calibration control knob locking ring.

5. Adjust the display to the calibration value indicated in the pressure/altitude chart printed on the back of the meter.

6. Re-lock the locking ring of the O2 calibration control knob to prevent accidental changes in the calibration setting.

7. Record the % saturated DO into the logsheet.

3.4.1.2. Air calibration (mg/L mode)

1. Place a moist sponge or a piece of cloth in the plastic calibration bottle. Loosen the bottle lid about ½ turn and slip the bottle over the probe guard up to the bottle. Place the probe in a protected location at room temperature. Switch the function switch to **TEMP**.

2. From the oxygen solubility chart printed on the back of the meter determine and record the mg/l value corresponding to the temperature indicated.

3. Determine the local altitude or the true atmospheric pressure. Using the pressure/altitude chart on the back of the meter, determine the correct calibration value.

4. Multiply the mg/l value from the oxygen solubility table by the calibration value from the pressure/altitude table and divide by 100 to determine the correct mg/l oxygen content of the saturated sample.

5. Readjust to zero if necessary.

6. Check that the salinity knob is set at 0.

7. Turn the function switch to 0.1 or 0.01 mg/l setting.

8. Unlock the O2 calibration control knob locking ring.

9. Adjust the display to the value calculated previously.

10. Allow two minutes to verify stability of the readings.

11. Readjust O2 calibration knob if readings are greater than 0.2 mg/L of the calculated saturation value.

12. Relock the locking ring of the O2 calibration control knob to prevent accidental changes to the calibration settings.

3.4.2. Field Measurement Procedures

With the instrument prepared for use and the probe calibrated, place the probe in the sample. If using the stirrer, connect it to the meter and turn the stirrer switch to ON.

1. Adjust the salinity control to the salinity of the sample.

2. Turn the meter function switch to **ZERO** and zero the meter with the **O2 ZERO** knob if necessary.

3. Turn the meter function switch to the desired readout setting and read the D.O. value in mg/l when the meter reading has stabilized.

4. Record the field data to the tenth place (do not round based on the hundredth place)

5. Enter the field parameter data into CEDS to the tenth place.

3.4.3. Maintenance

See Table 3.4

3.5. YSI 55 Handheld Oxygen and Temperature Meter

3.5.1. Calibration

1. Ensure that the sponge inside the instrument's calibration chamber is wet. Insert the probe into the calibration chamber.

2. Turn the instrument on by pressing the **ON/OFF** button on the front of the instrument. Press the **MODE** button until dissolved oxygen display is % Sat. Wait for the dissolved oxygen and temperature readings to stabilize (15 minutes to 1 hour).

3. Use two fingers to press and release both the **UP ARROW** and **DOWN ARROW** buttons at the same time.

4. The LCD will prompt you to enter the local altitude in hundreds of feet. Use the arrow keys to increase or decrease the altitude. When the proper altitude appears on the LCD, press the **ENTER** button once.

5. The instrument should now display CAL in the lower left of the display. The calibration value should be in the lower right of the display and the current % reading should be on the main display. Make sure that current % reading is stable, then press the **ENTER** button. The display should read SAVE then should return to the normal operation mode.

6. Record the % saturated DO onto the logsheet.

3.5.2. Taking Measurements

When arriving to the field, press the **ON/OFF** button to turn the unit on. The instrument will activate all segments of the display for a few seconds, followed by a self-test procedure that will last for several more seconds. The instrument will display a dissolved oxygen value registered as mg/L or percent saturation depending on the setting. If the instrument detects an internal problem, the display will show an error message.

The Model 55 will always display temperature and can display dissolved oxygen % saturation and dissolved oxygen mg/l. To choose between mg/L and % press and release the **MODE** button.

When taking dissolved oxygen measurements, the probe must be moved through the sample water at the rate of 1 foot per second to provide adequate stirring and representative results.

Record field parameter data and enter into CEDS based on the tenth unit of the display. Do not round to the nearest tenth based on the hundredth place.

3.5.3. Maintenance

See Table 3.4

3.6. YSI 85 Handheld Oxygen, Conductivity, Salinity and Temperature Meter

3.6.1. Calibration

3.6.1.1. Dissolved Oxygen

1. Ensure that the sponge inside the instrument's calibration chamber is wet. Insert the probe into the calibration chamber.

2. Turn the instrument on by pressing the **ON/OFF** button on the front of the instrument. Press the **MODE** button until dissolved oxygen is displayed % Sat. Wait for the dissolved oxygen and temperature readings to stabilize (15 minutes to 1 hour).

3. Use two fingers to press and release both the **UP ARROW** and **DOWN ARROW** buttons at the same time.

4. The LCD will prompt you to enter the local altitude in hundreds of feet. Use the arrow keys to increase or decrease the altitude. When the proper altitude appears on the LCD, press the **ENTER** button once.

5. The instrument should now display CAL in the lower left of the display. The calibration value should be displayed in the lower right of the display and the current % reading should be on the main display. Make sure that current % reading is stable, then press the **ENTER** button. The display should read SAVE then should return to the normal operation mode.

6. Record the % saturated DO onto the logsheet.

3.6.1.2. Conductivity

1. Turn the instrument on and allow it to complete its self-test procedure.

2. Select a calibration solution which is most similar to the sample you will measure.

3. Place at least 3 inches of solution in a clean glass beaker.

4. Press the **MODE** button to advance the instrument to display conductivity.

5. Insert the probe into a beaker deep enough to completely cover the oval shaped hole on the side of the probe. Do not rest the probe on the bottom of the container. Suspend it above the bottom by at least ¼ inch.

6. Allow at least 60 seconds for the temperature reading to become stable.

7. Move the probe vigorously from side to side to dislodge any air bubbles from the electrodes.

8. Press and release the **UP ARROW** and **DOWN ARROW** buttons at the same time.

9. Use the **UP ARROW** or **DOWN ARROW** buttons to adjust the reading on the display until it matches the value of the calibration solution you are using.

10. Once the display reads the exact value of the calibration solution being used, press the **ENTER** button once. The word "SAVE" will flash across the display for a second indicating that calibration has been accepted.

11. Record the calibration value into the logsheet.

12. Turn the unit off using the **ON/OFF** button to conserve battery power when traveling to the field.

3.6.2. Taking Measurements

When arriving to the field, press the **ON/OFF** button to turn the unit on. The instrument will activate all segments of the display for a few seconds, which will be followed by a self-test procedure that will last for several more seconds. The instrument will display the cell constant of the conductivity probe when the self-test is completed. If the instrument detects an internal problem, the display shows a continuous error message.

The Model 85 always displays temperature along with one of five measurements: Dissolved Oxygen %, Dissolved Oxygen mg/l, Conductivity, Specific Conductance, and Salinity. To choose one of the measurement modes above press and release the **MODE** button.

When taking dissolved oxygen measurements the probe must be moved through the sample at the rate of 1 foot per second to provide adequate stirring.

Record field parameter data and enter into CEDS based on the tenth unit of the display. Do not round to the nearest tenth based on the hundredth place.

3.6.3. Maintenance

See Table 3.4

Table 3.4. YSI 55, 58 and 85 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. Remove and discard old membrane or membrane cap. Rinse electrode with DI water. Blot dry. 2. Fill electrode well or cap with KCl solution provided in the membrane kit. **a. If using a membrane cap** i. Using a clean membrane cap, fill ½ to ¾ full with KCl solution. ii. Dislodge any bubbles by tapping the side of the cap with a pencil. iii. Lower the electrode into the membrane cap and tighten to finger tightness. iv. Turn the electrode over to note any bubbles, or wrinkles. If so, discard the membrane and try again. **b. Using membrane film** i. With the electrode facing up, fill with enough KCl to form a positive meniscus. Dislodge any air bubbles by tapping the side of the sensor with a pencil. ii. Using your free hand, obtain a single sheet of membrane film and lay flat on the surface of the KCl meniscus and sensor surface. iii. Place the new O-ring over the gold cathode. Do not use any type of grease on the O-ring. Secure the membrane with the O-ring by pushing down with your thumbs on both sides of the O-ring. iv. Note any air bubbles or wrinkles. If so, discard the membrane and try again. v. 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 dull gray or 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: high strength ammonia will dissolve silver faster so constantly check the anode if using high strength ammonia. 2. Remove electrode and rinse with DI water and blot dry. 3. If still tarnished, use a moistened 400 or finer grit sandpaper such as provided in the membrane kit and gently buff the surface. Rinse off sanding residue and blot dry. 4. Add a new membrane using steps listed above.
	Cleaning gold cathode. **Check for tarnished or dull gold color when changing DO membrane.**	1. Wipe with a clean lent-free cloth, or pencil eraser. The color should be matte gold. 2. If cathode is still tarnished, use a moistened 400 or finer grit sandpaper such as provided in the membrane kit and gently evenly buff the surface. 3. Rinse electrode with DI water and blot dry. 4. Add a new membrane using steps listed above.
Conductivity Sensor (YSI 85 only)	**Cleaning conductivity sensor.** **As needed**	1. Using a paper towel or cotton swab, clean the sensor of hardened foreign material using warm water and mild detergent. 2. Rinse sensor with DI 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 chamber 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. Remove membrane film or cap and rinse electrode with DI water and blot dry. Attach a dry membrane film or cap to the electrode and store in a cool, dry location. 2. Remove batteries.
	Battery replacement.	1.Remove the battery cover 2.Remove old batteries 3.Add new batteries of the same type and voltage (AA, C, etc.) 4.Replace the battery cover

3.7. Hydrolab Datasonde and Minisonde with Surveyor

3.7.1. Calibration

3.7.1.1. General procedures

Perform the calibration and postcheck procedures specified below on each day of use.

DO NOT turn off the display unit at any time during the calibration procedure. The newly calibrated data will not be saved if the unit is turned off.

Maintain a calibration log sheet in which all data pertaining to each precalibration, postcheck, or maintenance procedures are entered.

Replace the storage cup on multiprobe with a bottomless calibration cup. Take special care not to bump the probes as this may result in damage to the probes.

After calibrating each parameter, record the calibrated parameter values in the calibration sheet (see Appendix A) and be sure to store the data utilizing the display unit.

If the surveyor internal battery reading drops below 7.2 volts, recharge the batteries.

Standards should be selected that best mimic the anticipated ambient sampling conditions.

3.7.1.2. Barometric Pressure

Some Surveyor4 units are equipped with a barometer to calculate depth and dissolved oxygen levels. Once a week, check if the reported barometric pressure readings needs calibration, compare the instrument value (recorded in mmHg) to a NIST Traceable barometer. When the difference between Surveyor4 reading and traceable barometer is greater than 10 mmHg, the Surveyor4 needs to be calibrated to the traceable barometer. The traceable barometer needs to be calibrated annually or every two years depending on the certificate of accuracy.

You may also use nearest National Weather Service or NOAA weather station barometric pressure readings if a NIST Traceable barometer is not available. Appendix B contains additional information on using weather station barometric pressure readings.

To calibrate the Hydrolab BP sensor:

1. Disconnect the surveyor from the datasonde/minisonde.

2. Turn on the surveyor.

3. Select **Setup/Cal**.

4. Select **Calibrate**.

5. Select **BP Svr4: User Cal** and press **Select**.

6. Use the **arrow keys** to enter the BP reading from the NIST barometer or adjusted weather station BP reading and press **Done**.

3.7.1.3. Conductivity

Conductivity requires a two-point calibration. You will need to calibrate your sensor to "0" first, then to the value of the slope standard you are using.

1. Be sure to turn off the circulator by selecting **Setup/Cal**, **Setup**, and then **Sonde**. Using the **arrow keys** choose **Circulator: Off/On** then press **Select**. Select the number to **0** using the **arrow keys** and then select **Done**.

2. Dry the conductivity probe opening with Q tip or soft cloth.

3. Select **Setup/Cal, Calibrate, Sonde, SpCond: uS/cm**, and then **Select**. Select the number **0** using the **arrow keys** and select **Done**. Press any key and select **Go Back**. SpCond will read 0.0.

4. Rinse with the conductivity standard you will use for the slope calibration. Discard the rinse solution.

5. Fill the calibration cup with fresh conductivity standard until it reaches the O-ring of the DO sensor. Allow the temperature and conductivity reading to stabilize. A stable reading is when it changes $\leq$ 0.1 units in ten seconds. Usually this will take a minute. Record the initial conductivity reading in the calibration log book.

6. Select **Setup/Cal**, **Calibrate**, **Sonde**, then **SpCond:uS/cm**. Using the arrow keys, enter in the value of the standard in use and press **Done**. Press any key and select **Go Back**. SpCond will read the value of the standard. Record the calibration conductivity value on the logsheet.

3.7.1.4. Dissolved Oxygen Calibration- Clark Cell

The DO membrane and electrolyte solution should be inspected for any damage or air bubbles prior to calibration. If air bubbles or damage are present, replace the membrane according manufacturer manuals. To ensure accuracy, wait at least 4 hours after changing the membrane before using the instrument to allow the membrane to equilibrate.

Note: It is best to store water in a clean carboy at room temperature for dissolved oxygen calibration. This will reduce temperature equilibrium time. As an alternative, you may use the conductivity standard for calibration of dissolved oxygen.

1. Verify the circulator is off. If the circulator is on, turn it off by selecting **Setup/Cal**, **Setup**, **Sonde**. Using the arrow keys choose **Circulator: Off/On**, press **Select**. Select the number **0** using the arrow key and select **Done**.

2. Fill the cap cup with water to just below the O-ring of the DO probe. You may use the conductivity solution for this step if you did not discard it earlier.

3. Using a kimwipe or other soft towel, carefully remove any water droplets from the DO membrane. Do not apply pressure to the membrane.

4. Cover the calibration cup loosely with the plastic storage lid, and allow the unit to equilibrate until the temperature reading is stable (≤ 0.1 change in 10 seconds). Record this initial DO reading and temperature in the calibration log book.

5. Select **Setup/Cal**, **Calibrate**, **Sonde, DO %Sat** then **Select**. Enter the value of the current barometric pressure in mmHg using the **arrow keys**. Press any key and select **Go Back**.

6. Using a dissolved oxygen calibration table (Appendix B), read the oxygen concentration from the table based on the probe temperature set to Celsius degrees as determined by the far left column and first row of the chart. Multiply the oxygen concentration by the correction factor as determined by the barometric pressure. Record this value on the appropriate space on the calibration log sheet.

7. Record the theoretical and probe calibrated DO values (mg/l) in the logsheet.

8. The DO (mg/l) reading should be within ± 0.2 mg/l of the calculated saturated DO from the table.

3.7.1.5. Dissolved Oxygen Calibration- Optical Sensor

Inspect the optical membrane to ensure you cannot see light through the membrane tip greater than 1 mm in size. To do this turn on the handheld and sonde and cup your hand to provide shade or use a hollow tube like a cardboard paper towel roll to see the flashes of blue light. If you see blue light on the membrane tip (not the edges), replace the membrane following the maintenance section of this manual or manufacturer instructions.

Note: It is good practice to use water from a clean carboy stored at room temperature to perform the calibration. The water will be at room temperature to minimize equalization time. As an alternative, you may use the conductivity standard for calibration of dissolved oxygen.

1. Fill the calibration cup with water so that it covers the optical DO sensor but still has an air gap. You may use the conductivity solution for this step if you did not discard it earlier.

2. Tightly close the lid on the calibration cup and pick up the sonde. Shake vigorously for one to two minutes in a side to side and up and down motion to saturate the water with air.

3. Return the sonde to the ring stand and loosen the lid of the cup to allow air to enter and exit the calibration cup.

4. Allow the unit to equilibrate until the temperature reading is stable (< 0.1 change in 10 seconds). Record this initial DO reading and temperature in the calibration log book.

5. Record the temperature and barometric pressure from the display unit in the appropriate spaces on the logsheet.

6. Select **Setup/Cal**, **Calibrate**, **Sonde**, **LDO%:Sat** then **Select**. Enter the value of the current barometric pressure in mmHg using the **arrow keys**. Press any key and select **Go Back**.

7. Using a dissolved oxygen calibration table (Appendix B), read the oxygen concentration from the table based on the probe temperature set to Celsius degrees as determined by the far left column and first row of the chart. Multiply the oxygen concentration by the correction factor as determined by the barometric pressure. Record this value on the appropriate space on the calibration log sheet.

8. Record the theoretical and probe calibrated DO values (mg/l) in the logsheet.

9. The DO (mg/l) reading should be within $\pm$0.1 mg/l of the calculated saturated DO from the table.

3.7.1.6. pH

Note: Calibrate the instrument with pH buffer bracketing the expected values in the field.

1. Discard any liquid used to calibrate other sensors and rinse 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 these rinses down the drain.

3. Fill the calibration cup with Fresh pH 7.0 buffer sufficient to cover the pH sensor.

4. Allow at least two minutes for temperature readings to stabilize.

5. Select **Setup/Cal**, **Calibrate**, **Sonde** then use the **arrow keys** to find **pH:**Units and then press **Select**. Using the **arrow keys**, enter the value of the standard being used and select **Done**. Press any key and select **Go Back**. Allow the pH reading to stabilize. Record the calibration pH value in the logsheet.

6. Pour the used buffer into a storage bottle for use as a future rinse or end of day check.

7. Thoroughly flush the calibration cup and sensors twice with lab grade water.

8. Rinse the cup and sensors twice with a small amount of pH 10.0 or pH 4.0 buffer. Calibrate using the buffer you washed with and that will cover expected values on the sample run.

9. Fill the calibration cup with fresh pH 10.0 or pH 4.0 buffer to cover the sensor and wait for the instrument to equilibrate.

10. Record the pH value displayed in the logsheet.

11. Select **Setup/Cal**, **Calibrate**, **Sonde** then use the **arrow keys** to find **pH:**Units and then press **Select**. Using the **arrow keys**, enter the value of the standard being used and select **Done**. Press any key and select **Go Back**. Allow the pH reading to stabilize. Record the calibration pH value in the logsheet.

12. Pour the used buffer into a storage bottle for future rinses and end of day check. Flush the calibration cup and sensors thoroughly three times with lab grade water.

13. Remove the calibration cup and replace with the storage cup.

14. Place a sufficient amount of pH 4 buffer in the storage cup to keep the sensors moist. Tap water may be used but pH 4 buffer is the best. If using water, do not allow it to contact with the pH sensor.

3.7.1.7. Depth

When at the sample site, lower the probe to one meter depth using a measured length accounting for the sonde casing and cable. Calibrate the depth to one meter. If this is not possible, calibrate to a depth of zero at or near the surface of the water to be sampled. When calibrating the unit, be sure to remove the calibration cup and attach the probe guard as a sealed calibration cup will affect the pressure readings.

3.7.2. Preparation for use

If using the short calibration cable to calibrate the sonde, be sure to switch to the cable of sufficient length to sample with.

Remove the storage cup from the sonde, screw on the sensor guard when deploying the sonde. Between sample stations, either immerse the probe in a container of sample water or replace the guard with the calibration cup and some pH 4 buffer or water to keep the sensors moist.

3.7.3. Data Recording

The field parameter data should be recorded and entered into CEDS to the tenth place (do not round based on the hundredth place).

3.7.4. Data Logging

To set up a time triggered file in the Sonde with the Surveyor for continuous monitoring:

1. Connect the Sonde to the Surveyor and turn on.

2. Select **Files.**

3. Select **Sonde.**

4. Select **Create.**

5. Enter the name of the file. Using the **arrow keys**, spell out the name. When completed, press **Done.**

6. The next two screens will prompt you when the units should start and stop logging. Use the **arrow keys** to select the time in DDMMYYHHMMSS format. Select **Done** when complete.

7. The next screen will ask for the logging interval in HHMMSS format. Select **Done** when complete. Value entered must be at least 000030.

8. The next screen will ask for sensor warm-up time. This can be any length of time not less than 30 seconds and no more than 2 minutes. Select **Done** when complete.

9. The next selection is the circulator warm up time. If you do not want to enable the circulator, you may enter 000000. Select **Done** when complete.

10. It following screen will ask if you want the audio on or off. Select **0** to turn off the audio or **1** to turn it on using the **arrow keys**. Select **Done** when complete.

11. The next screen will allow you to select the parameters you wish to record. Use the **arrow keys** to select the parameters and press **Add**. Press **Done** when complete.

12. You have completed setting up a logging file in the Sonde. You may now deploy the Sonde in the location you wish to monitor.

3.7.5. File transfer from Sonde to PC

1. Connect the sonde to a PC computer.

2. Select **File**.

3. Select **Transfer**.

4. When you see “Power Down Probe?” Type **N** and **Enter**.

5. Select **Log File**. Type the name of the file you wish to download and **Enter**.

6. Select a spreadsheet that you will download the data to.

7. Select **Xmodem**.

8. Screen will say “Starting Xmodem Transfer”.

9. Move the cursor to the top of the screen and click **Open Transfer**.

10. Make sure you are using Xmodem protocol.

11. Select **Receive File**.

12. Now name the file using .csv extension.

13. Select **OK**.

14. Transfer will begin.

3.7.6. Storing data in clipboard

If you surveyor has clipboard memory, you can store up to 12 scans by using the store function. This feature allows you to capture the lines of data displayed at the time you press the **Store** key.

1. You need to select the parameters you would like to save to the Surveyor clipboard. Got to **Setup/Cal**, **Setup**, **Display**, then select **Tabular:Display** submenu using the **arrow keys** then hit the **Select** button.

2. Use the **arrow keys** and **Add** or **Remove** buttons to select parameters you wish to record.

3. Once you satisfied with your selection, press the **Done** key and hit the **Go Back** key three times.

4. When you are ready to record the displayed data, press the **Store** key. You have now saved 1 scan to your surveyor.

3.7.6.1. Reviewing clipboard scans

1. Using the Surveyor select **Files**, **Surveyor4**a, **Clipboard** using the **arrow keys** and **Select** button.

2. Depending on the number of scans in the surveyor clipboard contains, you can press the **down arrow key** and move to the other files to review the information you stored earlier.

3.7.6.2. Deleting clipboard scans

1. Using the Surveyor select **Files**, **Surveyor4**a, then **Delete** using the **arrow keys** and **Select** button.

2. You will see a screen showing all of the scans stored on the surveyor. You can either delete individual rows using the **arrow keys** and the **Select** button or all the stored data by selecting **All: Date-Time**

3.7.7. Maintenance

See Table 3.5

3.8. Hydrolab H2O Water Quality Multiprobe with Display

3.8.1. System components

The four basic components of the Hydrolab water quality multiprobe are the probe, the display unit, the sample circulator and the battery pack.

1. The multiprobe, in its sealed high pressure housing, contains all of the sensors for temperature, dissolved oxygen, conductivity, pH, and depth as well as the electronic circuits that sensors require for their operation.

2. The display unit which operates at the opposite end of the data cable from the multiprobe. The display unit interprets the signals from the sensors and displays the values on the readout display.

3. The sample circulator is an electrically powered magnetic stirrer similar to laboratory magnetic stirrers. The circulator is necessary to obtain the most representative results in the sample.

4. The auxiliary battery pack supplies power from a 12 volt recharger for the entire system.

3.8.2. Calibration

3.8.2.1. General Procedures

1. DO NOT turn off the display unit at any time during the calibration procedure. The unit will not save newly calibrated data if turned off prematurely.

2. Note the condition of the sensors to ensure they are in good working order. If there is the presence of foreign material or other defects to sensors. Refer to manufacturer's operation manual or table 3.5 for maintenance of sensors.

3. Record calibration data, end of day checks, and relevant maintenance notes on a calibration log sheet. A copy of the calibration log sheet is found in Appendix A

4. Perform calibration and end of day check procedures on each day of use.

5. Replace the storage cup on multiprobe with the bottomless calibration cup. Take special care not to bump probes as this may result to damage to the probes.

6. After the calibration of each parameter, record the calibrated parameter value in the calibration sheet (Appendix A) and be sure to store the data utilizing the display unit.

7. When the multiprobe battery reading drops below 10.0 volts or the low battery warning symbol appears on the display, replace the internal batteries or recharge the external battery as soon as possible.

8. Check the expiration dates on all standards. Use of expired buffer or standards will lead to inaccurate results. Standards must be used within six months of the date of preparation/first opened or by the printed expiration date.

3.8.2.2. Specific Conductance

Note: Readings are most accurate when they are close to the standard used to calibrate conductivity. Use your best judgment to determine the expected range of values you expect to encounter in the field prior to calibration.

1. Rinse the sensors twice with a small portion of the specific conductance standard you will use to calibrate the instrument. Discard the rinses down the drain.

2. With the sensors pointing towards the ceiling and calibration cup attached, slowly fill the calibration cup with fresh standard solution to just level of the O-ring used to hold the dissolved oxygen membrane. Be sure to avoid trapping air bubbles on the conductivity sensor.

3. When you have a stable specific conductance reading, the sensor is ready to calibrate.

4. Press the calibration button and use the left arrow key to choose conductivity and press **ENTER**. Use **UP, DOWN**, and **RIGHT** arrows to enter the value of your specific conductance standard and press **ENTER**. Use the **LEFT** arrow key to choose **Y** (yes) to save the calibration. Record the calibrated value in the logsheet.

5. When finishing calibrating, pour standard solution into a storage bottle to use as the standard rinse in the next calibration. Discard after using for the rinse.

3.8.2.3. Dissolved Oxygen

The DO membrane and electrolyte solution should be inspected for any damage or air bubbles prior to calibration. If air bubbles or damage are present, replace the membrane according manufacturer manuals. To ensure accuracy, wait at least 4 hours after changing the membrane before using the instrument to allow the membrane to equilibrate.

Note: It is best to store water in a clean carboy at room temperature for dissolved oxygen calibration. This will reduce temperature equilibrium time. As an alternative, you may use the conductivity standard for calibration of dissolved oxygen.

1. Check the membrane on the probe tip to see if it is wrinkled, bubbled, torn, dirty, or otherwise damaged. If so, see table 3.5 or the manufacturer's operation manual to change the membrane. It is good practice to change the membrane at least once or twice a month to ensure accuracy and extend sensor life.

2. With the multiprobe oriented so that the sensors are pointed toward the ceiling, fill the calibration cup with room temperature water until the water is just level with the O-ring used to secure the membrane. If you have just calibrated conductivity, you may use that solution to calibrate dissolved oxygen prior to pouring into the rinse bottle or sink drain.

3. Carefully remove any water droplets from the membrane with the corner of a lint free tissue. DO NOT apply pressure to the membrane surface. Cover the open top of the calibration cup to prevent air currents from affecting calibration.

4. Wait for several minutes to allow air in the cup to become water saturated. The sensor is ready to calibrate once the dissolved oxygen and temperature readings stabilize ($\leq$0.1 change for ten seconds).

5. Determine the barometric pressure from a calibrated barometer or adjusted weather station data. Record in the appropriate space on the calibration log sheet.

6. Record the temperature reading on the calibration log sheet once it is stable. Record the initial meter dissolved oxygen reading on the log sheet.

7. Using a dissolved oxygen calibration table (Appendix B), read the oxygen concentration from the table based on the probe temperature set to Celsius degrees as determined by the far left column and first row of the chart. Multiply the oxygen concentration by the correction factor as determined by the barometric pressure. Record this value on the appropriate space on the calibration log sheet.

8. Press **Calibrate**, and use **RIGHT** arrow key to choose (mg/L). When promoted, enter in the barometric pressure in mmHg. Press **ENTER**. Use the **LEFT** arrow key to choose **Y** (yes) to save the calibration. The unit will display a DO value (mg/l). Confirm that the % saturation equals 100.0 %. Record the reported DO mg/L value from the screen and record in the appropriate space on the log sheet. Compare the theoretical value obtained from the table to the display. They must be within ± 0.2 mg/l.

3.8.2.4. pH

Note: Calibrate the instrument with pH buffer bracketing the expected values in the field.

1. Discard any liquid used to calibrate other sensors and rinse 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 these rinses down the drain.

3. Fill the calibration cup with Fresh pH 7.0 buffer sufficient to cover the pH sensor.

4. Allow at least two minutes for temperature readings to stabilize.

5. Press **Calibrate** and **ENTER** to choose **P** for pH. Use the **UP, DOWN**, and **RIGHT** arrows to enter the pH of the zero standard (7.00). Press **ENTER**. Use the **LEFT** arrow to choose **Y** (yes) to save calibration. Record the pH value in the logsheet.

6. Pour the used buffer into a storage bottle for use as a future rinse or end of day check.

7. Thoroughly flush the calibration cup and sensors twice with lab grade water.

8. Rinse the cup and sensors twice with a small amount of pH 10.0 or pH 4.0 buffer. Calibrate using the buffer which will cover expected values on the sample run.

9. Fill the calibration cup with FRESH pH 10.0 or pH 4.0 buffer to cover the sensor and wait for the instrument to equilibrate.

10. Record the pH value displayed in the logsheet.

11. Press **Calibrate** and **ENTER** to choose **P** for pH. Use the **UP, DOWN**, and **RIGHT** arrows to enter the pH of the slope standard (4.00 or 10.00). Press **ENTER**. Use the **LEFT** arrow to choose **Y** (yes) to save calibration. Record the pH value in the logsheet.

12. Pour the used buffer into a storage bottle for future rinses and end of day check. Flush the calibration cup and sensors thoroughly three times with lab grade water.

13. Remove the calibration cup and replace with the storage cup.

14. Place a sufficient amount of pH 4 buffer in the storage cup to keep the sensors moist. Tap water may be used but pH 4 buffer is better. If using water, do not allow it to contact the sensors as it may damage the pH sensor.

3.8.2.5. Depth

When at the sample site, lower the probe to one meter depth using a measured length accounting for the sonde casing and cable. Calibrate the depth to one meter. If this is not possible, calibrate to a depth of zero at or near the surface of the water to be sampled. When calibrating the unit, be sure to remove the calibration cup and attach the probe guard as a sealed calibration cup will affect the pressure readings.

3.8.3. Transport to the Field

2. After calibrating, disconnect the display unit, battery pack, and data cable. Carefully place the instrument back in its case. Coil the cable and keep it kink-free.

3. At the sample site, remove the storage cup from the Sonde and screw on the circulator in its place. Connect the two ends of the data cable to the probe and circulator. Ensure that the probe is attached to the data cable with the pin and clip ring.

4. If sampling by boat, fill the sonde storage bucket with sample water so that the sensors will be submerged. Store the sonde in a bucket of water when not in use. Alternately, remove the circulator (do not disconnect) and replace the storage cup filled half full with sample water.

5. Ensure that the magnetically coupled impeller on the stirrer base is properly lubricated and rotating free on its shaft. Any obstruction to smooth rotation of the impeller may cause erratic DO measurements and excessive power drains to the battery.

6. Turn the display unit switch to **OFF**.

3.8.4. Field set-up and operation

If sampling by boat, the boat operator must maintain the boat's position and orientation with wind and tide movement to ensure that the Sonde hangs as vertically as possible.

While the unit is in the operation aboard the vessel, keep the probe away from the engine's propeller to ensure the safety of the unit and data cable, and to prevent interference from the propeller wash with all water quality measurements.

1. Remove the unit from storage.
    a. Screw on the circulator. Remove the storage cup from the Sonde and screw on the circulator in its place. Use caution to avoid any contact with the sensors.

    b. Connect the two ends of the data cable to the probe and circulator. – Ensure that the probe is attached to the data cable with the pin and clip ring.

    c. Calibrate depth. – At the station above the surface water, press the depth key.

    d. When not in use, store the Sonde in the plastic bottle used for DO Saturation checks with a small amount of water or bucket of water.

    e. Inspect the magnetic impeller on the stirrer base. Make sure magnetic impeller is rotating freely on its shaft. Any obstruction to smooth rotation of the impeller may cause erratic DO measurements and excessive power drains to the battery.

2. Lower the Sonde to 0.3 meter below the surface.

3. Wait for all readings to stabilize. Probes are considered stable when the smallest reporting unit (usually to the tenth value) does not change for more than 10 seconds. Record stabilized values on the field sheet. Record field data to the tenth place (do not round based on the hundredth place)

4. Turn the display unit off and retrieve the Sonde - At the end of the day put the storage cup back on the Sonde.

5. Upon return from the field and completing end of day checks, enter validated field parameter data entered into CEDS to the tenth place (do not round based on the hundredth place).

3.8.5. Maintenance

See Table 3.5

3.9. Hydrolab Quanta

3.9.1. Calibration

3.9.1.1. Preparation for Calibration

1. Select standards whose value is nearest what is expected to encounter in the field.

2. Clean and prepare the sensors and attach the calibration cup.

3. Using the calibration cap, thoroughly rinse the sensors several times by half-filling the calibration cup with analyte-free water and shaking the transmitter to make sure each sensor is free from contaminants that might alter your calibration standard.

4. In a similar manner, rinse the sensors twice with a small portion of calibration standard prior to calibrating with the standard discarding the rinse each time.

5. Discard used calibration standards appropriately. Do not reuse calibration standards except as to rinse sensors prior to calibration with fresh standard of the same strength.

3.9.1.2. Specific conductance

1. Pour the specific conductance standard to within a centimeter of the top of the cup and make sure there are no bubbles in the hole of the specific conductance sensor.

2. Selecting the **Calib** icon allows calibration of specific conductance. After selecting **Calib**, the Calib icon will remain lit in the operation icons and the parameter digits and units icons will be blank. The heading icons will display the items that can be calibrated.

3. From the displayed icons, select the item to be calibrated. The selected one will blink. The display will show the current value for the item selected. Press the arrow to change the numeric value to match the calibration standard. Once the value is correct, press the **Enter** key to send the updated calibration value to the transmitter.

3.9.1.3. Dissolved Oxygen % Saturation

The DO membrane and electrolyte solution should be inspected for any damage or air bubbles prior to calibration. If air bubbles or damage are present, replace the membrane according manufacturer manuals. To ensure accuracy, wait at least 4 hours after changing the membrane before using the instrument to allow the membrane to equilibrate.

Note: It is best to store water in a clean carboy at room temperature for dissolved oxygen calibration. This will reduce temperature equilibrium time. As an alternative, you may use the conductivity standard for calibration of dissolved oxygen.

1. With the probe sensors facing upward, fill the calibration cup with deionized or tap water until the water is just level with the O-ring used to secure the membrane. You may use the conductivity solution for this step if you did not discard it earlier.

2. Carefully remove any water droplets from the membrane with the corner of a tissue.

3. Turn the black lid upside down (concave up) and lay it over the top of the calibration cup.

4. Determine the barometric pressure for entry as the calibration standard. If a calibrated barometer is not available, you may use weather station pressure data by following the instructions found in Appendix B of this document.

5. Using the **arrow keys**, select the **DO%/BP** from the Calib icon, enter the barometric pressure and press the **Enter** key.

6. The unit should now display 100% for the dissolved oxygen percent saturation. Compare the mg/L readout with what the theoretical DO level using Appendix B of this document. If the probe is off by more than 0.2 mg/L of theoretical levels, service the probe and recalibrate before taking out to the field.

3.9.1.4. pH

pH is a two point calibration process. A pH standard 7 is treated as the "zero" and all other values are treated as the "slope".

1. Rinse the sensors with tap or lab water followed by two rinses of the pH 7 buffer that is either freshly prepared or buffer that was used in a prior calibration.

2. With the sensors facing upward, pour fresh pH 7 buffer to within a centimeter of the top of the cup. Be sure to immerse the pH and temperature sensors with buffer.

3. Select "pH" from Calib icons. Press the **arrow keys** to change the numeric value to select **7** and press **Enter** key.

4. After locking in the value, rinse the sensors with DI water thoroughly followed by two rinses with fresh or recently used pH 4 or pH 10 buffer.

5. Pour fresh pH 4 or 10 buffer to within a centimeter of the top of the cup.

6. Press the **arrow keys** to change the numeric value to enter **4** or **10** and press **Enter** key.

7. Verify pH calibration while immersed in the slope and pH 7 buffers. Record the readouts on the calibration log sheet. If pH readings are greater than 0.2 S.U. from the buffer standard, service the pH sensor and repeat calibration before going into the field.

3.9.2. Data Recording

Record all field parameter data to the tenths place when entering into CEDS. Do not round the value based on the hundredth place.

3.9.3. Maintenance

See Table 3.5

Table 3.5. Hydrolab Datasonde, Minisonde, Quanta, and H2O Series General Maintenance

Sensor Type	Item	Procedure
Clark Cell DO Sensor	**Changing DO membrane.** **Every 30 days, or if daily post check fails, or membrane is torn, wrinkled, has air bubbles, or sensor readings are erratic or slow.**	1. Remove the O-ring securing the membrane. Shake out the odd electrolyte. 2. Rinse the sensor cavity with DI water. Fill with fresh DO electrolyte solution until a positive meniscus of electrolyte forms above the surface of the sensor. Remove any bubbles by tapping on the side of the DO sensor. 3. Using a new DO membrane, lay it flat over the senor head. Be sure to not put fingerprints on the membrane that will be directly over the sensor. 4. Place the new O-ring over the gold cathode. Do not put grease on the O-ring. Secure the membrane with the O-ring by pushing down with your thumb and index finger of your free hand on both sides of the O-ring. If there are no air bubbles or wrinkles, carefully trim the excess membrane extending below the O-ring with the pair of scissors or a razor blade. 5. Allow the membrane to relax a minimum of four hours before calibrating. Whenever possible, allow 24 hours before calibration and use in the field.
	Cleaning silver anode. **Clean if majority of anode is dull gray or black while changing the 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: high strength ammonia will dissolve silver faster so constantly check the anode if using high strength ammonia. 2. Remove electrode and rinse with DI water and blot dry. 3. If still tarnished, use a moistened 400 or finer grit sandpaper and a Q-tip to buff the surface. 4. Rinse off sanding residue with triple rinses of DI water and blot dry. 5. Add a new membrane using steps listed above.
	Cleaning gold cathode. **Clean if cathode is tarnished or silver looking while changing DO membrane.**	1. Clean by wiping with a clean lent-free cloth, pencil eraser or hard paper. The color should be matte gold. 2. If cathode is still tarnished, use a moistened 400 or finer grit and gently buff the surface. 3. Rinse electrode three times with DI water and blot dry. 4. Add a new membrane using steps listed above.
Optical DO Sensor	**Cleaning sensor** **Whenever there is dirt, algae, or mold on the sensor housing or membrane.**	1. Flush the entire sensor with clean, fresh water. 2. Inspect the membrane cap. Use a Kimwipe™ (or similar lint free lab tissue), to gently wipe away any foreign material on the sensor cap. 3. If rinsing does not work, you may soak the sensor in household strength white vinegar for 15 minutes followed by 15 minutes soak in DI water. 4. Rinse with fresh water. 5. If you can see light through the side of the cap, use a black permanent marker to recolor the membrane . **DO NOT use the marker on the membrane tip.** **DO NOT clean with alcohol or organic solvents, as this will destroy the membrane.** **DO NOT remove the membrane cap unless you are replacing with a new cap.**
	Replacing membrane **Once per year 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 coat the O-ring or cap seal with grease**. 3. Screw in new membrane cap to the sensor. Tighten to finger tightness. a. Do not touch the outer or inner surface of membrane tip with your fingers. b. If you can see light through the side of the cap, use a black permanent marker to recolor the membrane . **DO NOT use the marker on the membrane tip.**
	Rehydrate optical membrane **If sensor is not kept in a 100% humid environment longer than 2 hours**	1. Remove the optical sensor from the housing. 2. Place 400 mL of water in a 600 mL glass beaker. Do not use plastic containers. 3. Heat the water on a hotplate or in an oven to a temperature of 50 +/-5 C. 4. Place the sensor tip containing the membrane in the warm water and maintain the elevated temperature for 24 hours. Cover the beaker to minimize evaporation. 5. After rehydration is complete, store the sensor tip in either water or water-saturated air at room temperature prior to calibration and deployment.

Table 3.5. Hydrolab Datasonde, Minisonde, Quanta, and H2O series General Maintenance- continued

Sensor Type	Item	Procedure
Conductivity Sensor	**Cleaning conductivity sensor.** **As needed**	1. Using a paper towel or cotton swab, clean the sensor of hardened foreign material using warm water and mild detergent or methanol. 2. Rinse sensor with DI water and blot dry. 3. If needed, repeat using a soft brush. Hard scouring will damage the sensor.
pH sensor	**Cleaning pH electrode.** **Slow readings, when obviously coated with foreign material or the glass sensor is scratched**	1. Remove the sensor from the multiprobe housing 2. Soak pH sensor in warm water with mild detergent for 10-30 minutes. 3. If ineffective, gently clean glass bulb sensor with a cotton ball/swab or lint free cloth soaked in methanol. 4. If ineffective or the sensor shows slow response, soak a few minutes with 10% HCl or several hours with pH 4 buffer. 5. After cleaning, rinse well with DI water and blot dry. 6. Reattach sensor to the probe housing. Apply grease if needed to the O-ring.
pH Reference Electrode **(H2O only)**	**Changing reference solution** **Erratic/slow pH calibration or response, electrode solution appears low or cloudy.**	1. Unscrew the Teflon junction. Pour out the old electrolyte solution. 2. Use a hypodermic syringe to rinse the reference electrode housing with pH electrolyte solution. 3. Pour out the solution and then use the hypodermic syringe to fill the housing with fresh electrolyte. Make sure that no bubbles are trapped in the reference electrode housing after it has been filled. 4. Use a standard screwdriver to screw the Teflon junction back on.
pH Reference Electrode **(Datasonde and Minisonde)**	**Changing reference solution** **Erratic/slow pH response, electrode solution appears low or cloudy.**	1. Gently pull the entire reference sleeve away from the multiprobe. The reference sleeve is the clear blue tube with a porous Teflon Reference Junction attached. 2. Discard the old electrolyte from the reference sleeve. 3. Drop two KCL salt rings into the reference sleeve. 4. Refill the sleeve to the top with reference electrolyte. 5. With the sensors pointed toward the floor, push the full reference sleeve back on to its mount until sleeve has just covered the first O-ring located on the mount. 6. Turn the multiprobe so that the sensors point toward the ceiling and push the sleeve the rest of the way onto its mount. 7. Rinse with tap water.
Temperature Sensor	**General cleaning**	Clean as necessary to remove hardened deposits using mild soap and water and a cloth. Do not bend, dent, or nick the sensor as this may alter temperature readings
General	**Short term storage (<1 month)**	Place a small amount of clean water (1/4 inch or so) to the storage cup and securely attach to the sensor end of the unit. Be sure the water does not directly contact sensors.
	Long term storage (>1 month)	1. Remove batteries (size AA or C) from units which will be in storage for more than 30 days. Do not remove the lithium battery (silver disk) from the multiprobe housing as this maintains the internal clock. 2. If possible, remove the pH sensor from the unit and place the glass bulb end into the provided pH storage bottle with pH electrode storage solution or 4.0 buffer 3. Do not remove the dissolved oxygen sensor. It must be stored in a humid environment. 4. Conductivity and temperature sensors may be stored in a dry or wet condition. 5. Secure all exposed ports with provided port caps. 6. Add ½ inch of clean water to the calibration cup and securely attach it to the sonde housing. Check periodically (30 days) to ensure water has not evaporated or become contaminated.

3.10. YSI 6-series Multiprobe Sonde

3.10.1. Calibration

The instrument must be calibrated prior to sampling.

1. Check the display/logger to determine the battery level to see if new batteries are needed.

2. During the calibration of sensors, never accept any calibrations when a warning message is displayed. You must determine the cause of the problem, correct the problem and recalibrate the probe before using the instrument.

3. Standards must be active (check expiration data) and fresh for all calibrations. Previously used standards may be used to rinse the probe but not for calibration. Discard and replace all expired standards.

3.10.1.1. Barometric Pressure

Barometric pressure is measured by the 650 MDS data logger and used for depth and dissolved oxygen calculations. It needs to be checked with a NIST traceable barometer at least once a week. When the difference between 650 MDS data logger BP reading and traceable barometer is greater than 10 mmHg, adjust the 650MDS data logger barometer to the lab barometer. The traceable barometer is calibrated annually.

1. Once the uncorrected barometric pressure has been determined, the 650 MDS can be calibrated. In the main menu, use the **arrow keys** to select “System Set Up” and then press **Enter**.

2. Using the **down arrow**, scroll down to “Calibrate Barometer”. Press **Enter**.

3. Record the barometric offset value on the log sheet in the comments section.

4. While the “mmHg value” is still highlighted, press **Enter**. Key in the lab barometric pressure and press **Enter** using the **keypad**. Record the new barometric offset on the log sheet. The barometric pressure calibration is complete.

3.10.1.2. Conductivity

1. Rinse the probe with DI water followed by a rinse with a small amount of the conductivity standard you will calibrate with. Discard the rinses.

2. Using fresh conductivity standard, fill the calibration cup is filled that the conductivity probe is completely submerged. The hole in the side of the probe must be under the surface of the solution and not have any trapped bubbles in the opening. If bubbles are trapped on the conductivity cell, gently rotate and/or move the Sonde up and down to remove them

3. Allow at least one minute for temperature equilibration before proceeding.

4. Select "Calibrate**"** on the handheld using the **arrow keys** and press **Enter**. Once inside the calibrate menu use the **arrow keys** to select "Conductivity" and press **Enter**. Select "SpCond" using the **arrow keys** followed by pressing **Enter**.

5. Use the **keypad** to enter the calibration value of the standard you are using (mS/cm or uS/cm at 25°C) and press **Enter**. The current values of all enabled sensors will appear on the screen and may change with time as they stabilize. Record the stabilized reading in the calibration log sheet

6. When the calibration has been accepted, check the conductivity cell constant using the **arrow keys** and **Enter** to select "Cal Constants" followed by "Advanced Menu". The acceptable cell constant is 5.0 ± 0.45.
    a. Numbers outside of this range usually indicate a problem in the calibration process or that a contaminated standard was used. Ensure the sensor is clean and repeat calibration process starting with step one.
    b. If the calibration constant is still out of range, service or replace the probe.

7. Rinse the calibration cup and sensors with tap or DI water and proceed with the next parameter calibration.

3.10.1.3. Rapid Pulse Dissolved Oxygen Probe

The DO membrane and electrolyte solution should be inspected for any damage or air bubbles prior to calibration. If air bubbles or damage are present, replace the membrane according manufacturer manuals. To ensure accuracy, wait at least 4 hours after changing the membrane before using the instrument to allow the membrane to equilibrate.

1. With sonde sensors facing up in the ring stand, place approximately 1/8 inch of water in the bottom of the calibration cup. Make certain that the DO and temperature probes are <u>not</u> immersed in the water.

2. Engage only 1 or 2 threads of the calibration cup to insure that DO probe is vented to the atmosphere. After five minutes, go to the sonde "Report Menu" and record the DO Charge. The number should be between 25 and 75. After 10 minutes, the air in the calibration cup is water saturated and the temperature is equilibrated.

3. Using the **arrow keys** and **Enter** button, from the 650 MDS main menu select "Sonde Menu", then "calibrate", select "DO", then "DO%" to access the DO percent calibration procedure.

4. Enter the current barometric pressure (BP) in mmHg. The BP reading should be located at the bottom of 650 MDS screen.

5. Press **Enter** and the current values of all enabled sensors will appear on the screen and change with time as they stabilize.

6. Observe the reading under DO%. When there is no significant change in the value for approximately 30 seconds (<0.1%), press **Enter**.

7. The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to return to the calibrate menu.

8. Record BP, temperature, calibrated DO in mg/l in the calibration logsheet.

9. Using the chart in Appendix B, calculate the saturated DO value using the temperature and BP readout on the probe. Record the saturated value in mg/L on the logsheet.

10. The calibrated DO should be within ± 0.2 mg/l of the saturated DO value. If not, recalibrate the Sonde.

11. When the calibration is complete, use the **arrow keys** to go to the Sonde's "Advanced Menu" and then to "Cal Constants" and record the "DO Gain". The gain should be between 0.7 and 1.4. If the DO gain is out of range, the probe needs servicing.

3.10.1.4. Optical ROx Dissolved Oxygen Probe

Inspect the optical membrane to ensure you cannot see light through the membrane tip greater than 1 mm in size. To do this, turn on the handheld and sonde and cup your hand to provide shade or use a hollow tube like a cardboard paper towel roll to see the flashes of blue light. If you see blue light on the membrane tip (not the edges), replace the membrane following the maintenance section of this manual or manufacturer instructions.

Note: Be sure to remove the wiper of the DO sensor when calibrating with a turbidity solution that contains formazin or sodium sulfide as this can alter DO readings.

It is good practice to use water from a clean carboy stored at room temperature to perform the calibration. The water will be at room temperature to minimize equalization time. As an alternative, you may use the conductivity standard for calibration of dissolved oxygen.

1. Fill the calibration cup with water so that it covers the optical DO sensor but still has an air gap. You may use the conductivity solution for this step if you did not discard it earlier.

2. Tightly close the lid on the calibration cup and pick up the sonde. Shake vigorously for one to two minutes in a side to side and up and down motion to saturate the water with air.

3. Select "Calibrate" from the main menu, and then select "Optic T Dissolved Oxy".

4. Select "ODO%" and use the **keypad** to enter the current barometric pressure in mmHg.

5. Press **Enter** and the sonde will do a four-second countdown and then say "Press Enter when the readings are stable". Wait at least 30 seconds before you press the **Enter** button

The screen will indicate that the calibration has been accepted and prompt you to press **Enter** again to return the calibrate menu.

Note: The probe will not display 100% saturation unless the barometric pressure is 760 mmHg. To determine the actual DO in % saturation, divide the barometer reading by 760 and then multiply that number by 100.

Example: 750mmHg / 760mmHg = 0.9868 x 100 = 98.68%

6. When the calibration is completed, go to the Sonde's "Advanced Menu" and then to the "Cal Constants" and record the "DO Gain". The gain should be between 0.75 and 1.25. If the DO gain is out of range, the probe needs servicing.

3.10.1.5. pH

1. Go to the sonde "Report Menu" and enable the pH mV output. This will allow the sonde to display the millivolt output fromt eh rpobe as well as the pH units during the calibration process.

2. Choose the appropriate standards that will bracket the expected values at the sampling locations.

3. Rinse the Sonde with tap water first then rinse with used pH 7 buffer solution, discarding the solution after rinsing. Fill the cup with the correct amount of fresh pH 7 buffer standard making sure that the temperature probe is submerged in the standard solution.

4. Allow at one minute for temperature equilibration before proceeding.

5. From sonde menu use the **arrow** and **Enter** keys, select "Calibrate", then "ISE1 pH" to access the pH calibration choices and then select "2 point". Press **Enter** and input the value of 1st pH using the **keypad**. Remember that pH values change based on temperature. Use the pH chart provided on each container of buffer to enter the correct pH value (7.02 at 20 C vs. 7.00 at 25 C).

6. Press **Enter** and the current values of all enabled sensors will appear on the screen and change with time as they stabilize in the solution. Observe the reading under pH and until there is no significant change (<0.1 unit) for approximately 10 seconds, press **Enter**. The display will indicate that calibration is accepted.

7. Record the pH millivolts for pH 7 buffer solution. The acceptable tolerance for pH 7 buffer is 0 ± 50 mv. When a probe is new, the ideal numbers are close to the 0, then as the probe begins to age, the number will move and shift to the higher side of the tolerance.

8. After pH 7 calibration is complete, press **Enter** again to continue. Discard the pH 7 buffer, rinse the sonde with tap water and then rinse with used pH 4 or pH 10 buffer. Discard all rinses.

9. Fill the cup with fresh pH 4 or pH 10 buffer to the appropriate level ensuring that the pH and temperature probes are submerged in the standard solution.

10. Press **Enter** and use the keypad to type in the correct value for the second buffer. Remember that pH values change based on temperature. Use the pH chart provided on each container of buffer to enter the correct pH value (4.01 at 20 C vs. 4.00 at 25 C).

11. Press **Enter** and the current values of all enabled sensors will appear on the screen and change with time as they stabilize in the solution. Observe the readings under pH and when they show no significant change (<0.1 unit) for approximately 10 seconds press **Enter**.

12. Record the pH millivolts for pH 4 or 10 buffer solution. The acceptable tolerance for pH 4 buffer is +180 ± 50 mv and 10 buffer is -180 ± 50 mv. When a probe is new, the ideal numbers are close to (+/-)180. When the probe begins to age, the value will shift to the higher side of the tolerance.

13. After recording the pH millivolts for the calibration points, determine the slope of the sensor. This is the difference between the mV values of the two calibration points that were used. The acceptable range for the slope is 165 to 180. Once the slope drops below 165, replace the sensor.

14. Discard pH 4 or 10 buffer solution and rinse the Sonde with tap water.

3.10.1.6. Turbidity

It is critical to the instrument's operation that the lens covering of the detection unit is kept clean during calibration and field use.

1. Check to make sure the turbidity probe and wiper are clean and free from any material.

2. Rinse the calibration cup with deionized water and ensure it is free of debris.

3. Active the wiper to make sure it is wiping and parking correctly.

4. To begin the calibration, place the 0 NTU standard (clear deionized or distilled water) into the calibration cup provided with your sonde. Immerse the sonde in the water**.**

5. In the turbidity calibration menu, input the value 0 NTU at the prompt, and press **Enter**. The screen will display real-time readings that will allow you to determine when the readings have stabilized.

6. Activate the wiper 1-2 times by pressing the **3 key** "3-Clean Optics" as shown on the screen, to remove any bubbles. After stabilization is complete, press **Enter** to confirm the first calibration and then, as instructed, press **Enter** to continue.

7. Rinse the sensors and calibration cup with the second turbidity standard that is slightly higher than what is expected to be found in the field. Use the **keypad** to input the correct turbidity value in NTU and press **Enter.** View the stabilization of the values on the screen in real-time. As above, activate the wiper with the **3 key**. After the readings have stabilized, press **Enter** to confirm the calibration and then press **Enter** to return to the Calibrate menu.

8. Discard the second standard and thoroughly rinse the probes and calibration cup with tap water

3.10.1.7. Depth

When at the sample site, calibrate the probe in the air near to the water level of the sample site.

1. From the Calibrate menu, select "Pressure-Abs" (or Pressure-Gage if you have a vented level sensor) to access the depth calibration procedure. Input 0.00 or some known sensor offset in feet.

2. Press **Enter** and monitor the stabilization of the depth readings with time. When no significant change occurs for approximately 30 seconds, press Enter to confirm the calibration. This zeros the sensor with regard to current barometric pressure.

3. Press **Enter** again to return to the Calibrate menu.

Note: For best performance of depth measurements, users should ensure that the sonde's orientation remains constant while taking readings. This is especially important for vented level measurements and for sondes with side mounted pressure sensors.

3.10.2. Data Recording

The field parameter data should be recorded and entered into CEDS to the tenth place (do not round based on the hundredth place). For example, if pH reads 6.83, you record 6.8. If pH reads 6.89, you record 7.8

3.10.3. Logging Data

The sonde can be used for to obtain discrete or long term continuous sampling. Unattended deployment involves using the sonde memory to log data. The display unit may be used to store data; however this requires the display to remain with the Sonde during monitoring. In addition, the display must have sufficient memory to save the expected number of data points.

3.10.4. Discrete sample measurement logging to MDS 650

The first step in this application is to make sure that the sample interval is set correctly for the logging study. The default sampling interval is 1 second which needs to be changed to 10 seconds.

1. Highlight the logging setup in the main menu and press "Enter".

2. Press enter at highlighted interval selection and use the arrow keys to scroll to the right and change the interval from 1 second to 10 seconds. Confirm the selection by pressing enter and then press esc to return to main menu.

3. Highlight the sonde run from the main menu and presses enter to begin data display.

4. Place the sonde in the water and then highlight the start logging selection and press "Enter". The display prompts for a filename and site description. For our application, it is not necessary to enter a filename or a site description. Highlight the OK window and press "Enter". The data will be logged to a file in the MDS 650 under the designation NONAME1.

5. The header of MDS 650 changes from logging to stop logging to confirm that the data storage to MDS 650 is active. When the measurement reading is steady, highlight the stop logging and press "Enter" to terminate logging.

6. The file can be viewed by selecting file from the main menu and pressing enter. Next highlight the view file selection and then select the selected file and press "Enter". Use the arrow keys to scroll horizontally in order to view all of the data.

3.10.5. Deploying Sonde for Unattended Logging

When calibrating the DO sensor for unattended sampling where the probe turns on and off at regular intervals, do not warm up the instrument before calibrating (it should be calibrated when the instrument is cool).

1. From the sonde menu, select the "Run/Unattended Sample" option and press **Enter**.

2. Follow the prompts on the screen to prepare the Sonde for unattended sampling including:
    a. Choosing sample interval time
    b. Logging start date
    c. Logging start time
    d. Logging duration (days)
    e. File name to store data
    f. Site name
    g. Battery life (make sure it will cover length of time monitoring)
    h. Memory space
    i. View parameters to log

3. Once these items have been correctly entered, toggle down to "Start logging" and press **Enter**. The display unit should show "stop logging".

4. The Sonde will now begin logging parameters at the entered start time and at the selected interval. Place the sensor guard on the Sonde. Turn the display off and disconnect the communication cable from the Sonde. Place the communication port plug on the Sonde.

Place the Sonde in the desired sample location and securely anchor using the bail on the top of the Sonde.

5. The Sonde is now in place and will continuously record data until it reaches the specified logging end date and time.

3.10.6. Post Sampling Verification and Data Evaluation

During use of the Sonde in the field, the instrument may experience "drift" and operate outside of the expected ranges. To determine the amount of drift the probes must be checked against the calibration standards at the end of sampling event.

3.10.6.1. Rapid Pulse Dissolved Oxygen Probe

The DO membrane and electrolyte solution should be inspected for any damage or air bubbles prior to calibration. If air bubbles or damage are present, replace the membrane according manufacturer manuals. To ensure accuracy, wait at least 4 hours after changing the membrane before using the instrument to allow the membrane to equilibrate.

1. With sonde sensors facing up in the ring stand, place approximately 1/8 inch of water in the bottom of the calibration cup. Make certain that the DO and temperature probes are <u>not</u> immersed in the water.

2. Engage only 1 or 2 threads of the calibration cup to insure that DO probe is vented to the atmosphere. After five minutes, go to the sonde "Report Menu" and record the DO Charge. The number should be between 25 and 75. After 10 minutes, the air in the calibration cup is water saturated and the temperature is equilibrated.

3. Record the current barometric pressure and stabilized temperature on the post check portion of the log sheet such as the one found in Appendix A.

4. Using the chart in Appendix B, calculate the saturated DO value using the temperature and BP readout on the probe. Record the saturated value in mg/L on the logsheet.

5. Record the stabilized DO reading (<0.1 mg/L change in 10 seconds).

6. The displayed DO reading should be less than 0.5 mg/L of the saturated DO value. If not, do not enter the DO data into CEDS from when the last check was performed (wet towel check or morning calibration).

7. When the check is complete, use the **arrow keys** to go to the Sonde's "Advanced Menu" and then to "Cal Constants" and record the "DO Gain". The gain should be between 0.7 and 1.4. If the DO gain is out of range, the probe needs servicing.

3.10.6.2. Optical ROx Dissolved Oxygen Probe

It is good practice to use water from a clean carboy stored at room temperature to perform the calibration. The water will be at room temperature to minimize equalization time. As an alternative, you may use the conductivity standard for calibration of dissolved oxygen.

1. Fill the calibration cup with water so that it covers the optical DO sensor but still has an air gap. You may use the conductivity solution for this step if you did not discard it earlier.

2. Tightly close the lid on the calibration cup and pick up the sonde. Shake vigorously for one to two minutes in a side to side and up and down motion to saturate the water with air.

3. Place the sonde back in the ring stand so that the sensors are pointed up and loosen the lid to allow air passage. Allow the probe to equilibrate for five minutes.

4. Record the current barometric pressure and stabilized temperature on the post check portion of the log sheet such as the one found in Appendix A.

5. Using the chart in Appendix B, calculate the saturated DO value using the temperature and BP readout on the probe. Record the saturated value in mg/L on the logsheet.

6. Record the stabilized DO reading (<0.1 mg/L change in 10 seconds).

7. The displayed DO reading should be less than 0.5 mg/L of the saturated DO value. If not, do not enter the DO data into CEDS.

8. When the check is complete, use the **arrow keys** to go to the Sonde's "Advanced Menu" and then to "Cal Constants" and record the "ODO Gain". The gain should be between 0.85 and 1.15. If the ODO gain is out of range, the probe needs servicing.

3.10.6.3. pH and conductivity

1. Clean all the probes on the Sonde with deionized water. Rinse the probe with used standard calibration solutions.

2. Place calibration standard solution of particular parameter (e.g. pH, conductivity, etc.).

3. Allow measurements to stabilize, record the data in the logsheet.

4. The post sampling verification data should be compared with QC limits listed in table 3.2. If the data do not meet these criteria, the entire parameter data collected on that day should not be recorded in CEDS.

3.10.7. Data and Records Management

All the results of calibration, post sampling verification and field data sheets must be documented and kept in a safe place for five years. Enter field data meeting QA into CEDS.

3.10.8. Maintenance

See Table 3.6

Table 3.6. YSI Series 6 Multiprobe General Maintenance

Sensor Type	Item	Procedure
Clark Cell DO Sensor	**Changing DO membrane.** **Every 30 days, or if daily post check fails, or membrane is torn, wrinkled, has air bubbles, or sensor readings are erratic or slow.**	1. Remove the O-ring securing the membrane. 2. Shake out odd electrolyte. Rinse with DI water. 3. Refill with fresh DO electrolyte solution (2M KCL) until a positive meniscus of electrolyte forms above the surface of the sensor. Remove bubbles trapped in the electrolyte solution by tapping the side of the DO sensor. 4. Holding a clean DO membrane film between the thumb and index fingers of both hands, stretch the membrane over the sensor face 5. Place the new O-ring over the gold cathode. Do not use any type of grease on the O-ring. Secure the membrane with the O-ring by rolling across with your thumb. 6. Check to be sure membrane has no air bubbles, wrinkles or tears on the area face enclosed by the O-ring. 7. Carefully trim the excess membrane extending below the O-ring with the pair of scissors or razor blade. 8. Allow the membrane to relax for 4-24 hours before calibration for field use.
	Cleaning silver anode. **Check for majority of anode is dull gray or 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: high strength ammonia will dissolve silver faster so constantly check the anode if using high strength ammonia. 2. Remove electrode and rinse with DI water and blot dry. 3. If still tarnished, use a moistened 400 or finer grit sandpaper such as provided in the membrane kit and gently buff the surface. Rinse off sanding residue with triple rinses of DI 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 DO membrane.**	1. Clean by wiping with a clean lent-free cloth, pencil eraser or hard paper. The color should be matte gold. 2. If cathode is still tarnished, use a moistened 400 or finer grit sandpaper such as provided in the membrane kit and gently evenly buff the surface. 3. Rinse electrode three times with DI water and blot dry. 4. Add a new membrane using steps listed above.
Optical DO Sensor	**Cleaning sensor**	1. Flush the entire sensor with clean, fresh water. 2. Inspect the membrane cap. Use a Kimwipe™ (or similar lint free lab tissue) to wipe away any foreign material. 3. If rinsing does not work, you may soak the sensor in household strength white vinegar for 15 minutes followed by 15 minutes soak in DI water. 4. Rinse with fresh water. **DO NOT clean with alcohol or organic solvents as this will destroy the membrane.** **DO NOT remove the membrane unless you intend to replace the membrane cap.**
	Rehydrate optical membrane **If sensor is not kept in a 100% humid environment longer than 2 hours**	1. Remove the optical sensor from the housing. 2. Place 400 mL of water in a 600 mL glass beaker. Do not use plastic containers. 3. Heat the water on a hotplate or in an oven to a temperature of 50 +/-5 C. 4. Place the sensor tip containing the sensor membrane in the water and maintain the elevated temperature for 24 hours. Cover the beaker to reduce evaporation. 5. After rehydration is complete, store the sensor in either water or water-saturated air at room temperature prior to calibration and deployment.
	Replacing wiper **When appears to be worn or dirty**	1. Remove the wiper assembly using the supplied hex wrench. 2. Replace old wiper assembly with a new wiper 3. Reattach wiper using the supplied hex wrench.

Table 3.6 YSI Series 6 Multiprobe General Maintenance –continued

Sensor Type	Item	Procedure
Optical DO Sensor (cont)	**Replacing membrane** **Once per year or damaged membrane or LED seen through scratches (≥1mm)**	1. Remove the old membrane and wiper assembly using the supplied hex wrench. 2. Screw in new membrane cap to the sensor. Tighten to finger tightness. a. Do not touch the outer or inner surface of membrane with your fingers 3. Reattach wiper assembly using the supplied hex wrench.
Conductivity Sensor	**Cleaning conductivity sensor** **As needed**	If the multiprobe housing blocks direct access to the conductivity sensor, detach the sensor from the housing following manufacturer instructions. 1. Using a paper towel or cotton swab, clean the sensor of hardened foreign material using warm water and mild detergent or methanol. 2. Rinse sensor with DI water and blot dry. 3. If needed, repeat using a soft brush. Hard scouring will damage the sensor.
pH sensor	**Cleaning pH electrode** **Slow readings, when obviously coated with foreign material or the glass sensor is scratched**	1. Remove the sensor from the multiprobe housing 2. Soak pH sensor in warm water with mild detergent for 10-30 minutes. 3. If ineffective, clean glass bulb sensor with a cotton ball/swab or lint free cloth soaked in methanol. 4. If ineffective or the sensor shows slow response, soak several minutes with 10% HCl or several hours with pH 4 buffer. 5. After cleaning, rinse well with DI water and blot dry. 6. Reattach sensor to the multiprobe housing, apply grease if needed to the O-ring.
Temperature sensor	**General cleaning**	Clean as necessary to remove hardened deposits using mild soap and water and a cloth. Do not bend, dent, or nick the sensor as this may alter temperature readings
General	**Short term storage (<1 month)**	Place a small amount of clean water (1/4 inch or so) to the storage cup and securely attach to the sensor end of the unit. Be sure the water does not directly contact sensors.
	Long term storage (>1 month)	1. Remove batteries (size AA or C) from units which will be in storage for more than 30 days. Do not remove the lithium battery (silver disk) from the multiprobe housing as this maintains the internal clock. 2. If possible, remove the pH sensor from the unit and place the glass bulb end into the provided pH storage bottle with pH electrode storage solution or 4.0 buffer 3. Do not remove the dissolved oxygen sensor. It must be stored in a humid environment. 4. Conductivity and temperature sensors may be stored in a dry or wet condition. 5. Secure all exposed ports with provided port caps. 6. Add ½ inch of clean water to the calibration cup and securely attach it to the sonde housing. Check periodically (30 days) to ensure water has not evaporated or become contaminated.

3.11. In-Situ Multiprobe Troll 9500 with Rugged Reader

3.11.1. Calibration

3.11.1.1. General procedures for calibration

The calibration cup's fill lines indicate the recommended amount of solution for most calibration standards and ensure the temperature sensor is immersed. With a full complement of sensors, use the lower line as a guide. Except for the 100% dissolved oxygen calibration, the temperature sensor should be immersed in about an inch of fluid.

1. First rinse the sensors with tap water, then with distilled or analyte water followed by a rinse with the solution to be used for calibration.

2. There are three calibration status indicators.
   a. Unstable: indicates the sensor response does not meet the criteria for a valid calibration point.
   b. Nominal: indicates the sensor deviation meets early stabilization criteria.
   c. Stable: displays when the readings have stabilized sufficiently to take a valid calibration point. The calibration proceeds automatically to the next screen.

3. Each time a sensor is calibrated, the information is written to the sensor, where it is stored until the next calibration.

4. After a certain amount of use, the instrument will not be able to calculate the calibration coefficients accurately. The slope will gradually become lower and lower. At this point, the sensor should be replaced

3.11.1.2. Specific Conductance

1. Remove the black PVC end cap from the calibration cup and screw the top of the stirrer to the bottom of the calibration cup.

2. Fill the calibration cup to the lowest fill line with solution.

3. Select "MP TROLL 9000" in the navigation tree.

4. Select "Conductivity" in the parameter list.

5. Select "Calibrate".

6. Select the calibration solution the sensor is soaking in. If the calibration solution concentration is not in the selection. Select other and enter the specific conductance of the solution in µS/cm.

7. Select "Next" to continue.

8. In the following screen, select "Run" to begin the stabilization.

9. When the sensor reaches the stable stage, the display unit will move to the final screen showing the new cell constant as calculated for the selected range during the calibration process.

10. Select finish to program the sensor with the displayed cell constant. The cond cell constant should range between 0.33-0.43. If the constant is out of the specified range, check the sensor for fouling of the electrode.

11. Record the calibration conductivity value in the calibration logsheet.

12. The conductivity sensor is now calibrated and ready to use in the range for which it was calibrated.

3.11.1.3. pH

1. Rinse the calibration cup with tap water, followed by distilled or DI water and first pH standard to be calibrated.

2. Fill calibration cup (with stirrer attached) with the standard solution to the lowest fill line.

3. Select “MP TROLL 9000” in the navigation tree.

4. Select “pH” in the parameter list.

5. Select “Calibrate”.

6. Select the number of calibration points for this calibration, and the pH value of the calibration solution for each point. Since the range of pH encountered in the field may be unknown. Calibrating to three points (4, 7, and 10 buffers) is recommended, but a two point calibration bracketing expected values is acceptable.

7. Select “Next” to continue.

8. Select “Run” to begin the stabilization.

9. After the calibration is recorded in the sonde, the wizard returns to the previous screen and waits for you to situate the probe in the next calibration solution.

10. Remove the calibration cup, discard the solution, rinse the calibration cup with tap, DI water and the next standard solution. Refill the calibration cup with the next solution and attach it to the instrument.

11. Select “Run” to begin the stabilization for the second calibration point.

12. Repeat step 9 and 10 if doing the third calibration point.

13. The final screen shows the sensor slope and offset calculated during the calibration process. For a 3-point calibration, two sets of calculated coefficients will be shown.

14. Offsets calculated by the software for pH 7 will typically be between 390-450 mV. If the offset falls outside these limits, it may be time to replace the sensor. The calculated slope will be typically between -54mV and -62 mV. A calculated slope greater than -50mV or less than -66mV may indicate the sensor needs replacing.

15. Select "Finish" to program the sensor with the newly calculated calibration coefficients.

16. Record the pH 7, 4 and/or 10 calibration values, offsets, and slopes on the calibration logsheet.

3.11.1.4. Dissolved Oxygen

1. Invert the TROLL so the DO sensor faces upward.

2. Fill the calibration cup (without stirrer attached) with water to just below the DO membrane.

3. Use soft swab or the corner of a tissue to remove moisture from the membrane.

4. Reattach the end cap loosely to insure the chamber is not pressurized. For proper venting, loosen the end cap until the small hole in the treads near the O-ring is visible.

5. Select the "TROLL 9000" in the navigation tree.

6. Select "Dissolved Oxygen" in the parameters list.

7. Select calibrate.

8. If the instrument has a vented cable, choose use vented cable. Click OK to proceed.

9. Select continuous mode and one point calibration.

10. Click next to continue.

11. Select run to begin the stabilization for 100% DO calibration point.

12. The final screen shows the sensor slope and offset calculated during the calibration process. The slope of a properly functioning sensor should be between 25 and 35 nA/(mg/l). If the slope is slightly outside this range, repeat the calibration. The sensor may require further conditioning. Sensors that repeatedly fall outside this range may require a new membrane. The default offset for a one-point calibration is 2nA. Offsets for two-point calibrations should be less than 10 nA.

13. Select finish, to program the sensor with the newly calculated calibration coefficients.

14. Record saturated DO from table, calibration DO and the DO slope on the logsheet.

15. The DO sensor is now calibrated and ready to use.

3.11.2. Set up

The short calibration cable is just used for calibration; switch the cable to be utilized in the field.

1. Remove the storage cup from TROLL, screw on the restrictor and store the sensor in a moist environment when not in use.

2. At the station, turn on the IPQA.

3. Select MP TROLL 9000 in the navigation tree.

4. Select parameters in the navigation tree.

5. Select profiler to start the profiler.

6. After a moment, each active channel will be read sequentially, and the reading will be displayed. Readings are updated approximately every two seconds as the profiler cycles through the available channels in turn. The currently selected measurement unit is shown below each reading. The field parameter data should be recorded and entered into CEDS to the tenth place (do not round based on the hundredth place).

7. When you are ready to exit the profiler, click close. The parameter view will return to the screen.

3.11.3. Logging profiler data

To log a single set of profiler readings, click or tag the snapshot button in the view. The snapshot button flashes when logging. When it is done, it returns to not logging status.

Retrieving logged profile data:

1. Expand the data folder in the navigation tree by tapping the +.

2. Expand the node for the device type and serial number.

3. Look for the data file named with date and time of the profiler reading.

3.11.4. Maintenance

See Table 3.7

Table 3.7 In-Situ Troll 9500 Multiprobe General Maintenance

Sensor Type	Item	Procedure
Clark Cell DO Sensor	**Changing DO membrane.** **Every 30 days, or if daily post check fails, or membrane is torn, wrinkled, has air bubbles, or sensor readings are erratic or slow.**	1. Make sure the area around port 2 is free of dirt and moisture, then remove the sensor. Remove and discard the used membrane. 2. Fill electrode well or cap with solution provided in the membrane replacement kit. 3. Dislodge any bubbles by tapping the side of the cap or sensor with a pencil 4. Lower the electrode into the membrane cap and tighten to finger tightness. 5. Turn the electrode over to note any air bubbles or wrinkles. If so, discard the membrane and try again. 6. Install the sensor back to the multiprobe. Add O-ring grease if needed. 7. Allow the sensor to equalize (4 to 24 hours) before calibrating deployment.
	Cleaning silver anode. **Check for majority of anode is dull gray or 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: high strength ammonia will dissolve silver faster so constantly check the anode if using high strength ammonia. 2. Remove electrode and rinse with DI water and blot dry. 3. If still tarnished, use a moistened 400 or finer grit sandpaper such as provided in the membrane kit and gently buff the surface. Rinse off sanding residue with triple rinses of DI 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 DO membrane.**	1. Clean by wiping with a clean lent-free cloth, pencil eraser or hard paper. The color should be matte gold. a. If cathode is still tarnished, use a moistened 400 or finer grit sandpaper such as provided in the membrane kit and gently evenly buff the surface. 2. Rinse electrode three times with DI water and blot dry. 3. Add a new membrane using steps listed above.
Optical DO Sensor	**Cleaning sensor**	1. Flush the entire sensor with clean, fresh water. 2. Inspect the membrane cap. Use a Kimwipe™ (or similar lint free lab tissue), to gently wipe away any foreign material on the sensor cap. 3. If rinsing does not work, you may soak the sensor in household strength white vinegar for 15 minutes followed by 15 minutes soak in DI water. 4. Rinse with fresh water. **DO NOT clean with alcohol or organic solvents, as this will destroy the membrane.** **DO NOT remove the membrane cap unless you intend to install a new cap.**
	Replacing membrane **Once per year or damaged membrane or LED seen through scratches (≥1mm)**	1. Pull the used sensor cap off of the sensor, without twisting. 2. Remove the existing O-rings from the sensor. 3. Use a lint-free cloth to remove any moisture from the sensor body. 4. Using your finger, apply a layer of grease around the O-ring grooves. Place the O-rings on the sensor. Apply a thin layer of grease to the O-rings and grooves. *Do not transfer grease to the lens or sensor pins.* 5. Clean the lens on the sensor with the wipe provided in the kit and allow to dry thoroughly. Inspect for scratches or dirt. 6. Remove the new cap from its sealed packaging and attach it to the sensor. Carefully press the cap firmly, without twisting, until it seals over the probe body. Make sure not to pinch or roll the O-rings between the cap and sensor. 7. Perform a 2-point (100% and 0%) calibration.
Conductivity Sensor	**Cleaning conductivity sensor.** **As needed**	If the multiprobe housing blocks direct access to the conductivity sensor, detach the sensor from the housing following manufacturer instructions. 1. Using a paper towel or cotton swab, clean the sensor of hardened foreign material using warm water and mild detergent or methanol. 2. Rinse sensor with DI water and blot dry. 3. If ineffective, repeat the process using a soft brush. Care is needed as hard scouring easily damages the sensor.

Table 3.7 In-Situ Troll 9500 Multiprobe General Maintenance –continued

Sensor Type	Item	Procedure
pH Electrode	**Cleaning pH electrode.** **Slow readings, when obviously coated with foreign material or the glass sensor is scratched**	1. Remove the sensor from the multiprobe housing 2. Soak pH sensor in warm water with mild detergent for 10-30 minutes. 3. If ineffective, clean glass bulb sensor with a cotton ball/swab or lint free cloth soaked in methanol. 4. If ineffective or the sensor shows slow response, soak a few minutes with 10% HCl or several hours with pH 4 buffer. 5. After cleaning, rinse well with DI water and blot dry. 6. Reattach sensor to the multiprobe housing; apply grease if needed to the O-ring.
	Replacing the reference solution **Every four to six months or if readings drift or if pH 7 buffer has a mV value is outside 0±20 mV**	1. Unscrew the reference junction cap located at the end of the sensor. 2. Holding the sensor at an angle, shake out the filling solution. 3. Protect the connector end of the sensor with the soft cap it shipped with, or wrap the sensor in a paper towel to prevent solution from entering the electrical connector. 4. Using the dispenser cap on the filling solution bottle, insert the tube into the bottom of the empty reservoir. Squeeze a steady stream of solution into the reservoir until it overflows and no bubbles are observed. Continue to add solution while withdrawing the tube. 5. Screw in the reference junction, and hand-tighten until snug. Some filling solution will overflow. Wipe the excess off the sensor body. 6. Soak the sensor in tap water for at least 15 minutes. 7. Recalibrate the sensor.
Temperature Sensor	**General cleaning**	Clean as necessary to remove hardened deposits using mild soap and water and a cloth. Do not bend, dent, or nick the sensor as this may alter temperature readings
General	**Removing sensors** **For inspection, cleaning and storage**	1. Remove a sensor by positioning the yoke of the sensor removal tool at the point where the sensor enters the sensor block. 2. Firmly pry the sensor upward until it pops out.
	Installing sensors	1. Check lubrication of the sensor O-rings. If the O-ring appears dry, apply a silicone lubricant before installing. 2. Align the mark on the sensor with the alignment mark on the correct port. 3. Press the sensor with sensor insertion tool firmly into the port until it is securely seated. When properly inserted, a gap of about 0.06" (width of the sensor removal tool) remains between the widest part of the sensor and the instrument body.
	Short term storage (<1 month)	Place a small amount of clean water (1/4 inch or so) to the storage cup and securely attach to the sensor end of the unit. Be sure the water does not directly contact sensors.
	Long term storage (>1 month)	1. Remove batteries (size D) from units which will be in storage for more than 30 days. Do not remove the lithium battery (silver disk) from the multiprobe housing as this maintains the internal clock. 2. If possible, remove the pH sensor from the unit and place the glass bulb end into the provided pH storage bottle with pH electrode storage solution or 4.0 buffer 3. Do not remove the dissolved oxygen sensor. It must be stored in a humid environment. 4. Conductivity and temperature sensors may be stored in a dry or wet condition. 5. Secure all exposed ports with provided port caps. 6. Add ½ inch of clean water to the calibration cup and securely attach it to the sonde housing. Check periodically (30 days) to ensure water has not evaporated or become contaminated.

CHAPTER 4: FIELD SAMPLING PROCEDURES

The chapter details the collection of water and sediment samples utilized by nearly all agency water quality monitoring programs.

4.1. Use of protective gloves

Gloves serve a dual purpose:

1) Protecting the sample collector from potential exposure to sample constituents and
2) Minimizing accidental contamination of samples by the collector.

Wearing protective gloves at all times while sampling is recommended, however, their use is not mandatory if:

1) The sample source is considered to be non-hazardous, or
2) The samples will not be analyzed for trace (i.e. part per billion levels) constituents.

Latex or nitrile gloves may be used for common sampling conditions. The use of new, disposable, powder-free latex or nitrile gloves is required for clean metal sampling. Discarding the gloves is optional but in no circumstances should the gloves be reused for clean metal or similar trace analysis sampling.

4.2. Equipment Rinse

When collecting samples, the collection equipment shall be rinsed once with sample water before the actual sample is taken. For a sampling bucket, fill the bucket with site water, swirl the water around and dispose of the rinse water away from the sampling site. For sampling devices, clean the device inside and out by dipping it into and out of the site water or by washing with site water.

4.3. Surface Water sampling

4.3.1. General

1. Safety always comes first. All sampling should be conducted with the proper equipment and least amount of danger to field personnel. Use of safety cones and vests is recommended. Additional sampling safety information is in Chapter 7.

2. Sampling staff are responsible for determining safe site access. If access is needed on private property, staff must obtain permission from landowners.

3. Care should be taken not to disturb the bottom when sampling. When entering a stream, always walk in an upstream direction towards the sample site.

4. If collecting water samples and sediment samples at the same location, collect water samples first to avoid disturbed sediment contaminating the water sample.

5. Collect surface water samples facing upstream and in the center of main area of flow. Unless safety is an issue, obtain samples from a bridge or directly instream.

6. Whenever possible, collect field measurements (DO, pH, temp, etc.) directly form a stream and not from a sample bucket. If the field parameters need to be measured in the bucket, collect water quality samples (nutrients, etc.) first before placing the multiprobe instrument in the bucket.

7. If utilizing discrete sampling bottles follow the manufacturer's instructions for maintenance, cleaning and use.

8. When there are obvious standing pools of water during low or no flow conditions, do not collect samples or field measurements. Make a note of this on the field sheet and upon return to the office include a remark in CEDS explaining the site conditions.

9. Prior to sampling, rinse sample bottles with a small amount of sample water (except bacteria bottles or if sample bottles contain acid preservative). Dump rinse water away from the sample bucket or sample location.

10. When collecting bacteria samples:

    a. Do not rinse the bacteria sample bottle before collecting the sample.

    b. If sample bottles contain a dechlorinating tablet (usually small white tablet) and you are collecting an unchlorinated sample, dump out the tablet before collecting the sample. Do not rinse out any dechlorinating residue from the bottle.

    c. Never collect bacteria samples in an unsterilized sample container and transfer to a sterile container.

    d. Be careful not to insert fingers into the mouth of the container or on the interior of the cap. Bacteriological sampling must always be collected as a grab sample and must never be composited.

    e. If the volume of sample exceeds the mark on the bacteria sample bottle, pour off sufficient sample so the volume is approximately equal to or above the mark on the bottle shoulder and securely cap and label the container.

11. Cubitainers may be opened for filling by either removing the cap and pulling it open, or by removing the cap and blowing it open. Rinse cubitainers with sample water prior to filling. This is especially true with cubitiainers inflated by mouth.

12. Except for specific, non-routine samples such as VOCs, do not fill sample bottles completely. An air space is necessary to ensure proper mixing by the laboratory. Usually filling bottles to shoulder or neck is sufficient.

4.4. Sampling from a bridge

4.4.1. General

1. Safety always comes first. All sampling should be conducted with the proper equipment and least amount of danger to field personnel. Use of safety cones and vests is recommended. Additional sampling safety information is in Chapter 7.

2. Sample in the center of main flow from the safest side of the bridge and where contamination is least likely to occur. Typically, the upstream side of the bridge will have less chance of contamination.

3. During rainy periods, avoid sampling where storm water runoff from the bridge can affect the sample.

4. Collect field parameters before deploying the sample bucket. If the field parameters need to be measured in the bucket, collect water quality samples (nutrients, etc.) first before placing the multiprobe instrument in the bucket.

5. When there are obvious standing pools of water during low or no flow conditions, do not collect samples or field measurements. Make a note of this on the field sheet and upon return to the office include a remark in CEDS explaining the site conditions.

6. Prior to sampling, rinse sample bottles with a small amount of sample water (except bacteria bottles or if sample bottles contain acid preservative). Dump rinse water away from the sample bucket or sample location.

7. Cubitainers may be opened for filling by either removing the cap and pulling it open, or by removing the cap and blowing it open. Rinse cubitainers with sample water prior to filling. This is especially true with cubitainers inflated by mouth.

8. Except for specific, non-routine samples such as VOCs, do not fill sample bottles completely. An air space is necessary to ensure proper mixing by the laboratory. Usually filling bottles to shoulder or neck is sufficient.

4.4.2. Bacteria Sampling Using a Modified Stainless Steel Bucket

1. Lower the bucket into the center of main flow facing into the current and carefully rinse the bucket with ambient water one or two times. Raise the bucket after rinsing.

2. Do not rinse the bacteria sample bottle before collecting the sample.

3. If sample bottles contain a dechlorinating tablet (usually small white tablet) and you are collecting an unchlorinated sample, dump out the tablet before collecting the sample. Do not rinse out any dechlorinating residue from the bottle.

4. Remove the cap on the bacteria sample bottle and place the bottle into the rubber tubing on the inside of the bucket.

5. Place the rope through the clip located on the side of the bucket.

6. Slowly lower the bucket into the center of main flow. Once the bucket has a sufficient sample, jerk the rope to free the rope from the clip and pull the bucket up toward you. There should be sufficient water to fill the bacteria sample bottle.

7. Remove the bacteria sample bottle from the rubber tubing and cap it. Do not pour additional sample into the bacteria sample bottle or dip the bacteria sample bottle into the bucket to increase the volume of the sample.

8. If the volume of sample exceeds the mark on the bacteria sample bottle, pour off sufficient sample so the volume is approximately equal to or above the mark on the bottle shoulder and securely cap and label the container.

9. Place the bacteria sample into the mesh bag and surrounded with wet ice.

10. If collecting a chlorophyll sample, follow the steps described in Section 4.7 or 4.8 prior to collecting any further samples.

11. Pour the remaining samples directly from the bucket into any additional containers to be collected and cap them. Leave approximately one inch of air space in the bottles.

12. Add appropriate preservatives as described in the DCLS catalog in CEDS. Note: DCLS may add preservatives to the sample bottles upon receipt if they are not processed promptly to meet unpreserved holding times.

13. Place chlorophyll filters separately into Ziploc bags, close the bags and place them in the cooler on top of the wet ice. Make sure the opening of the bag containing the chlorophyll filter hangs out of the cooler when the cooler lid is closed.

14. Place all other sample containers in the cooler up to the neck of the bottle with wet ice.

4.5. Streambank and Instream Sampling

4.5.1. General

If possible, wade into the stream to collect the sample. If streambanks are too steep or stream flow or volume is unsafe as outlined in Chapter 7, sampling from the streambank is permitted

When sampling from the streambank, care should be taken to sample from an area that will most closely represent the entire stream. Typically, this will be the area of the greatest flow in the stream and away from stagnant pools or eddies.

If all samples are obtained directly from the stream, any necessary preservatives should be added after obtaining the grab sample.

When residual chlorine may be present, the sodium thiosulfate tablet can be added in the bottle later for bacterial samples if not already included in the bottle.

4.5.2. Instream Sample Collection

1. Walk upstream to the sample location. Be sure any sediment or debris disturbed from your movement in the streambed is not present where you will be submerging your sample bottle.

2. Collect the bacteria sample bottle first.
   a. Submerge the bacteria bottle; neck first into the water. The mouth of the bottle should be below the water surface approximately 3-6 inches.

   b. Invert the bottle so the neck is upright and pointing into the water flow.

   c. Move the bottle forward away from the body for at least six inches.

   d. Return the filled container quickly to the surface. Pour any excess water and cap.

3. Rinse the sample bucket with stream water 2 times. Pour the water away from where you will be collecting the sample. Fill the bucket and return to shore to fill the remaining containers.

4. Place the bacteria sample into the mesh bag and surrounded with wet ice.

5. If collecting a chlorophyll sample, follow the steps described in Section 4.7 or 4.8 prior to collecting any further samples.

6. Pour the remaining samples directly from the bucket into any additional containers to be collected and cap them. Leave approximately one inch of air space in the bottles.

7. Add appropriate preservatives as described in the DCLS catalog in CEDS. Note: DCLS may add preservatives to the sample bottles upon receipt if they are not processed promptly to meet unpreserved holding times.

8. Place chlorophyll filters separately into Ziploc bags, close the bags and place them in the cooler on top of the wet ice. Make sure the opening of the bag containing the chlorophyll filter hangs out of the cooler when the cooler lid is closed.

9. Place all other sample containers in the cooler up to the neck of the bottle with wet ice.

4.5.3. Streambank Sample Collection

1. If wading into the stream is not possible, using a bucket attached to a rope, throw the bucket to the water where the flow is the most representative of the stream. Allow the bucket to partially fill and retrieve using the rope.

2. Rinse the sample bucket with stream water and discard rinses away from the water to avoid disturbing sediment. Repeat rinse one more time.

3. After rinsing, throw the bucket into the stream at the most representative location of the stream and allow the fill. Carefully retrieve the bucket with the rope avoiding disturbing the sediment to the point it may enter the bucket.

4. After retrieving the bucket, collect the bacteria sample bottle first.
   a. Using a clean pair of latex or nitrile gloves, submerge the bacteria bottle; neck first into the water. The mouth of the bottle should be below the water surface approximately 3-6 inches.

   b. Invert the bottle so the neck is upright and pointing into the water flow.

   c. Move the bottle forward away from the body for at least six inches.

   d. Return the filled container quickly to the surface. Pour any excess water and cap.

5. Place the bacteria sample into the mesh bag and surrounded with wet ice.

6. If collecting a chlorophyll sample, follow the steps described in Section 4.7 or 4.8 prior to collecting any further samples.

7. Pour the remaining samples directly from the bucket into any additional containers to be collected and cap them. Leave approximately one inch of air space in the bottles.

8. Add appropriate preservatives as described in the DCLS catalog in CEDS. Note: DCLS may add preservatives to the sample bottles upon receipt if they are not processed promptly to meet unpreserved holding times.

9. Place chlorophyll filters separately into Ziploc bags, close the bags and place them in the cooler on top of the wet ice. Make sure the opening of the bag containing the chlorophyll filter hangs out of the cooler when the cooler lid is closed.

10. Place all other sample containers in the cooler up to the neck of the bottle with wet ice.

4.6. Sampling From a Boat

4.6.1. General

1. Collect bacteria samples from the water in the direction of the current. Do not collect bacteria samples using a pump and hose. Do not contaminate the sterile bottle by touching the inner surfaces of the bottle or cap.

2. Collect all water samples before collecting sediment samples.

3. Make sure that hose is adequately marked to ensure accurate readings of the depth of the hose intake.

4. If possible, sample as far away from the engine as possible in the direction of the current.

5. Place the bacteria sample into the mesh bag and surrounded with wet ice.

6. If collecting a chlorophyll sample, follow the steps described in Section 4.7 or 4.8 prior to collecting any further samples.

7. Pour the remaining samples directly from the bucket into any additional containers to be collected and cap them. Leave approximately one inch of air space in the bottles.

8. Add appropriate preservatives as described in the DCLS catalog in CEDS. Note: DCLS may add preservatives to the sample bottles upon receipt if they are not processed promptly to meet unpreserved holding times.

9. Place chlorophyll filters separately into Ziploc bags, close the bags and place them in the cooler on top of the wet ice. Make sure the opening of the bag containing the chlorophyll filter hangs out of the cooler when the cooler lid is closed.

10. Place all other sample containers in the cooler up to the neck of the bottle with wet ice.

4.6.2. Collection of Samples With a Pump and Hose

Two methods may be utilized to ensure a sufficient volume of water is allowed to flow through the hose to clear it:

A) Air Bubble method:

Rinse the pump and hose using the air bubble method by turning the pump on with the hose intake out of water to draw air into the hose. Then turn the pump off and lower the hose to the desired sampling depth. Turn the pump back on and wait for the air to completely exit the hose then let the pump run an additional 30 seconds before rinsing and filling the sample containers.

B) Calculated clearing time method:

Calculate the volume of the hose in gallons: $(r/12)^2 * 3.14 * L * 7.48 = V$

Where
r = radius (inches) of hose inner diameter
L = length (feet) of hose
V = volume (gallons)

Determine pump capacity (gallons per minute pumped) from the pump specifications.

Calculate time to flush hose: V/ (gpm) = time

Where
gpm = gallons per minute pumped
time = minutes to flush hose
Multiply the time by 1.25 as a minimum clearing time for the hose.

Note: Each time a different length or diameter of hose or pump is installed, the clearing time needs to be calculated in the above manner to ensure the waiting time is sufficient to clear the hose.

1. When sampling close to the surface make sure the hose does not come out of the water and inadvertently pull some surface skim water through the hose.

2. Lower the hose to the desired depth, turn on the pump and wait the time calculated to clear the hose or for the bubbles to clear from the hose to adequately rinse the hose before filling the containers. The pump and hose must be rinsed between each sample depth.

3. After filling the sample containers, filter and preserve the sample as required. Check the containers and label for the correct station, date, time, depth, and sample type and put the appropriate labels on all samples before collecting another set of samples. Place filter in Ziploc bags and put the bags on the top of ice. Place all sample containers in a cooler and surround the samples with wet ice.

4.6.3. Secchi Disk Measurements

1. Use a Secchi disk measuring 20 cm in diameter and attached to a line or chain marked in 0.1 m increments with paint or tape. **Note: the marks need to be checked once a year with a measuring tape for accuracy.**

2. Lower the Secchi disk into the water on the shaded side of the boat until the black and white quadrants are no longer distinguishable. **Do not wear sunglasses while obtaining this reading.**

3. Note the depth where the line meets the water when the disk is no longer distinguishable. Lower the disk several increments and then raise the disk until the quadrants are again distinct. Note the depth of this second reading.

4. The recorded Secchi depth is the average of the two depths to the closest 0.1 m.

4.6.4. Light Attenuation

LICOR sensors need to be recalibrated every 2 years. Optimally, rotation of sensors should occur to allow only a single year of use in the field. However, no sensors will be utilized in the field for periods greater than 2 years. The Photo Diodes will degrade even when the sensors are not deployed in the field therefore each region will need to track purchases and recalibration dates accordingly. Additionally, regions need to track the dates sensors are utilized in the field.

Purchases/recalibrations and utilization dates should be tracked for each sensor utilizing a LI-COR sensor tracking sheet. The tracking sheets should be kept in a logsheet at each region.

The LICOR instrument requires an air or water multiplier for each sensor depending on the media it will be utilized in. The multiplier is the calibration coefficient for each sensor and is specified on the calibration documentation for each sensor. While the multipliers are stored in the data logger and do not need to be entered prior to each use, a new multiplier is required when installing a new sensor or replacing a sensor recalibrated by the manufacturer.

A. Entering multipliers/ initial set up for LI-1400:

1. With the front panel face up and the display at the top, connect the light sensor into the BNC connector I1 located on the top left of the LI-1400 unit and the underwater sensor into the BNC connector I3 located on the top right of the LI-1400 unit.

2. Turn on the instrument by pressing the key labeled **ON.**

3. Press the key labeled **Setup** on the data logger.

4. Select **CHANNELS** and press **ENTER**.

5. Using the right or left arrow keys select **I1=light**. Press **ENTER**.

6. Pressing the **shift** key to access the alpha characters on the numeric key pad (press shift once for the upper character or twice for the lower character) type QUANTUM for the description. Press ↓.

7. Type in the multiplier for use in air from the tag attached to the air sensor or from the most recent certificate of calibration. Press **ENTER**.

8. Type SA for the label**.** Label is a two character alpha numeric code. **Channel 1 is SA** for surface air, **Channel 2 is UU** for underwater up. Press ↓.

9. The LED will display **ave=** number. **Ave** is the number of seconds that the data logger will calculate a running average. Use the number keys to set the averaging time to 5 seconds. Press ↓.

10. Use the right or left arrow to se the Log Routine to **none**.

11. Press **Esc** twice to return to the setup menu.

12. Complete steps 3-8 for channel 2 entering the water multiplier in step 7 for the depth sensor.

13. Press the **View key** and use the left and right arrow to toggle to New Data. Press **ENTER**.

14. Use the left and right arrow key to toggle the display until channel I1I is displayed. The LI-1400 is now configured to display the running average of the 5 previous seconds' instantaneous values of the quantum sensor and is now ready for use.

B. Enter multipliers/initial set up for LI-1000:

1. With the front panel face up and the display at the top, connect the light sensor into the BNC connector located on the top left of the LI-1000 unit and the underwater sensor into the BNC connector located on the top right of the LI-1000 unit.

2. Turn on the instrument by pressing the key labeled **FCT ON.**

3. Press the key labeled **CFG** on the data logger.

4. The LED will display **Mode is INST**. If the LED displays **LOG,** press the arrow key until the LED displays **INST** then press "Enter".

5. The LED will display **Ch1 is LIGHT**. If not, press the arrow key until the LED displays LIGHT and press "Enter".

6. The LED will display **range=A.** Using range=A, the data logger automatically sets the range to cover the widest input signal range with the best resolution. If not, press the arrow key until the LED displays **A**, then press "Enter".

7. The LED will display **mult = number**. The multiplier is the calibration coefficient for each sensor and is specified on the calibration documentation for each sensor. Enter the correct air multiplier number for the sensor that is attached to channel 1 (Deck sensor) using the number keys, and then press "Enter".

8. The LED will display **Label = SA. Label** is a two character alpha numeric code. **Channel 1 is SA** for surface air, **Channel 2 is UU** for underwater up. Press enter after each code to confirm.

9. The LED will display **ave= number**. **Ave** is the number of seconds that the data logger will calculate a running average. Use the number keys to set the averaging time to 5 seconds then press "Enter".

10. The LED will display **ch 2 is LIGHT**. Complete steps 1-8 for channel 2 entering the water multiplier in step 6 for the depth sensor.

11. The LED will display **ch 3 is off.** Press enter until the LED displays **1A** followed by a series of numbers. The data logger is now configured to display the running average of

the 5 previous seconds' instantaneous values of the quantum sensor and is now ready for use.

C. Data Collection:

1. Visually inspect Licor meter probes and connections. Check battery level and ensure probes are positioned properly on deck and subsurface mountings.

2. Connect the deck sensor cable to the BNC connection on the top left of the data logger.

3. Secure the small deck sensor in an unobstructed area on the vessel.

4. Run the end of the cable for the underwater sensor to the data logger and connect the end of the underwater sensor to the BNC connector on the top right of the data logger.

5. Attach sufficient weight to the underwater sensor frame such that the sensor remains upright as it is lowered to depth.

6. On the sunny side of the boat, lower underwater sensor to depth just below the surface ensuring that the probe will not rise out of the water with wave action (Note: the depth for this reading is recorded as 0.1 meter in WQM).

7. Turn the instrument ON.

8. Obtain profile. Report values in whole numbers except for the last depth of the profile, for this observation report values in tenths.

    a. At each depth, a light attenuation reading is obtained from the deck sensor as well as from the water sensor.

    b. Take initial readings with the deck sensor and just below the surface with water sensor.

    c. Take second water sensor reading at depth of 0.5 meters.

    d. Take successive water sensor readings at 0.5 meter increments.

    e. Continue the profile until the underwater sensor displays either a value of approximately 10 micro Einstiens or a depth value that is 20% of the surface depth value.

    f. Allow a minimum of 5 seconds between readings (to create a 5-second average value).

    g. If the Licor instrument is being used as a data logger, press the "enter" key with each reading.

9. When profile is complete, turn the instrument OFF.

10. Record data on Licor Attenuation Sheet and transmit to CBO at the end of the month, along with the other field documentation sheets.

4.7. Vacuum Filtering Method (In-Line Filtering)

Some samples may require filtration utilizing a vacuum pump and filtering system. Particulate or dissolved nitrogen, particulate or dissolved phosphorus, and chlorophyll a require vacuum filtration.

Filtering should occur as soon as possible after sample collection but no later than two hours to prevent the biological and chemical processes occurring in the sample from significantly changing the analytical results. If samples are not vacuum filtered immediately, they should be kept in the cooler away from light until processed.

Currently, regions possess a system consisting of 3-4 filtering stacks inter-connected by PVC pipes and connected by rubber tubing to a vacuum pump on one end and catch flasks at the other. Each filter stack has a bell (tower), frit (base) and a valve that enables the user to close off the vacuum to each individual filtering unit. Some regions have an unused stack used as a pressure release valve allowing the user to "bleed" off the excess pressure for easier removal of the filters from the base. The pump is connected to a 12-v battery power source.

Example of in-line filtering system setup connected to vacuum pump and catch container

4.7.1. Supplies Needed

A. Filter stacks and filters:

1. Particulate nitrogen/carbon (PNC) samples:

   a. Filtering stack: Pall Gelman 25 mm 200 ml filter funnel and base (or equivalent).

   b. Filters: muffled 25 mm diameter, 1.0 um pore-sized Gelman type A/E glass fiber filters (see section 2.5 for muffling instructions). Prepare one filter for each sample to be analyzed and at least one additional filter per sampling event to be submitted as a

dry filter. The dry filters are utilized by DCLS for the determination of background carbon on the filters.

2. Particulate phosphorus (PP) and Chlorophyll a samples:

   a. Filtering stack: Pall Gellman magnetic 300 ml filter funnel and base (or equivalent).

   b. Filters: 47 mm, 0.7 um pore-sized Whatman GF/F filters. One filter per PP sample to be collected and 1-3 filters per Chlorophyll a determination. 1-2 Dry filters must also be sent to the lab for a check for the possibility of background contamination.

B. Ancillary supplies:

1. 1-3 rinsing bottles filled with fresh DI water

2. Stainless steel forceps

3. Graduated cylinders – 200 ml, 100 ml and 50 ml graduated cylinders each of which are notched at the 200, 100 or 50 ml mark ensuring accurate delivery of 200 ml, 100 ml and 50 ml volumes of sample.
   a. To ensure accuracy of notches on graduated cylinders, fill an appropriate clean (acid washed) TD (to dispense) volumetric flask or pipette so the bottom of the meniscus (U shaped depression) rests on the line of the volumetric glassware and transfer the contents to the graduated cylinder.

   b. Pour the contents of the flask or pipette into the graduated cylinder.

   c. Notch the cylinder based on where the bottom of the meniscus rests on the cylinder.

4. One clean Petri dish for PNC and PP sample collected

5. One piece (usually 3 in^2) of heavy duty aluminum foil per chlorophyll sample. Having a few extra pieces on hand is helpful incase the foil tears.

6. One 250 ml plastic bottle per dissolved nutrients (NTNP) sample needed and an additional 250 ml bottle as needed to ensure adequate volumes of NTNP sample.

7. One sample tag and/or label per sample collected.

C. Pump and hose or other approved water collection apparatus

Preparation:

Preparation for the filtering process includes:

1. Muffling 25 mm diameter glass fiber filters utilized for PNC

2. Acid washing the towers, graduated cylinders and plastic sample bottles and any reused 250 ml plastic NTNP bottles used in the filtering process

3. Rinsing the forceps with DI water

4. Collecting all the necessary supplies needed to properly identify the samples and ensure the delivery of uncontaminated, dry filter samples to DCLS.

Filter Muffling:

PNC filters must be muffled prior to their use to burn off as much background carbon and nitrogen as possible. The filters can reabsorb carbon and nitrogen with the opening and closing of the desiccators. Do not use filters for PNC analysis if muffled over 30 days ago. Procedures for muffling filters is in Section 2.5

Cleaning Filtration Equipment:
Do not use detergent to clean filtering equipment or the sample bottles utilized for the collection of filtered samples (filtrate) as detergents can contain contaminants.

1. Take all towers, graduated cylinders and tweezers and put them in the lab for cleaning.

2. Observe all laboratory safety precautions as outlined in Chapter 7.

3. Disassemble filter towers and clean each piece individually.
   a. Rinse all towers with DI water. Use a clean brush to remove any sediment.

   b. Use 25 ml of 10% hydrochloric acid (HCl) solution to strip any contaminants.

   c. Rinse 3 times again with DI water. Check for sediment or other foreign material. If found, repeat cleaning steps 3a and 3b.

4. Graduated cylinders.
   a. Rinse and strip the graduated cylinders in the same manner as the towers using approximately 10% of the total volume of graduated cylinder of 10% HCl using a brush if needed to remove any remaining sediment.

5. After use in the field, water will be trapped in the filtering apparatus. Use the flexible tube from the water collection tank to suck up water from inside the towers. Disconnect the water collection tank, empty and rinse with DI water and reconnect.

6. Store the clean filtration equipment in a manner that prevents contamination (e.g. wrap or cover openings with aluminum foil or plastic).

7. Rinse all utensils with DI water 3 times.

a. Do not use HCl on metal utensils, as it will result in rusting.

b. Wipe off any foreign material with a Kim Wipe or bush and rinse three times with DI water.

8. Acid wash all plastic bottles and their caps that will be reused in the collection of filtered samples with 10% HCl and triple rinse with DI water prior to their use in the field. Triple rinse any new bottles that have been stored without their lids on with DI water. Air dry bottles and replace the caps to seal the bottles until their use.

   a. If reused bottles are observed to be significantly discolored or contain large number of scratches or nicks, discard and obtain new sample bottles.

D. Manifold Set-up

1. Connect the rubber tubing on the right side of the PVC manifold to outflow valve of the vacuum pump.

2. Connect the rubber tubing on the left side of the PVC manifold to the catch flasks.

3. Connect the catch flasks tubing to intake valve the vacuum pump.

4. Slide rubber stoppers onto the bases of PNC and PP towers.

5. Rinse all towers and bases thoroughly with DI water.

6. Secure bases into PVC manifold by firmly pressing stoppers into PVC openings leaving one opening available to “bleed” off excess pressure.

7. Place towers (funnels) onto bases.

8. Connect the vacuum power pump to the battery.

9. Close off all valves, turn on the pump and adjust the pressure to 10 psi. Turn off the pump – it is now ready for use.

4.7.2. Filtration of Samples

A. Sample Collection.

Using a pump and hose (or an appropriately cleaned sampling device), rinse an acid washed and DI rinsed container such as a 2 L HDPE bottle or ½ gallon container with sample water. Fill the sample container with the sample including a sufficient quantity of water to filter more than one sample should it become necessary.

B. Equipment rinse:

1. Thoroughly rinse the inside of filtration towers with DI water (stored in a high-density polyethylene container).

2. Turn the towers upside down and rinse the area that connects to the filter base as it will contact the filter and could result in contamination.

3. Rinse frits (base) with DI including the stems of the PP bases to prevent contamination to the filtrate in the 250 ml NTNP bottles.

4. Set up tower and base for filtering.

5. Ensure that there are water catch flasks connected between the manifold and the vacuum source. This will prevent water from damaging the vacuum pump.

6. Connect vacuum power pump to battery.

C. Filtering

1. Place filters on bases.

   a. Using clean forceps and gripping on the filter edges, transfer a 47-mm Whatman 0.7 um GF/F glass fiber filter onto the bases for PP/Chlorophyll filtration and a muffled 25-mm filter for PNC.
      i. Be sure that PNC filters are "muffled" prior to use and placed rough side down.
      ii. Place PP and chlorophyll filters on the filter tower in the same direction they come out of the box (rough side up)

   Note: Discard any filters if they are dropped or if the surface is scratched or holes observed.

   b. Replace each filtration tower on its base after applying the filter.

2. Place clean NTNP bottles under the PP bases:

   250 ml of filtered sample (filtrate) is required for the Dissolved nutrient analyses (NTNP sample). Depending on the turbidity of the sample, 100-250 ml is typically filtered through the PP filters. If less than 250 ml can filter through a single PP filter, place a second 250 ml bottle under another filter stack set up for a 47 mm filter use the filtered water in the second bottle to top off the sample.

3. Rinse graduated cylinders with sample:

Mix sample thoroughly by shaking or inverting the plastic sample container, then rinse graduated cylinders with sample water twice. Be sure the rinse water contacts the entire inner surface of the cylinder.

4. Transfer sample to towers:

Note: - The volume of sample filtered will depend on the turbidity of the sample. If the sample is highly turbid, thoroughly mix the sample water and start with a small volume and then add 50 ml increments while continuing to mix the sample water until you get to the required volume or if the sample barely passes through the filter or if the filter is well colored. If the towers are color coded with tape, towers with green tape are used for PP samples and the blue for PNC samples.

1. Use 300 ml for Chl-a filters.

The volume of sample filtered in combination with the color of the filter will determine how many filters should be utilized. In general, if you have filtered 300 ml to 1 L of sample and have green color on the filter, that filter will be sufficient. If you have filtered less than 300 ml of sample and have color other than green, you will need to filter one or two more filters. Be sure to filter the same volume in each of the successive filters (e.g. if the first filter processed 50 ml of sample and was brown in color, you will need to filter 2 more filters using 50 ml of sample each).

2. The usual volume filtered for PP is 250 ml (200 - 300 ml) and 100 ml (100 - 150 ml) are filtered for PNC.

A greater or lesser volume may be filtered depending on sample turbidity. In general, PNC volumes should measure 50% of the volume filtered for PP.

b. Turn pump on.

- Keep the vacuum below 12 psi of Hg (10 psi of Hg is preferable).

- Filtration must not exceed 10 minutes per filter. If it will take longer than 10 minutes to filter the selected sample volume, discard filter and remaining sample in bell, rinse the filtration apparatus and start again using a smaller sample volume.

c. Add $MgCO_3$ to the last 25 ml of the Chl-a sample for samples with pH less than 7.0 SU.
Note: If using the filtrate of the Chlorophyll sample for dissolved nutrients, be sure to remove the NTNP bottle from under the base prior to this step. Shake to re-suspend the $MgCO_3$ solution (ratio of 1 g $MgCO_3$ to 100 ml DI water) to the last 25 ml (approximately) of sample filtered in the filtration bell.

d. Close valves or turn off pump to remove filtration vacuum.
Close valves just prior to filters being completely dry but dry enough to ensure none of the filtered material will be lost when the filter is folded for storage.

If filter pads are not "colored", continue mixing, measuring and adding known volumes of sample water to each tower until the filters turn color.

e. Bleed excess pressure off.
Open valve to the PVC opening that does not have a filter stack on it.

f. Open the vacuum valves of the stacks slowly.
Close each valve after the sample filters through each tower.

g. Rinse forceps with DI water

h. Remove filters from base.

1. Fold the filter in half using forceps, being careful not to touch or disturb the material on the surface of the filter.

2. Place Chlorophyll filter pads on a square of aluminum foil. If multiple filter pads are used for chlorophyll analysis, they must be packaged as one sample. Either wrap all filter pads in one piece of foil or wrap all separate foil packages together with one piece of foil.

3. Place PP filters in Petri dishes and seal them (do not use aluminum foil as this may contaminate the filters). If using color coded tape, code PP dishes with green tape and PNC filters in Petri dishes sealed with blue tape.

i. Record the volume filtered.
Record the volume filtered on the sample tag (or label) and the WQM field sheet.
All volumes MUST be recorded on the sample tag (or label) and the EDT field sheet

j. Remove NTNP bottle from PP cylinder and cap tightly.
Note: The filtrate bottle should be filled to the bottom of the neck If there is not enough filtrate, more sample needs to be filtered. In such an event, the "used" filters must be removed, placed in a Petri dish (only the first filter will be put in the Petri dish and analyzed), a new filter must be placed on the tower, and additional sample (after mixing) must be filtered. If the filters are being clogged very quickly due to large amounts of solids in the sample, continue replacing the clogged filters and add sample water as necessary.

- Pour filtrate from the bottle to rinse the cap.

Note: There have been some problems with filling the filtrate bottles to the proper volume. Common causes for these problems are:

1. The old filter was not removed, water would not flow through, water in the tower and filter pad had to be thrown away and the process started again (filtrate was not contaminated and could be saved).

2. New filter was not placed in tower, so sample water just poured into filtrate bottle. Bottle was contaminated and filtrate had to be thrown away (used filter pad was already in Petri dish and could be used).

k. Label samples.
- Place a label on the foil square marked with the following information:

1. Station ID
2. Date (yy/mm/dd) and time (24 hour military) of collection
3. Depth of collection (in meters)
4. Unit code
5. Collector's initials
6. Group code (PNC, PP, NTNP, or FCHLR)
7. Container number
8. Volume of sample filtered (NOTE: When multiple filter pads are used, the volume recorded would be that used for each filter i.e. if 3 pads are used and 100 ml filtered through each pad, the filtered volume recorded should be 3 X 100 ml NOT 300 ml).

l. Place samples on ice.

1. All filters may be placed together in one Ziploc bag if properly labeled.

2. **Make sure no water from the cooler touches the Ziploc bags.**

3. Place the 250 ml NTNP bottle in cooler and pack with ice to a level just below the bottom of the cap.

m. Completely rinse the empty filtration apparatus for the next sample.
Rinse towers and bases three times with at least 30 ml per rinse of deionized water prior to seating a new filter for the next sample.

n. Empty all ½ gallon jugs and/or 2 L HDPE bottles.
Rinse all reusable sample containers with sample water prior to collecting the next sample.

A few things to remember:

1. Always shake sample containers thoroughly before pouring any aliquot. Leaving a small air gap in the sample container will help aid in mixing.

2. Be certain to rinse all filtering towers with DI water between samples. Three 30 ml rinses is the minimum requirement.

3. Double check each tag for completeness and clarity.

4. Rinse graduated cylinders with DI water and then with sample water before processing each sample.

5. Rinse 250-ml bottle with filtrate and rinse cap with sample before closing.

6. Periodically the wastewater collection tank on the filtering units will require dumping. Make sure wastewater does not get into the overflow tank.

7. Make sure all paperwork is complete and matches the sample tags exactly.

4.8. Chlorophyll a Collection Using Syringe Filtration

Field filtration for Chlorophyll a can be done using a syringe and filter housing. Use of the syringe to collect Chlorophyll a samples is ideal when needing only a few samples or the vacuum apparatus would not be appropriate.

The use of the syringe method requires the filtering of approximately 300 ml of site water (or sufficient volume to produce a visible green residue on the filter) through a 150cc polypropylene syringe as follows:

1. Open the filter holder and remove the "O-ring". Using clean forceps, place a filter on the holder with a GF/F filter. Replace the O-ring, rinse the filter with at least 30 ml of DI water, close the filter holder and set aside.

2. Rinse the syringe by drawing a small amount (20 to 50 ml) of sample water up into the syringe and shaking it then discard the rinse water. The best way to do this would be to have the plunger situated at about the 100 ml mark and then draw up the rinse water to ensure rinsing the entire syringe interior. Expel all rinse water and air by fully depressing the syringe plunger.

3. Fill the syringe past the 150cc mark (150cc mark is the middle of "Y" on the syringe). Holding the syringe upward, tap on the side to eliminate as many air bubbles as possible and depress the plunger until the first ridge of the plunger aligns with the middle of "Y" on the syringe.

4. Screw the syringe into the filter holder and apply gentle pressure on the plunger. The goal is filter 300 ml of sample or until the filter paper clogs and there is green color on the filter. If at any time, you feel back pressure from the filter, the filter is clogged so stop the filtration and record the volume filtered on the filed data sheet.

    a. To refill the syringe, carefully detach the filter assembly, fill the syringe past the 150cc mark, remove any air bubbles, push the plunger to 150cc mark and continue with the filtration until the desired volume has been processed or until no water will pass through the filter with gentle pressure.

    b. If at any time the filter becomes clogged due to suspended solids, stop the filtration and <u>record the volume filtered on the field data sheet.</u>

5. If sample water has a pH less than 7.0 SU, detach the syringe from the filter assembly and pull the plunger back an inch or so. Shake the Magnesium Carbonate bottle, add 1 ml of saturated $MgCO_3$ solution (10mg/L) to the syringe, reattach the filter holder and apply

gentle pressure to force the remaining water in the filter holder and $MgCO_3$ onto the filter.

6. Record the final volume of the sample water filtered on the field data sheet.

7. Remove the filter holder from the syringe and open the holder. Using forceps, carefully remove the filter from its holder and gently fold it in half so the pigment is inside. Should the filter tear during the removal process, discard the filter and start over again.

8. Place the filter in the center of a piece of 3 by 3 inch aluminum foil, gently fold the aluminum foil into quarters and attach the sample tag with the sample tag wire or a staple or seal the foil using the adhesive label.

9. Place the aluminum foil in a Ziploc bag and store the bag in the cooler on top of the wet ice. Make sure the opening of the bag hangs out of the cooler when the lid is lowered.

10. Rinse the filter holder apparatus thoroughly twice using 20 to 50 ml D.I. water to clean the syringe for the next sample.

11. Enter the volume filtered into CEDS along with the other field data.

4.9. Sediment Sampling

4.9.1. Sampling Methodology

Deep water sediment samples are usually collected with a Petite Ponar (6”x6”) grab sampler. In shallow water, sediments may be hand-sampled using a scoop. Other program’s specific QAPPs may require other methods. If procedures outlined in this SOP differ from the SOP or QAPP of the program the sample is being collected for, the other document takes precedence.

4.9.2. Sampling Location and Substrate Selection

When sampling from a boat, samples must be collected away from the propeller wash whenever possible. When sampling in shallow streams, collect samples from the submerged streambed and not from the stream bank or from the floodplain. The most representative samples are from recently deposited sediments with ideal sediment containing fine particles with high organic content. Sediments with high organic content will appear dark brown or black.
Other program’s specific QAPPs may have other sample sitting requirements or other sediment type requirements. Again, follow the other program’s QAPP or SOP if the procedures differ from this document.

4.9.3. Collecting Sediment with a ‘Dredge’ Type Sampling Device

1. Collect any water chemistry and field probe samples prior to performing sediment sampling.

2. Put on powder free protective gloves before collecting the samples.

3. Prior to use, the dredge and all equipment which contacts with the sample must have been thoroughly cleaned and properly stored to prevent contamination as outlined in Chapter 2.2.2.

4. Rinse sediment dredge equipment with sample site water.

5. Open the jaws of the dredge and latch it so the dredge remains open.

6. Check the knot attaching the rope to the dredge to make sure it is secure.

7. Hold the dredge over the edge of the boat or bridge and lower it straight into the water. Once in the water lower at a rate of about one foot per second. This will ensure the dredge does not disturb surface sediments.

8. Once the dredge hits the sediment, give the rope some slack and then pull up on the rope to force the dredge to close.

9. Raise the dredge at a rate of about one foot per second to minimize the effect of turbulence that would disturb sediment trapped in the dredge.

10. A successful grab is one having relatively level, intact sediment over the entire area of the dredge and a sediment depth at the center of at least 3 centimeters. (Refer to Figure 1.) The sample is examined for suitability using the following criteria:

    a. Complete closure of the dredge jaws.

    b. No evidence of sediment washout through the dredge jaws.

    c. An even distribution of the sediment in the dredge.

    d. Minimum disturbance of the sediment surface.

11. Drain the overlying water from the dredge being careful to prevent loss of fine sediments. Reopen the jaws of the dredge, allowing the sediment to gently slump into a clean stainless steel pan. The stainless steel pan should not have rust or deep scratches.

12. Use a stainless steel scoop to remove the top 2 to 3 cm of the sediment and place it into a second clean stainless steel pan. Repeat steps 3 – 10 at least two more times or until collection of a sufficient volume of surface sediment. The container containing the composited sediment should remain covered with a stainless steel top or a sheet of aluminum foil during the collection of additional sediment until a sufficient volume of sediment is obtained.

13. Remove any undesired materials (e.g. shells, leaves, stones) using clean stainless steel forceps. The sediment should be thoroughly stirred with a clean stainless steel scoop until it

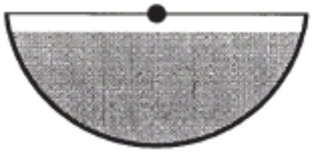
is uniform in texture, color and moisture. Fill the appropriate container leaving at least 1 inch of air space to prevent bursting of the sample container if frozen for long term storage.

14. Pour off any remaining water in the sample while avoiding losing fine sediment.

15. Cap and label the container and place in a cooler containing wet ice.

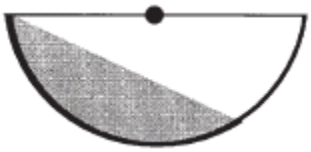
Illustrations of acceptable and unacceptable grab samples are provided in Figure 1.
Figure 1- Acceptable and Unacceptable Dredge Samples

Acceptable Grab:



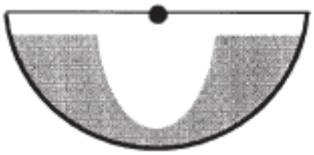
At least 3 cm of sediment with an even surface

Unacceptable Grab:



Grabs with sloping surface

Grabs with insufficient volume



Grabs with sediment washed-out

Grabs overfilled with sediment

Note: The condition of sediment at each site is variable and may result in difficulty obtaining an ideal grab sample. If, after a couple of attempts, you are unable to obtain the ideal grab, use your professional judgment to obtain a suitable grab sample. Note the condition of the sample on the field sheet if it is not of the ideal condition described above.

4.9.4. Collecting Sediment with the 'Scoop and Pan' Method

Prior to use, all equipment which contacts with the sample must have been thoroughly cleaned and properly stored to prevent contamination. See Chapter 2.2.2 on cleaning sediment sampling equipment. Rinse all sediment sampling equipment with site water prior to sampling.

1. At the site, the sampler generally must wade into the waterbody to obtain a scooped sample. The sampler should approach the sample site from the downstream direction and walk upstream, against the current. Allow any sediment or debris disturbed from wading to move downstream prior to sampling.

2. Do not disturb the sediment at the sampling location prior to collecting the sample.

3. The sample should be scooped in an upstream direction, against the flow. Slow movement when scooping the sample will prevent excess sediment from washing from the scoop resulting in an invalid sample.

4. Scoop a sample from the top 2-3 centimeters of the sediment. Transfer the sediment into a pre-cleaned stainless steel compositing tray. After the suspended sediment settles, siphon off as much water as possible. Collect three to five scoops of sediment, preferably of approximate equal volumes from different areas of the sample site for compositing. If sufficient sample volume was not collected with the initial scoops, collect an additional three to five scoops of sediment until the required amount of sediment is obtained.

5. Remove any undesired materials (e.g. shells, leaves, stones) using clean stainless steel forceps. The sediment should be thoroughly stirred with a clean stainless steel scoop until it is uniform in texture, color and moisture. Fill the appropriate container leaving at least 1 inch of air space to prevent bursting of the sample container if frozen for long term storage.

6. Pour off any remaining water in the sample while avoiding losing fine sediment.

7. Cap and label the container and place in a cooler containing wet ice.

4.10. Pollution Response Program Sampling Procedures (PReP)

4.10.1. Cyanide

To minimize loss of cyanide due to vaporization of CN-, preserve samples to pH 12 or more with 12N NaOH after testing for interferences or oxidizers. The sample pH must be 12 or more to hold HCN in solution and ensure a representative sample.

4.10.1.1. Total cyanide

Generally, if residual chlorine is present in the sample, sulfide will not be present. If sulfide is present, residual chlorine will not be present.

1. If the sample contains residual chlorine, add 0.6 g. ascorbic acid per liter of sample volume.

2. Samples containing sulfide have a maximum holding time of 24 hours. To extend the holding time requirement, samples may be tested with lead acetate paper pre-moistened with acetic acid solution before pH adjustments in order to determine if sulfide is present.

    a. If sulfide is present, add cadmium nitrate powder until a negative lead acetate paper spot test is obtained. Once neutralized, filter the sample using filter paper.

3. Add enough sodium hydroxide (NaOH) pellets to raise the pH to 12. Either add 2 ml of 10 N NaOH or adding a premeasured amount of 1 N NaOH (40 g NaOH to 1.0 L of DI water) to the sample - then test the sample with pH litmus paper and continue to add NaOH drop by drop until a pH of 12 is achieved.

4. Immediately after collection and pH adjustment, place sample bottles in a cooler filled with wet ice.

4.10.2. Sulfide

1. Collect sample with minimum aeration in a one quart cubitainer.

2. Fill bottle completely to remove any air gaps, cap container and place in an ice chest.

4.10.3. Pesticides and Herbicides

1. If sampling from an open body of water, fill the sample container with water from a representative area. Sampling equipment, including automatic samplers, must be free of plastic tubing, gaskets, and other parts that may leach interfering analytes into the water sample. Automatic samplers that composite samples over time should use refrigerated glass sample containers if possible.

2. Samples to be analyzed for herbicides/pesticides must have a pH between 5.00 and 9.00 SU. DCLS can adjust pH of samples upon receipt by the lab. If samples are tested within 72 hours from collection, DCLS may not need to adjust pH.

a. If the sampler wishes DCLS to check the pH of the sample, a note should be made on the sample tag for the lab to check the pH.

3. For the analysis of aldrin, remove any residual chlorine if present by adding 3.2 mg sodium thiosulfate to the water sample in the sample bottle.

4. Immediately after collection and any addition of sodium thiosulfate, place sample bottles in a cooler filled with wet ice.

4.10.4. Purgeable Organic Compounds (Volatiles)

1. If the sample is expected to contain residual chlorine, add 25 mg ascorbic acid preservative to the empty sample vial just prior to transport to the sampling site or at the sampling site.

   a. If more than 5 mg/L residual chlorine is suspected, perform a chlorine residual test. Add an additional 25 mg ascorbic acid for every 5 mg/L residual chlorine

2. Rinse and collect sample water using a clean 500 ml stainless steel beaker.

3. Fill two 40 ml sample vials to a positive meniscus in such a manner that no air bubbles passes through the sample when filling the vial. Do not over fill the vial.

4. Seal the vial so that no air bubbles are inside. Turn the vial upside down to check for air bubbles. Place vials in Styrofoam packing to prevent breakage.

5. Immediately after collection, place Styrofoam containers holding the sample vials in a cooler filled with wet ice.

6. Under certain conditions, biological activity will break down some aromatic compounds, notably benzene, toluene, and ethyl benzene. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, fill a separate vial and duplicate vial containing 6N HCL acid preservative.

7. A traveling blank (trip blank) must accompany all volatile organic samples. The trip blank should be prepared identically as the sample will be prepared. For example, if 6N HCL is added to the sample, then acidify the blank. Add 25 mg ascorbic acid if chlorine residual is expected to be present, etc. Fill a 40 ml glass vial with Teflon lined septum (acidified if sample is acidified) with DI water at the office. Place vial where other sampling vials will be kept throughout the whole sampling process, i.e., it travels with the sampling vial

4.10.5. Volatile Aromatic Hydrocarbons Including BTEX in Water

Note: Volatile Aromatic Hydrocarbons including Benzene, Toluene, Ethyl Benzene and Xylene (BTEX) is the appropriate analysis for gasoline.

1. Collect sample in a clean 500 ml stainless steel beaker. Acidify sample with 6 N HCl acid to a pH ≤2. If sample contains residual chlorine, add 25 mg ascorbic acid to the 40 ml glass vial prior to adding sample.
    a. If more than 5 mg/L residual chlorine is suspected, perform a chlorine residual test. Add an additional 25 mg ascorbic acid for every 5 mg/L residual chlorine

2. Fill two vials slowly with acidified sample to a positive meniscus. Tightly secure cap. Invert to check for air bubbles. Do NOT unscrew cap on vial once cap has been secured. Resample with a new vial if air bubbles are present and discard old sample.

4.10.6. Base/neutrals and Acid Extractables (Semivolatiles)

1. Check the pH of the sample. If pH value falls outside the range of 6.0-9.0, adjust pH accordingly.

2. If the sample contains residual chlorine, add 80mg sodium thiosulfate preservative to each of two, one liter amber glass bottles and mix well. This will provide a thiosulfate concentration of 0.008%

3. Immediately after collection, place sample bottles in a cooler filled with wet ice and kept away from light.

4.10.7. Petroleum Identification and Quantification in Water Samples

1. When collecting pure petroleum product from the surface of water, fill one 40 ml glass vial that has a Teflon lined cap. Exclude as much water as possible from vial. It is not essential to obtain air free sample (i.e. a positive meniscus). Tightly secure cap.

2. Immediately after collection, place sample bottles in a cooler filled with wet ice and kept away from light. Samples must be analyzed within 14 days after collection.

3. The analysis of pure product will provide petroleum identification only. No VOC or other analysis can be performed with this sample.

4.10.8. Total Petroleum Hydrocarbon (TPH) in Water Samples

- TPH analysis is appropriate for kerosene, diesel, or heavier oils.

- TPH determinations are more appropriate for soils as follow-up regulatory monitoring or when the nature of petroleum is known (i.e. tank closure).

1. Collect water samples directly in sample containers whenever possible.

2. If residual chlorine is present, add 80 mg sodium thiosulfate to the 1/2 pint amber glass jar prior to adding sample.

3. Collect sample in one 1/2 pint amber glass jar with Teflon lined cap.

4. Immediately after collection, place sample bottles in a cooler filled with wet ice and kept away from light.

5. Refrigerate samples until analysis. It is ideal to analyze samples within 7 days of collection. However, Federal regulations allow storage up to 14 days.

4.10.9. Sampling when Petroleum Product is Unknown

When visual inspection of sample does not indicate noticeable pure product, but petroleum odors exist, or you are uncertain of either a noticeable product or odor, the following sample containers are needed: one 1/2 pint amber glass jar with Teflon lined cap and one 40 ml glass vial with Teflon lined septum.

1. Collect water samples directly in sample containers whenever possible. If the sample cannot be collected directly in the sample container, collect the sample using a clean 500 ml stainless steel beaker.

2. Acidify sample with 6 N HCl acid to a pH ≤ 2. Check pH with litmus paper. If the sample contains residual chlorine, add 80 mg sodium thiosulfate to 1/2 pint amber glass jar and 25 mg ascorbic acid to 40 ml vial prior to adding sample.

3. Fill jar and vial slowly with acidified sample so that no air bubbles pass through the sample. Fill vial to a positive meniscus and immediately cap containers. Do NOT remove cap once it is secured. Invert to check for air bubbles. If air bubbles are present, fill another vial and discard old sample and container.

4. Immediately after collection, place sample bottles in a cooler filled with wet ice and kept away from light.

5. Refrigerate samples until analysis. Analyze sample within 14 days after collection.

4.10.10. Toxicity Sampling

1. Rinse sample containers with sample water. Discard rinse water away from the sampling location.

2. Fill the sample containers. Do not add any preservatives to the container and remove all air from the container. If there is a need to add sodium thiosulfate to the sample due to the presence of chlorine, note this on the chain of custody form along with the amount of thiosulfate added.

3. Immediately after collection, place sample bottles in a cooler filled with wet ice and keep away from light.

4. Transport or overnight ship the sample to a contracted toxicity laboratory. All samples must be addressed to the contracted laboratory and should contain the statement: Deliver Immediately to Laboratory.

5. Toxicity samples must be run within 36 hours after collection. Exceeding this time limit voids the sample.

4.10.11. Total Petroleum Hydrocarbon and BETX Identification in Soil Samples

1. Collect a soil sample directly into a glass container for surface samples or use a clean stainless steel or Teflon scoop.

2. Fill a half-pint wide mouth glass jar with Teflon lined cap. Sample should be protected from light by using an amber jar or wrapping the jar with aluminum foil.

3. Immediately after collection, place sample bottles in a cooler filled with wet ice and kept away from light.

4. Samples should be refrigerated during storage and analyzed within 14 days.

4.10.12. Sample Packing and Shipping

1. Place all sample containers in the cooler upright. The neck of the sample containers should be above the level of water and ice.

2. Ship all samples to the lab as soon as practical (generally the same day as collected).

4.11. Collection of Trace Element Samples (Clean Metals)

Clean metals samples are designed to quantify levels of trace elements for diagnostic programs such as TMDL or PROBMON. Because the standards for these parameters are for the dissolved component, we are looking for trace amounts (parts per billion) a “clean hands/dirty hands” approach to sampling is required.

4.11.1. Scope

This Standard Operating Procedure is intended to be used by the Department’s Ambient Water Quality Monitoring Staff and Permit Inspection Staff for the collection of freshwaters, saltwaters, and wastewaters with subsequent analysis by the Division of Consolidated Laboratory Services (DCLS) for dissolved and or total trace elements.

4.11.2. Applicability

The freshwaters appropriate for collection include all surface water and groundwater with a specific conductivity of approximately 1000 umhos/cm or less. Appropriate wastewaters include treated effluents with specific conductivities less than 1000 umhos/cm). Saltwater, brackish water, and highly turbid wastewater such as landfill leachates, are also appropriate for this procedure and are collected identically as the freshwaters but require special laboratory preparation and analysis.

The protocols contained in this Standard Operating Procedure are applicable to the compounds listed in Table 4.1 Target Analytes (page 109).

NOTE: This SOP is intended for concentration ranges of toxic trace elements (toxic metals) generally below 200 ug/L. The 200 ug/L threshold should be applied cautiously as this is only a generalization of the effect of contamination. For example, because of well documented contamination problems with Copper and Zinc, if a final effluent has historically had copper or zinc reported in the 200 ug/L range use of this protocol may reveal that the actual concentrations are significantly lower. However if the historical numbers for cadmium, arsenic, or mercury have been greater than 200 ug/L, use of this protocol may not affect these concentrations.

For concentrations above approximately 200 ug/L, existing 40 CFR 136 procedures are adequate and contain the necessary Quality Controls (including the requirement to collect blanks) to make reliable measurements in the high ug/L range. The United States EPA Region III has prepared extensive guidance for existing and new data that falls into this higher range.[1], [2]

Table 4.1 lists the Method Detection Limits established for each parameter using the protocols specified in this guidance. Method Detection Limits (MDL) was measured using the procedure specified in 40 CFR 136, Appendix B.

4.11.3. Summary

Ambient samples are collected in midstream by submerging a 4 liter plastic bottle referred as a Bridge Bottle (**see Figure 4.1 Bridge Bottle)**. Using a piece of flexible tubing connected to the bridge bottle and inline with a groundwater capsule filter, the sample is transferred by peristaltic pump from the bridge bottle into a plastic sample container**, see Figure 4.2 Loop Sample Container and Figure 4.3 Sample Container Schematics**. A provision is made where the bridge bottle is substituted for a sampling wand for collecting while wading, from a boat, or anytime the sampler can easily access the sampling zone , **see Figure 4.4 Ambient Sampling Apparatus**.

Effluent samples are collected directly into a sample container by submerging a Teflon tube at the sample site. Using a peristaltic pump, sample water is transferred though a piece of flexible tubing inlined with Teflon tubing though a capsule filter and into the sample bottle. **See Figure 4.5 Effluent Sampling Apparatus**.

It is recommended that two field technicians work with collecting trace metals.

4.11.4. Significance and Use

This method is primarily intended for the use in identifying and comparing dissolved trace metal concentrations to Virginia's Water Quality Standards. Water quality standards for dissolved metals are significant because the concentrations are significantly low (trace) and the criteria are expressed as the dissolved metal species and not as total recoverable.

This SOP should be used when trace metal concentrations are expected to be less than 1 mg/L range, typically less than 200 ug/L. This will prevent contamination from occurring resulting in biased results.

Contamination can occur from three main sources:

1. Improperly cleaned sample bottles and sampling equipment,
2. Improper handling sampling equipment,
3. Atmospheric debris and dust.

Bridge bottles and related equipment is prepared and tested for quality control at DCLS. Proper training on the sample collection protocols minimizes contamination introduced by improper technique. The design of the bridge bottle and sample bottle minimizes the risk of exposing the sample to dust and debris.

4.11.5. Equipment Preparation and WQM Scheduling of Sample Kits

4.11.5.1. Regional Field Equipment Preparation

The field equipment needed to collect trace metals should be stored in a plastic container to prevent dust contamination. Ideally, equipment needed for sampling one station should be stored in a clean plastic bag to allow ease of transport to the field and prevent contamination during transport.

Prior to0 sampling, the peristaltic pump batteries should be charged using the cigarette adapter and charger accompanying the pump. No other battery chargers should be used as the battery system is matched to the charger. A charged battery will work continuously for about 7 hours.

Run through your checklist to ensure that you have adequate supplies to collect the scheduled samples. Gloves are the main item you should always have in excess.

4.11.5.2. Ordering Kits

It is appropriate to maintain an adequate supply of clean metals sampling tubing and containers.

Note that all quality control blanks are handled as separate samples and one blank should be ordered for each ambient and effluent sample.

Ambient sampling sites should be established as stations in CEDS prior to sample collection and then processed using the CEDS system.

Prior to sample collection, the sample containers must be ordered directly from the laboratory and the samples must be scheduled through CEDS.

ORDER SAMPLE CONTAINERS from DCLS by e-mailing Norma Roadcap (Norma.Roadcap@dgs.virginia.gov) and Cindy Johnson (Cynthia.Johnson@deq.virginia.gov) with the number and type (by group codes) of samples you wish to collect, when they will be collected, and your region. PLEASE ALLOW 6 WEEKS FOR DELIVERY.

Please refer to Table 2 Parameter Group Codes for the parameter codes to request for containers based on the sample matrix type.

Freshwater samples include the following supplies:

1. One bridge bottle,
2. One tubing kit,

3. Two loop sample containers,
4. and two 100ml Mercury bottles.

Saltwater samples include the following supplies:

1. One tubing kit,
2. Two loop sample containers, and
3. Two 100ml Mercury bottles.

Effluent samples include the following supplies:

1. One tubing kit,
2. Two loop sample containers, and
3. Two 100ml Mercury bottles.

It is appropriate to store additional sample containers and kits for those situations that require rapid sample collection. Do not store sample containers longer than six months. Using a FIFO (First In, First Out) inventory system will ensure containers will not exceed this storage time.

4.11.5.3. Monthly Run Schedule

The vast majority of routine metals sampling collected by the VADEQ are for dissolved metals. Only when you have a special study should you collect total metals in addition to dissolved metals. Sampling only for total metals is not an efficient use of time and resources as it provides limited information.

1. Schedule samples with DCLS through the WQM system using the group codes in Table 4.2 Parameter Group Codes on page 109.

2. Refer to Figure 4.6 WQM Monthly Run Schedule Parameters (page 112). Refer to the Run ID that corresponds to your type of sample whether it is an effluent (EFF), freshwater (FRESH), or saltwater (SALT). Field equipment blanks (EB) do not have a separate group code. It is extremely important that you properly identify the equipment blanks in the **Blank/Dup** field. For the TCMET1 group code there is no EB field. This is because the EB for DCMET1 covers both group codes. However if only TCMET1 is being collected, then you will need to enter two TCMET1 entries. One for the sample noted as R, and the other for the equipment blank noted as EB.

   Note: Because of the time and effort needed to collect metals, usually a maximum of four or five sites can be sampled per day.

4.11.6. Equipment and Supplies

4.11.6.1. Items Which Should Be Stored in Equipment Box

The supplies are those which are needed when sampling for metals and which should protected from dust are listed in Table 4.3 Equipment on page 109.

4.11.6.2. Ancillary Items

Other items which may be needed include those listed in Table 4.4 Ancillary Supplies on page 110.

Batteries need to be charged overnight. Prior to each sampling run, check to make sure that you have enough supplies and that your portable battery is charged and functioning. The leads and fuse system on the batteries are delicate and prone to breaks and shorts. Every six months, completely discharge batteries and recharge to extend battery life.

4.11.6.3. Sampling Apparatus, Bottles and Containers

DCLS will supply all the necessary SAMPLE CONTAINERS, BRIDGE BOTTLES, and TUBING KITS based on the number and types of samples ordered through WQM.

- When placing orders for samples, try to group four to five sites that field teams can sample on the same day. DCLS will send out coolers with kits and bottles batched for the number of samples scheduled. You may use this same cooler to return the samples to DCLS for analysis.

- The tubing kits including the filters, bridge bottles, and mercury bottles are disposable/recyclable and should be discarded after each use.

4.11.7. Collection Protocol for Freshwater and Saltwater Using the Bridge Bottle

4.11.7.1. Equipment Setup

1. Identify an area where sample processing will occur. This should be an area free of falling debris and swirling dust and is on a flat, smooth surface protected from the wind. The tailgate of an enclosed truck bed or SUV is a good location.

2. Place the equipment box and coolers containing the sample containers and kits in the area where sample processing will occur.

3. Cover the work area with a large piece of plastic sheeting. Set out the pump and connect the battery. Switch the pump on to check that it is working. Dial the pump speed to 5 and turn off the pump until needed.

4. Remove a tubing kit, two loop sample containers, two mercury bottles and a bridge bottle from the plastic bag in the cooler and place on the plastic near the pump.

5. Remove a pack of powder free, vinyl gloves from the storage container and place on the plastic.

6. Remove the plastic sample caddy from the storage box and place it on the sample processing area near the pump. Secure the sample bottles in the caddy.

4.11.7.2. Bridge Bottle Filling

1. Locate the sample weights for connection to the bridge bottle.

2. Locate the polypropylene sampling rope spool, cut a sufficient length of rope to allow for deployment.

3. Don one or two pairs of vinyl gloves using clean precautions. Only touch the areas of gloves with bare hands which will not contact with sampling equipment.

4. Tie one end of the sampling rope to the five pound weight leaving approximately one foot at the end to connect to the bridge bottle.

5. Untie or open by tearing the top of the outer plastic bag containing the bridge bottle.

6. Reach into the outer bag and untie or tear the inner bag near the handle connection. Check the configuration of the tubing to ensure that proper filling will occur. Inspect the smaller vent tubing and adjust if it appears crimped due to storage. While the bottle is still in the inner bag, it is acceptable to remove the top fitting to check the inner sipper tube. Adjust all fittings appropriately.

7. When the fittings have been properly secured and adjusted, remove the bridge bottle from the inner bag and lay on the plastic film. Tie the weighted end of the rope onto the handle of the bottle leaving about six inches of line between the bottle and the weight.

8. Proceed to the sampling location with the bridge bottle apparatus. Carry several extra pairs of gloves to the site to facilitate bridge bottle handling.

9. When deploying from bridges with moderate to low stream velocities collect the sample upstream of the bridge by lowering the assembly into the water. Ensure that the assembly does not contact any structures or other objects as it is lowered into the water.

10. Once in the water the weight will partially submerge the BRIDGE BOTTLE, which will begin to fill. Check to insure the air release tube is above the water level and not obstructed. When the bottle is first submerged, a good indication it is filling properly is a small slug of water may be expelled from the air vent tube. If the bottle is properly adjusted, it will fill within a few minutes.

- Problems with filling from bridges can occur when stream velocities are high. Sampling on the downstream side of bridges is acceptable to avoid the risk of losing the assembly due to the current sweeping it under a bridge or other obstruction. When stream velocities are high, adding 7.5 pounds of weight will aid in sample collection. The added weight will cause the container to sink lower when partially filled which may submerge the vent tube. The vent tube can be extended past the bottom of the bottle to prevent filling with water when the weight is heavy or the water is rough.

- Other problems with filling can occur when the inlet tube is clogged, the vent tube contains a slug of water or other obstruction, the vent tube is below the surface of the water, the weight is not positioned close enough to the bottle, or the vent tube or inlet tube has become disconnected from the bottle.

11. When the BRIDGE BOTTLE is approximately 1/2 to 2/3 full retrieve the bottle and return to the sample processing area. It is acceptable to allow the bridge bottle to sink completely below the surface as long as the inlet tube does not contact the bottom. Ensure that the assembly does not contact any structures or other objects as it is retrieved.

12. When deploying while wading or from a small craft, the bridge bottle can be submerged by hand without using weights.

13. When the water level at the sample site is very shallow it may be difficult to submerge the bridge bottle deep enough to begin siphoning. The alternative is to use the effluent sample configuration (on page 112) where the stream sample is pumped directly into the loop sample container. This will require bringing a pump assembly to the sample site. This is best accomplished by attaching the pump assembly to a backpack.

14. Once the bridge bottle has been brought back to the sample processing area, set it next to the pump and remove the weight. With the inlet and vent tubing properly configured, the bridge bottle can remain on the plastic outside of a bag without any danger of atmospheric contamination.

4.11.7.3. Dissolved Grab Sample Blank Procedure

Refer to Figure 4.4 Ambient Sampling Apparatus on page 112 for the schematic of the field sampling equipment used to process blanks and samples.

Determine which tech will be **clean hands** and which will be **dirty hands**.

1. **Dirty hands** and **clean hands** don one or two pairs of vinyl gloves. Only touch the areas of gloves with bare hands which will not contact sampling equipment.

2. **Dirty hands** opens the sample bottle outer plastic bag.
   Clean hands opens the inner plastic bag. At this point, there should be two loop bottles and two mercury bottles accessible to **clean hands**.

3. **Dirty hands** opens the grab kit's outer plastic bag.
   Clean hands opens the inner plastic bag and removes the tubing assembly.

4. **Clean hands** disconnects one side of the sample loop on the first sample container and connects the end of the tubing kit opposite the filter to the opened sample container. Remember the sample container is full of clean water from the lab.

5. **Dirty hands** connects the peristaltic tubing at approximately the mid-point of the length to the field pump.
   Clean hands inverts the sample container.
   Dirty hands switches on the pump.

6. Process the entire contents, 1000ml, of the sample container through the tubing and filter apparatus at a flow rate of 500ml/min (pump setting of 5). At the beginning of the sample processing, orient the filter cartridge with the flow arrow pointing up. This will insure proper wetting of the filter.

7. **Dirty hands** switches off the pump when the last continuous stream of water enters the filter. The filter must not be allowed to go dry. This is to prevent problems with excessive back pressure causing tubing separating from the filter.

8. **Clean hands** disconnects the pump tubing from the empty loop bottle and reconnects this same end to the second loop bottle containing ultrapure water provided by the lab for the sample blank and inverts the container.
   Dirty hands switches on the pump. Process the blank water from the loop bottle until approximately 125mls have been expelled from the filter. **Dirty hands** switches off the pump.

9. **Clean hands** opens the first mercury container and discards the water.
   Clean hands holds the outlet of the capsule filter just above the open mouth of the mercury bottle. Fill the mercury bottle to overflowing and cap. Make sure there are no air bubbles in the bottle that are larger than a pea.
   Clean hands then connects the capsule filter outlet to the empty loop container via the sample loop tubing. Process the remaining contents (~900 ml) of the sample container through the tubing and filter apparatus into the first sample container.
   Dirty hands switches off the pump before the filter is completely dry.

10. **Clean hands** disconnects the outlet tubing from the blank sample container and immediately reconnects the loop tubing on the top of the blank bottle.

    The field blanks collected in this manner is comprehensive blanks because they are collected in the same equipment as the sample and processed like the sample through all steps of the protocol. This is the most important check of contamination in the protocol.

4.11.7.4. Dissolved Grab Sample Procedure

1. **Clean hands** immediately (immediately means less than one minute) disconnects the vent tubing from the bridge bottle containing sample water and then connects the inlet side of the pump tubing in place of the vent tubing.

2. **Dirty hands** switches on the pump. Process the sample water from the bridge bottle until approximately 125mls have been expelled from the filter. **Dirty hands** switches off the pump.

3. **Clean hands** opens the second mercury container and discards the water. **Clean hands** holds the outlet of the capsule filter just above the open mouth of the mercury bottle. Fill the mercury bottle to overflowing and cap. Make sure there are no air bubbles in the bottle that are larger than a pea.

4. **Clean hands** unscrews the cap of the second loop sample container and discards the small amount of water remaining in the container.
   Clean hands returns the top to the container and then connects the capsule filter outlet to the second empty loop container via the sample loop tubing. Process the sample into the loop container until the container is full. It is acceptable to fill the sample container to overflowing, however avoid filtering more than 1000 ml through the filter.
   Dirty hands switches off the pump.

5. **Clean hands** disconnects the outlet tubing from the sample container and immediately reconnects the loop tubing back in place to seal the sample bottle.

6. **Clean hands** holds the blank loop container in a manner to allow **dirty hands** to place the WQM label directly on the midsection of the bottle. The mercury container also has a WQM label placed on the midsection in the same manner as above.

7. **Clean hands** places the mercury blank container into the inner bag of the blank loop container and seals the inner bag.
   Dirty hands seals the outer bag.

8. Step 7 is repeated for the sample bottle and mercury sample.

9. The bags containing the blanks and samples are immediately placed on ice in a separate sample cooler containing only clean metal containers. This is to prevent wire sample tags used on other sample containers from contaminating the clean samples.

   Note: It is no longer necessary to record the ultra bottle numbers but it is important that the WQM labels are used to identify the samples and blanks. The container numbers chosen in WQM do not need to correspond to the ultra bottle numbers.

10. Rinse the rope and weights with ambient water to remove any visible dirt, place inside a plastic bag, and store in the storage container. Rope may be reused several times if rinsed frequently.[RES1]

4.11.7.5. Total Recoverable Grab Sample Procedure

If total recoverable samples are to be collected in conjunction with dissolved samples, the BRIDGE BOTTLE must be shaken during sample processing to ensure proper mixing of suspended solids. Additionally during the total recoverable sample collection, the BRIDGE BOTTLE must be shaken during sample processing.

When collecting for total recoverable samples after first collecting for dissolved samples protect the tubing used to collect the dissolved fractions after the last dissolved samples are collected.

1. **Clean hands** removes the capsule filter from the tubing and opens the third mercury container and the third total recoverable loop bottle and discards the water. The loop bottle cap should be replaced immediately after discarding the water.
   Dirty hands switches on the pump

Clean hands holds the outlet of the capsule filter just above the open mouth of the mercury bottle. Fill the mercury bottle to overflowing and cap. Make sure there are no air bubbles in the bottle that are larger than a pea.

2. **Clean hands** connects the tubing to the total recoverable loop container and fills the container until full.
   Clean hands immediately reconnects the loop tubing to seal the container.

3. **Clean hands** holds the total recoverable loop container in a manner to allow **dirty hands** to place the WQM label directly on the midsection of the bottle. The mercury container also has a WQM label placed on the midsection in the same manner as above.

4. **Clean hands** places the mercury total recoverable container into the inner bag of the total recoverable loop container and seals the inner bag.
   Dirty hands seals the outer bag.

5. The blanks and samples should be immediately placed on ice in a separate sample cooler containing only clean metal containers. This is to prevent wire sample tags used on other sample containers from contaminating the clean samples.

4.11.7.6. Other Parameters

1. The clean protocol is complete at this step and field parameters can now be taken from the remaining water in the BRIDGE BOTTLE. Recommended field parameters include: pH, conductivity, temperature, and dissolved oxygen. Additional recommended laboratory samples include the solid series and total organic carbon should be collected and are identified by the group codes: SOLID and TOC.

2. Rinse the rope and weights with ambient water to remove any visible dirt, place inside a plastic bag, and store in the storage container. Rope may be reused several times if rinsed frequently.[RES2]

4.11.8. Effluent Sample Collection Protocol

4.11.8.1. Equipment Setup

1. Locate an area near the final effluent sampling location where sample processing will occur. This should be an area free of falling debris and swirling dust, is flat and smooth, and protected from the wind. The tailgate of an enclosed truck bed or SUV is suitable.

2. Locate the equipment box and coolers containing the sample containers and kits in the area where sample processing will occur.

3. Cover the work area with plastic sheeting. Set out the pump and connect the battery. Switch pump on for a quick burst to check that it is working. Dial the pump speed to 5.

4. Remove a tubing kit and two sample containers from the cooler and place on the plastic near the pump.

5. Remove a pack of powder free vinyl gloves from the storage container and place on the plastic. Refer to figure 4.5, for the schematic of the field sampling equipment used to collect effluent grab samples.

6. Remove the plastic sample caddy from the storage box and place it on the sample processing area near the pump.

7. Locate the sample wand used for positioning the Teflon sample tubing in the effluent.

4.11.8.2. Dissolved and/or Total Recoverable Grab Blanks and Samples

1. Refer to Figure 4.5 Effluent Sampling Apparatus on page 112 for the schematic of the field sampling equipment used to process blanks and samples.

2. The effluent grab blanks and samples are collected in exactly the same manner as the ambient grab blanks and samples. Please refer to pages 98-101.

3. Instead of collecting from a bridge bottle, use a PVC sample wand equipped with a special notch to hold the Teflon tubing.

4. **Clean hands** presents a section of the Teflon tubing just past the inlet to **dirty hands** who then attaches the tubing to the sample wand.

5. The entire assembly: sample caddy containing the empty sample container, sample tubing, pump/battery, and sample wand are transported to the effluent sampling location.

6. **Dirty hands** places the sample wand into the collection zone taking precaution not to touch the tip of the sampling tube on any items. Take every precaution to ensure that the tip of sampling tube does not contact sediment or debris.

7. At this point, refer to section 4.10.7.4 on how to process samples. The procedure is complete once you have reached step 9.

4.11.9. Sample labeling

When the sample run is scheduled, the labels should be printed from the CEDS. Make sure to use the label has good quality glue on it (i.e. Avery). Unless specified otherwise, labels shall be filled out with date and time and affixed to bottles prior to bottles getting wet. Do not stick the label outside the plastic bag, as this will result in an unlabeled sample bottle.

4.11.10. Sample Shipping

4.11.10.1. Supplies and Materials

1. An insulated shipping cooler with polyethylene lid. A 28 quart cooler is usually sufficient to hold sample bottles, wet ice packs, and protective material while still being a convenient size and shape for handling, stacking, and storing.

2. Liner Bags which are 30 gallon plastic trash bags with a dimension of 30”x36”x1 mil.

3. Non-DCLS laboratories may request wrapping the 1 L sample bottles with plastic bubble wrap. If so, use packing material with 1/2” bubbles in 12”x16” sheets used for wrapping the 1 liter sample containers. The material is available in various configurations with the 1’ wide roll suitable for the loop containers.

4. Rubber bands size 33 or large enough to secure the bubble wrap around 1 liter containers packed in two Ziploc bags.

5. Wet ice cubes. Use of blue gel packs for most samples is prohibited due to not ensuring proper transport temperature.

6. Strapping tape, 1” filament type.

7. Duct tape, 2” utility type.

8. Sealing tape, 3” clear acrylic adhesive. Suitable for sealing ice bags and protective labels on bottles and coolers.

9. Packing list envelopes, clear plastic self adhesive type. Overnight services are a good source.

10. Address Labels, specific to the carrier.

4.11.10.2. Sample Packaging

1. Immediately following sample collection, place sample bottles in storage cooler with wet ice for transport back to the regional office for packing into shipping coolers.

2. Insert two trash bags into the cooler for double lining.

3. Fill the cooler with sufficient ice to submerge the sample bottles to the shoulder of the bottle.

4. Place the chilled sample containers upright into the lined cooler and surround with ice. The sample containers and ice should be tightly packed. When the cooler is properly packed there will be no extra space left in the cooler.

5. Seal each liner bag by twisting the top of the bag and tying in a knot.

6. If appropriate, attach a 1 gallon Ziploc bag that will hold the packing list envelope to the underside of the shipping cooler lid. Insert the appropriate sample documentation (e.g. chain of custody form, field data sheets, or special lab instructions) and seal the envelope. Place the sealed envelope in the Ziploc bag and seal bag for protection.

7. Close the lid, seal horizontal joints with duct tape, and secure with strapping tape.

8. Attach address label to side of cooler and protect with clear sealing tape.

4.11.10.3. Sample Transportation

For those samples shipped via standard DCLS courier service, no special precautions beyond normal shipping procedures are required. Sample packaging and transportation items listed below are provided for those samples that are to be shipped long distances (generally interstate) and are intended for worst case shipping conditions.

1. Samples shipped by common carrier must comply with applicable Department of Transportation Hazardous Materials Regulations, 40 CFR Part 172. The person offering such material for transportation is responsible for ensuring such compliance. See 40 CFR Part 136 Table II for guidance on applicability of preserved environmental samples.

2. Ship samples on the day of collection and use a reliable courier service for priority or next day delivery.

3. A large amount of effort is required to sample four to five sites and a large amount of work preparing the sample equipment and containers has taken place. Four samples represents over $1000.00 in equipment preparation and analytical costs so coordinate sample shipment closely with DCLS and continue follow-up communication until delivery is confirmed and condition of samples upon receipt is verified.

4.11.11. Quality Control

1. The protocols in this SOP are designed to include all the necessary Quality Control steps needed to produce reliable accurate data.

2. Table 4.5 Quality Control Recommendations for Trace Metals Sample Collection on page 112 lists the critical control points of the sampling protocol. These control points are the minimum steps required for the collection of samples. When field contamination is detected additional blanks and other quality control samples are necessary to identify and correct the problem.

3. Field equipment blanks (identified as EB with a depth of 0.0 in WQM) should be collected with every sample including the mercury blank. If total recoverable samples are also collected, the dissolved equipment blank will be representative of the total recoverable sample. If only total recoverable samples are collected, then a field equipment blank is required.

4. For effluent sites, blank samples must be collected prior to each sample and all trace metal samples.

5. If ambient site conditions indicate potential problems then it would be wise to collect additional samples. Some site conditions which would warrant blanks prior to sample collection are:

a. Road construction producing visible dust,
b. Any operation causing visible dust emissions,
c. High total suspended solids conditions instream,
d. Recent deicing of bridges,
e. High traffic volume on bridge and,
f. Heavy rain events during sampling.

At a frequency of greater than 10%, collect field duplicates. Field duplicates for ambient sample collection involve processing an additional blank and sample from the bridge bottle.

Field duplicates for effluent sample collection involve processing an additional blank and sample in series from the effluent. This may produce variable results due to temporal variability.

4.11.12. Clean Metals Quick Reference Guide

Ambient Sample Collection Quick Reference

1. Tie the 5 pound weight to the bridge bottle.
2. Collect the sample using the bridge bottle.
3. Untie the 5 pound weight.
4. Connect the tube to the first loop container.
5. Rinse the filter with the contents of the container.
6. Remove the tube from the empty bottle and place on the second loop container.
7. Pump 125mls of water to waste through the filter to purge previous sample.
8. Fill the mercury blank and seal.
9. Connect the filter to the empty first loop container.
10. Pump out of the second loop container into the first without letting the filter go dry.
11. Seal the blank loop container.
12. Remove the tube from the now empty second loop container and reconnect the tube to the bridge bottle vent tube.
13. Pump 125mls of water to waste through the filter to purge previous sample.
14. Collect the mercury sample.
15. Unscrew the lid of the second loop container and discard the water and replace the lid.
16. Connect the filter to the second loop container and fill.
17. Seal the sample containers.
18. Remove the filter and collect the total recoverables.
19. Collect field parameters.
20. Pack in ice and transport.

Effluent Sample Collection Quick Reference

1. Connect the tube to the first loop container.
2. Rinse the filter with the contents of the container.
3. Remove the tube from the empty bottle and place on the second loop container.
4. Pump 125mls of water to waste through the filter to purge previous sample.

5. Fill the mercury blank and seal.
6. Connect the filter to the empty first loop container.
7. Pump out of the second loop container into the first without letting the filter go dry.
8. Seal the blank loop container.
9. Remove the tube from the now empty second loop container and connect the tube to the sample wand.
10. Pump 125mls of water to waste through the filter to purge previous sample.
11. Collect the mercury sample.
12. Unscrew the lid of the second loop container and discard the water and replace the lid.
13. Connect the filter to the second loop container and fill.
14. Seal the sample containers.
15. Remove the filter and collect the total recoverables.
16. Collect field parameters.
17. Pack in ice and transport.

Ambient Sample Collection without the Bridge Bottle Quick Reference

1. Connect the tube to the first loop container.
2. Rinse the filter with the contents of the container.
3. Remove the tube from the empty bottle and place on the second loop container.
4. Pump 125mls of water to waste through the filter to purge previous sample.
5. Fill the mercury blank and seal.
6. Connect the filter to the empty first loop container.
7. Pump out of the second loop container into the first without letting the filter go dry.
8. Seal the blank loop container.
9. Remove the tube from the now empty second loop container and connect the tube to the sample wand.
10. Pump 125mls of water to waste through the filter to purge previous sample.
11. Collect the mercury sample.
12. Unscrew the lid of the second loop container and discard the water and replace the lid.
13. Connect the filter to the second loop container and fill.
14. Seal the sample containers.
15. Remove the filter and collect the total recoverables.
16. Collect field parameters.
17. Pack in ice and transport.

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[1] NPDES Self-Monitoring Data and Data Audit Inspections (DAIs), United States Environmental Protection Agency Region III, Central Regional Laboratory, Fall 1994.

[2] Metals' Data Position Paper, United States Environmental Protection Agency Region III, Rev 5.0, May 1, 1995.

[3] Tom Fieldsend of Sample Control Center, Operated by DynCorp Environmental for USEPAs Engineering and Analysis Division provided the protocol for sample shipping.

4.11.13. Referenced Documents

The methods and research articles used to develop the field sampling equipment are:

1. Benolt, Gaboury, Clean Technique Measurement of Pb, Ag, and Cd in Freshwater: A Redefinition of Metal Pollution, Environ. Sci. Technol., Vol. 28, No. 11, 1994.

2. Horowitz, A.J. et. al., The Effect of Membrane Filtration Artifacts on dissolved Trace Element Concentrations, Wat. Res. Vol. 26, No. 6, pp. 753-763, 1992.

3. Horowitz, Arthur J., et.al., On the Problems Associated with Using Filtration to Define Trace Element Concentrations in Natural Water Samples, U.S. Geological Survey.

4. Martin, Gary R., et.al.,), A Comparison of Surface-grab and Cross Sectionally Integrated Stream-water-quality Sampling Methods, Water Environment Research, Volume 64, 866 (1992.

5. Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels, EPA 821-R-95-034, April 1995.

6. Geological Survey Protocol for the Collection and Processing of Surface-Water Samples for the Subsequent Determination of Inorganic Constituents in Filtered Water. United States Geological Survey, Open-File Report 94-539.

These methods provide an excellent source of information on the proper handling of sampling equipment and the collection of samples for the analysis of trace metals. The techniques used to collect contamination free environmental samples are involved and require a significant understanding of the principles of trace analysis. It is beyond the scope of this SOP to provide all the details that would be necessary to allow an inexperienced field team to collect trace metal samples. These protocols should be used by persons experienced in the collection of environmental samples for trace analysis and who are familiar with the sources and magnitude of ambient contamination

Table 4.1: Target Analytes

Parameter	CAS Number	Method Detection Limits, ug/L					
		ICPMS USN Freshwater	ICPMS USN Saltwater	ICPMS USN TR Freshwater	ICPMS USN TR Saltwater	ICPMS	ICP AES USN
Aluminum	7429-90-5			0.04		0.37	
Antimony	7440-36-0	0.05		0.03		0.33	
Arsenic	7440-38-2	0.07		0.03		0.24	5.37
Barium	7440-39-3						
Beryllium	7440-41-7						
Cadmium	7440-43-9	0.05		0.04		1.72	2.37
Calcium	7440-70-2						0.08
Chromium	7440-47-3	0.02		0.04			2.27
Copper	7440-50-8	0.02		0.06		0.87	4.98
Iron	7439-89-6						2.30
Lead	7439-92-1	0.17		0.03		0.28	
Magnesium	7439-95-4						0.00
Manganese	7439-96-5	0.02		0.03		1.32	0.58
Mercury	7439-97-6					0.12	
Nickel	7440-02-0	0.04		0.02		0.39	1.71
Selenium	7782-49-2			0.06		0.77	
Silver	7440-22-4	0.19		0.03		0.15	
Thallium	7440-28-0						
Zinc	7440-66-6	0.26		0.03		2.18	1.95

ICPMS USN- Inductively Coupled Plasma Mass Spectrometry sample introduced by Ultrasonic Nebulization
ICPMS USN TR- Inductively Coupled Plasma Mass Spectrometry sample introduced by Ultrasonic Nebulization Total Recoverable
ICPMS- Inductively Coupled Plasma Mass Spectrometry
ICP AES USN- Inductively Coupled Plasma Atomic Emission Spectrometry sample introduction by Ultrasonic Nebulization

Table 4.2: Parameter Group Codes

SALTWATER	
DCMET1	Dissolved clean metals in saltwater
TCMET1	Total clean metals in saltwater
FRESHWATER	
DCMET	Dissolved clean metals in freshwater
TCMET	Total clean metals in freshwater

MERCURY ONLY	
DCHG	Dissolved mercury in freshwater
TCHG	Total mercury in freshwater
EFFLUENT	
CMEFF	Dissolved clean metals in effluents
TCMEFF	Total clean metals in effluents

The group codes include mercury and the method blank container. For mercury only, use code DCHG or TCHG.

Table 4.3: Equipment

	Item	Supplier	Catalog Number
1	Peristaltic pump unit	Cole-Parmer	H-07533-40
2	Quick release pump head	Cole-Parmer	H-07518-60
3	Cigarette lighter adapter cable	Cole-Parmer	H-07573-02
4	Portable battery pack	Cole-Parmer	H-03276-50
5	Powder free vinyl gloves	Fisher Scientific	11-387-3
6	Clear polyethylene drop cloth	Hardware store	4 to 6 mil
7	Preprinted laser jet waterproof labels	Avery	5163
8	Indelible markers	Office supply store	Sharpie
9	Bridge bottle	DCLS	N/A
10	Bridge bottle tubing kit	DCLS	N/A
11	Teflon tubing kit	DCLS	N/A
12	Sample bottles	DCLS	N/A
13	One gallon Ziploc bags	Grocery store	N/A
14	Two gallon Ziploc bags	Grocery store	N/A
15	Bridge bottle weights	Sporting goods store	N/A
16	White polypropylene line	Hardware Store	N/A

Table 4.4: Ancillary Supplies

	Item	Supplier	Catalog Number
1	Plastic bubble wrap	Consolidated Plastics	87600LG
2	Rubber bands	Office supply store	Large
3	Ice	RO ice machine	N/A
4	Duct tape	Hardware store	N/A
5	Knife or cutters	Hardware store	N/A
6	Fuses for pump and battery	Hardware store	N/A

Table 4.5: Quality Control Recommendations for Trace Metals Sample Collection

Sampling Requirements	Criteria	Frequency
Type of Method	Performance based by demonstration of no detectable contamination of target analytes or interferences in samples or blanks. Method 1669 and the sampling apparatus and techniques used by the DEQ are recommended for sample collection.	Demonstration of contamination free samples and blanks every time a variation is made to the method.
Media Type	Freshwater and treated final effluent wastewater for dissolved and total recoverable metals	N/A
Training	Sample collection by only thoroughly trained personnel. Personnel must demonstrate proficiency in collecting contaminant free blanks and samples	Train a minimum of one time prior to any sample collection. Stop and provide additional training if field QC demonstrates problems until the criteria is achieved
Filtration	0.45 um capsule filter with nominal surface area of 60.0 cm^2 Maximum sample volume 1000 ml through single use filter	Onsite at time of collection or within one hour for composite samples after the sample sequence is complete.
Sample Containers	No detectable target analytes above MDL	Minimum of 1% of containers checked by the laboratory per batch after initial demonstration of acceptable blank QC
Sampling Equipment	No detectable target analytes above MDL	Minimum of 1% of equipment checked by the laboratory per batch after initial demonstration of acceptable blank QC
Comprehensive Grab Field Blank	Blanks must be <10% sample concentration or if sample is < MDL, field blank contamination is OK.	Process one with every sample collected. When duplicate samples are collected, only one blank is necessary.
Comprehensive Composite Field Blank	Blanks must be <10% sample concentration or if sample is <MDL, field blank contamination is OK	Process one per site for every ten samples. When 10% frequency rule is applied, blanks are to be collected with the first sample. Process field blanks every time equipment is field cleaned to be reused between sites or sample events.
Field Duplicate	Statistically equivalent to the RPD of the matrix spike and matrix spike duplicates for quantifiable concentrations	Process one per site for every ten samples.
Preservation	Samples must be iced in the field. Composite samples must be iced during collection. Adjust pH <2 SU within 72 hours of collection and samples must remain in original containers for a minimum of 18 hours prior to digestion or analysis.	All samples must be acid preserved in the field or by the laboratory using ultra pure HNO_3 to pH <2 SU. Samples should be iced in field immediately after collecting.
Documentation	Sampling activities must be documented on paper or by computerized sample tracking.	Documentation must be done per sample, per site.

Figure 4.1: Bridge Bottle

Figure 4.2: Loop Sample Container and Mercury Container

Figure 4.3 Sample Container Schematics

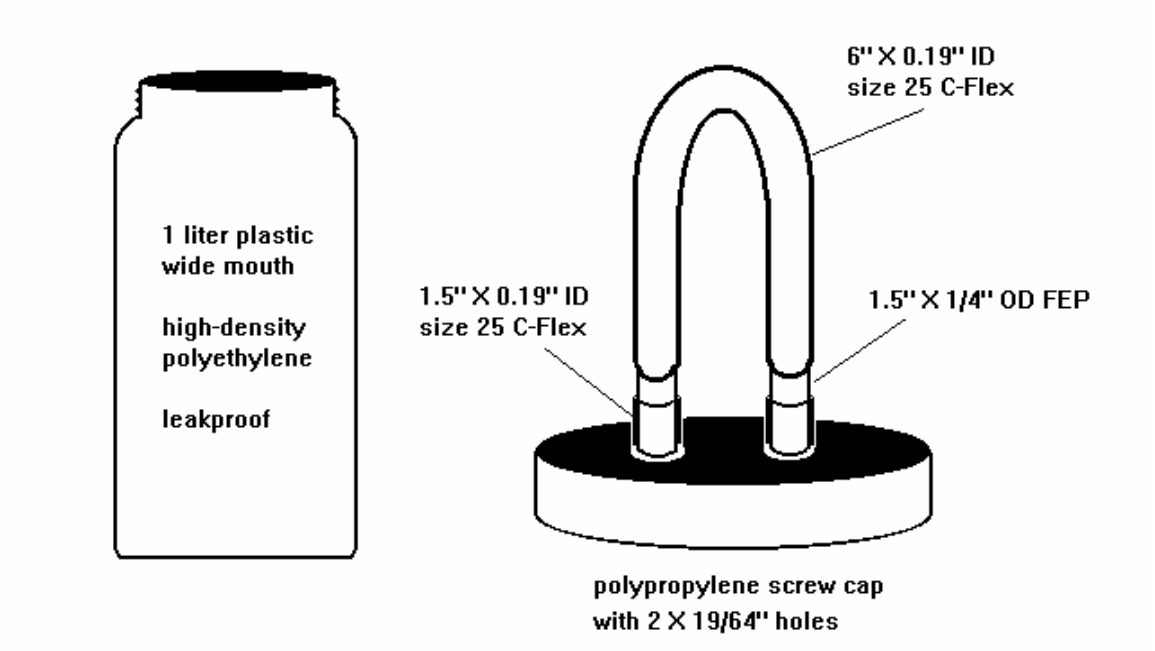


Figure 4.4: Ambient Sampling Apparatus

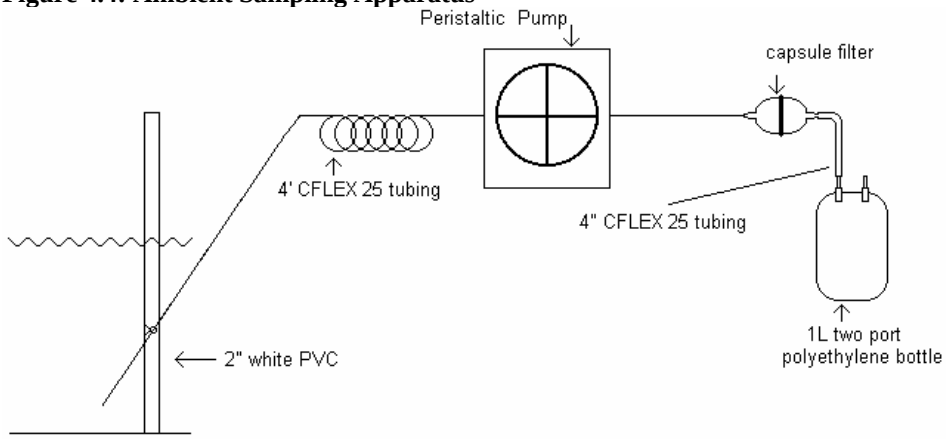


Figure 4.5 Effluent Sampling Apparatus

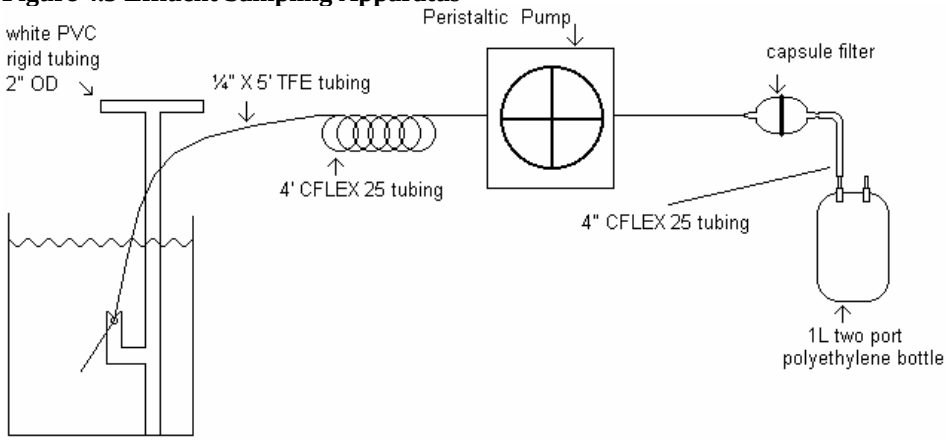
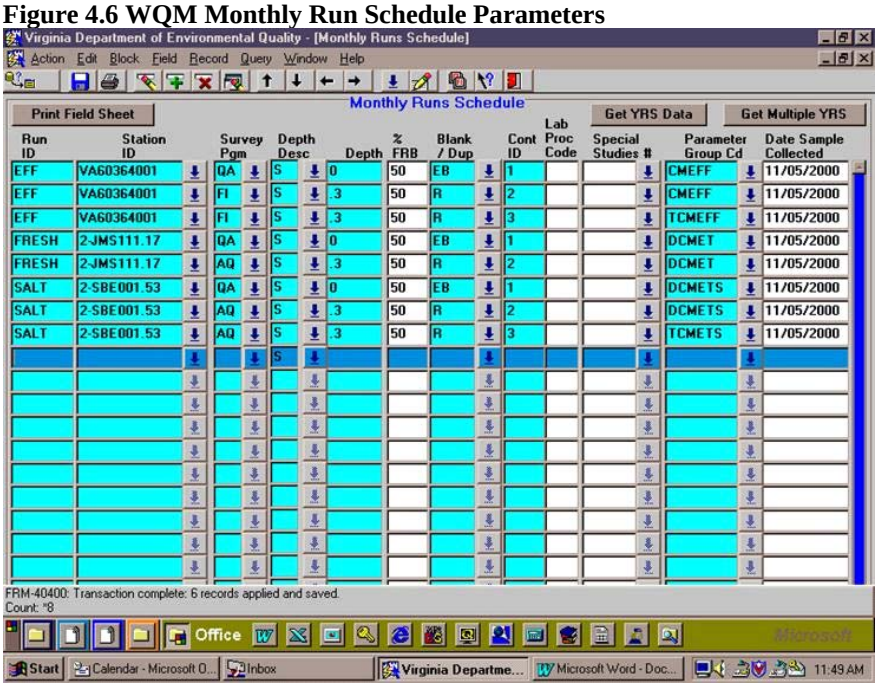


Figure 4.6 WQM Monthly Run Schedule Parameters



4.12. Chain of Custody Procedures and Completing COC Record

The Chain of Custody Record (COCR) shall provide documentation of everyone who has custody of the samples. The sample collector starts the COCR when the samples are collected. The COCR travels with the samples that are listed on the COCR. The COCR must contain the written signature of everyone that has custody of the samples and must document the relinquishment and receipt of the samples between sample custodians.

4.12.1. Transferring COC of samples from person to person

When custody of the samples changes, i.e. when samples are transferred from one person to another, the Custody Record must show the samples being relinquished by one person and received by another in the presence of each other. The custody of the samples and the responsibility of maintaining sample integrity are transferred during this process. The transfer process is documented on the bottom of the Chain of Custody Record form.

4.12.2. Transferring COC of samples via courier

This transfer is the same as above except that the transfer is not face to face. Both the collector and receiver document the integrity of the shipping container and the samples therein. The actual sample transfer is via courier in a tamper-proof shipping container.

4.12.3. Priority Codes

Code 7 – Standard turn around time (TAT), listed price
Code 6 – Chain of custody, standard TAT, listed price, no court action anticipated
Code 5 – ½ standard TAT, 1.5X listed price
Code 4 – 7 day TAT, 2X listed price
Code 2 – Chain of custody, court samples, standard TAT, 1.1X listed price.
Code 1 – Emergency sample, price will be determined after completion. Since this is requires lab employees to work around the clock to complete the analysis, these samples must be approved by a RD or agency director.

Bear in mind that timed analysis (BOD_5) cannot be run any faster and samples requiring immediate analysis (bacteria) will be done immediately so only use priority code 7 or 6.

4.12.4. Preparing the COCR Form

The COCR form may be prepared using one of two methods, in CEDS or manually. The preferred method is to use the CEDS system and the COCR form printed from information entered into the CEDS system. When using lab sheets, a multiple-part preprinted carbonless form is used. The COCR does not take the place of a lab sheet or CEDS data shipment. Consideration must be taken of the number of samples being shipped, as **a separate form must be completed for each cooler shipped**. Care must be taken to ensure that the information on the form concerning the samples exactly matches that found on the samples. Samples must be rejected if they do not match the COCR form.

4.12.5. COCR Fields

The COCR form contains several fields that unless otherwise identified as optional, must be filled out by the staff person filling out the COCR form. Below are descriptions of each field and how it should be filled out.

4.12.5.1. Sample Priority

Using the COCR does not in itself indicate the samples are a priority. The priority code number should be recorded on the COCR in the space where the 24-hour contact information is located. When using CEDS, this information is also captured in the field data screen and shipped to lab. When using lab sheets, each lab sheet must have the priority code entered. Advance notice of priority samples, or samples being delivered on a weekend, should be given the lab by contacting persons on the contact list. Only high priority samples (Priority Code 1), or samples with short holding times should be shipped to the lab for weekend delivery.

For samples requiring a COCR for use in TMDL monitoring or other sampling where a custody record is desired but samples do not need to be retained for potential legal action, regions should use priority code 6. This will ensure a COCR is on file for the sample, but the sample can be discarded after analysis as per standard DCLS procedure.

For samples requiring COCR because they must be retained for potential legal action such as enforcement or pollution response, regions should then use priority code 2.

4.12.5.2. Shipping Seal Number

This is number of the wire seal provided by lab that will be used to seal the individual coolers. All coolers not delivered in person to DCLS must have a shipping seal in place with the shipping seal number recorded on the COCR. The seal number is unique and entering it on the COCR makes that record unique. When the cooler is received by lab, they will compare the number of the unbroken seal on the cooler with the seal number that is recorded on the COCR.

4.12.5.3. Form Number

The multi-part forms will have a unique form number in the top right hand portion of the page. This number identifies the form from all others when no shipping seal number is used.

4.12.5.4. Samplers

The person who collects the samples or is present when the samples are collected and labeled and takes initial custody of the samples signs the COCR in this area.

4.12.5.5. Case No., PC No., VPDES No. or Other

This area is used to record the reference number for the sampling event. The number is program specific. Program protocols should be followed when entering this number. When using CEDS this reference number is referred to as the RUN ID.

4.12.5.6. Lat. & Long (optional)

If known, enter the latitude and longitude at the top of the page. This will help to identify the location of a sampling event. If collecting samples from multiple sites, enter lat/long into CEDS and do not fill out the lat/long space at the top of the page.

4.12.5.7. Region or Unit and Address

Enter the name of the region or central office unit responsible for collecting the samples and the address of the region or unit. This address may be used when returning certificates of analysis.

4.12.5.8. Phone & Fax Numbers and E-Mail

Enter this contact information for the collector. This information will be used to contact the collector if questions arise concerning the samples or analysis.

4.12.5.9. 24-Hour Contact Information

Enter the sample priority in this area. If the samples have a high priority (1), enter contact information that can be used 24-hours a day to confer about sample analysis and results. Examples include: home, work, pager and cell phone numbers as well as e-mail addresses.

4.12.5.10. Station ID

Brief description of the station at which the samples listed (on the same line) were collected. Limit the description to information necessary for you to identify the station from all others collected. This information must match the information on the sample tags and in the field log.

4.12.5.11. Date/Time

The date and time at which the samples were collected. The date is in the form MMDDYYYY and record time of sample using military time.

4.12.5.12. Basic Station Description or Container Type (optional)

Used for additional station description information if necessary. For situations where pre-cleaned containers are used, the container description information may be entered here.

4.12.5.13. Comp./Grab (optional)

This space may be used to identify samples other than routine grab samples. In the case of composite samples, the number of samples or time frame of the composite should be entered. If a horizontal or vertically integrated sample is collected, the information may be entered in the station description or under “FIELD OBSERVATIONS”.

4.12.5.14. Tests to be Run in the Lab

Enter the group code from the DCLS catalog of services which contains the analysis desired. If you have questions concerning the appropriate group code, contact DCLS using the contact list.

4.12.5.15. Observations & Field Tests (optional)

Enter field observations or field tests. These should be entered in the field log and on the lab sheets as well.

4.12.5.16. Relinquished By: Date/Time, Received By:

This area is used to record transfers of the sample custody. The sample custodian must relinquish the sample custody in the presence of the person receiving custody of the samples (except when shipping samples to lab). This is accomplished by the sample custodian signing the COCR form in “relinquished by” section and entering the date and time. The new custodian must sign the COCR form in the “received by” section in the company of the original custodian.

This process is followed in any subsequent changes in sample custody. Space is provided for four such transactions plus the final acceptance of custody by DCLS and the date and time the samples are received by DCLS. The supervising member and sample custodian of sampling the team must identify themselves as the supervision in this section.

4.12.5.17. Shipping Seal Received Intact, No. of, Lab Remarks

This area is used by DCLS to record the number of the seal broken to gain access to the contents of the cooler. They will note if the seal is intact. They will also note any other remarks such as the condition of the samples, ice present, etc.

4.12.6. Using CEDS for COCR Forms

CEDS can be used for shipping sample information to lab and for printing the COCR from the field data entered in the field data screen. Since the COCR is printed from CEDS after returning from the field, this method may not be used when sample custody is transferred from person to person in the field.

When using CEDS for shipping sample information to the lab, stations must be established in the station screen and all the pertinent information must be entered in the field data screen using established protocols. Additionally, you will need to **record the Shipping Seal Number** you will be using on the cooler and the **Shipped Date**. Review the information on-screen with the information on the sample tags and in the field log before printing the COCR form. Information on the COCR form may be changed in CEDS and reprinted if errors are made up until the time that the data is shipped to DCLS.

Click on the <PRINT CHAIN CUST> button on the field data screen to print the COCR form. Print two copies.

Sign one copy as "Relinquished by:" and complete the date/time at which the samples are relinquished or sealed in the cooler. This signed COCR is the official COCR. Place it <u>inside the cooler</u> in a Ziploc type waterproof container. The collector retains one copy with the field log.

4.12.6.1. Multiple-Part Preprinted Form

Complete the multi-part form using the definitions above. All sections not indicated as optional must be completed. Make certain that the information on the form exactly matches the information on the sample tags, the field log, and the lab sheets. Field teams may correct minor mistakes while filling out the form may be corrected by a single line cross out and initialing the crossed-out portion. Major mistakes will require a new form to be completed. The form with the mistake must be destroyed.

Sign the bottom portion of the COCR as "Relinquished by:" and complete the date/time at which the samples are relinquished or locked in the cooler. The original signed copy is sent <u>inside the cooler</u> in a Ziploc type waterproof container. The collector retains one copy.

4.12.7. Preparing Samples for Shipment

Before shipping samples, make sure that the information on the sample tags exactly matches the information on the COCR, the field log, and either lab sheets or the CEDS field data entry screen.

1. Ensure that the sample labels on wired tags are securely wired to the sample containers and/or that sample labels are securely adhered to the containers.

2. Fill the cooler with fresh ice. Sufficient ice is enough ice to fill the bottom and sides of the cooler with at least two inches of ice as well as any voids between sample containers. The maximum weight of the cooler is 70 lbs.

3. Record the wire shipping container seal number on the COCR. Put the original signed copy of the COCR form inside the cooler inside of a waterproof Ziploc type bag. If used, include the lab sheets inside the Ziploc bag as well. Each individual cooler must contain all the documentation for the samples it contains and only for the samples it contains.

4. Close and seal the cooler by closing the hasp (the bail over the eyelet) and securing it using the seal supplied by lab. Put the wire seal individually through both the bail and the eyelet of the hasp. Before securing the wire seal, make sure the serial number of the wire seal matches the number entered on the COCR form.

4.12.8. Possible COCR Problems and Solutions

Problem 1: The sample tags do not match either the CEDS information or the lab sheets, or the COCR form.

Solution: The collector can correct these errors only if their field log contains information that will rectify the error. A correction of this type must be meticulously documented in the field log. Only the collector can make changes of this type. If the samples have been shipped, the collector will have to go to the lab to make the corrections.

Problem 2: Cooler is sealed before all the samples are placed inside.

Solution: The seal may be broken to inspect the cooler contents and to add or remove samples as needed. If the seal is broken, a new seal will be required and a new COCR must be developed. In CEDS, return to the field data screen (prior to automated data shipment @ 0900 and 2200, check for the lab send date in the CEDS field data screen), change the seal number, print a new COCR and replace all three of the original COCRs. You may simply scratch through the original seal number on the COCRs and write the new number by it and initial the change then make the correction in CEDS. If using the multiple- part COCR, clearly correct the seal number on the COCR and initial the correction.

Problem 3: You do not have any wire seals.

Solution: If wire seals are not present, the use of packing tape as a special sample seal may be used. Using the COCR template found below, print and enter the required data as you would normally with a standard wire seal. Place this form in a Ziploc bag and tape to the cooler so that the opening of the bag is sealed with tape. Place sufficient tape around the cooler lid to secure the lid shut and two straps of tape to bind the lid to the cooler to prevent unauthorized access.

Another option is for the sampler or someone else who assumes custody of the samples accompany the samples to DCLS. Samples delivered by the sample custodian do not require custody seals if they are in your control the entire time. Container seals are suggested however.

Problem 4: The wrong wire seal number is recorded on the COCR.

Solution: See problem 2.

4.12.9. The Personal Field Log

The personal field log is a legal document used to record information concerning all aspects of an investigation. The log must have bound and numbered pages. The log should be kept in a secure place. Only the owner of book should make notations in the personal field log.

At a minimum, the field log should record information which links that section of the field log with the information found on the COCR. The following information should be page headers:

1. Investigation identification information such as PC, or permit number
2. Date of investigation

The field log should also contain information that supports but does not duplicate information found on the COCR. This includes, but is not limited to:

1. Names, addresses, phone numbers of complainant, permit holder, operators, etc.
2. Detailed descriptions of the sampling sites
3. Variations, if any, from the WQM SOP manual
4. Types of samples collected (grab, straight timed composite including time frame, volume weighted composite, cross-section composite, vertically integrated composite).
5. Pre and post meter calibration information
6. Number and type of QA/QC samples collected
7. Detailed observations of the site including physical lay of the land such as upstream, up-gradient, east/west, etc.
8. Detailed information included as comments in CEDS or on the lab sheets such as “expect high BOD”
9. Documentation of changes to the COCR.
10. Shipping seal number.

4.12.10. Call List for Sample Related Issues

This is a list of persons, listed in order of priority, to call for help to the listed problems. This list is continually updated. The most updated copy can be found on DEQNet at http://deqnet/docs/water/Water_quality/monitoring_and_assessments_program/DCLS/CALL_LIST_FOR_SAMPLE_RELATED_ISSUES.doc

4.12.10.1. Emergency Laboratory Services

After Hours Emergency Services Officer: (804) 418-9923 (pager)
If no reply after 15 minutes, call 1 800-283-8252

Order	Contact Name and Title	Phone Number(s)	E-Mail
1	Ed Shaw – DCLS Asst. Dir. of Analytical Services & DEQ Coordinator	804-648-4480 x152 804-371-7973 (F)	Ed.Shaw@dgs.virginia.gov
2	Dr. Thomas York - DCLS Deputy Director	804-641-7071 (C) 804-378-8203 (H)	Tom.York@dgs.virginia.gov
3	Dr. James L. Pearson – DCLS Director	804-648-4480	Jim.Pearson@dgs.virginia.gov

4.12.10.2. Routine Sample Delivery Problems

Order	Contact Name and Title	Phone Number(s)	E-Mail
1	Cindy Johnson- DEQ & DCLS Liaison	804-698-4385 804-437-6185 (C)	Cynthia.Johnson@deq.virginia.gov
2	Roger Stewart- CEDS Administrator	804-698-4449 804-370-8043 (C)	Roger.Stewart@deq.virginia.gov
3	Tish Robertson- Standards Analyst	804-698-4309 804-212-2253 (H)	Tish.Robertson@deq.virginia.gov
4	Melody Morton- DCLS	804-648-4480 x 140 804-418-9932 (P) 804-786-4270 (F)	Melody.Morton@dgs.virginia.gov
5	Lewis Baker- DCLS	804-698-4480 x 141 804-786-4270 (F)	Louis.Baker@dgs.virginia.gov
6	Elaine Mason-DCLS	804-648-4480 x138	Elaine.Mason@dgs.virginia.gov

4.12.10.3. Problems Specific to Data Transfer

Order	Contact Name and Title	Phone Number(s)	E-Mail
1*	Cindy Johnson- DEQ & DCLS Liaison	804-698-4385 804-437-6185 (C)	Cynthia.Johnson@deq.virginia.gov
2*	Roger Stewart- CEDS Administrator	804-698-4449 804-370-8043 (C)	Roger.Stewart@deq.virginia.gov
3*	Tish Robertson- Standards Analyst	804-698-4309 804-212-2253 (H)	Tish.Robertson@deq.virginia.gov
4	VCCC Helpdesk- Obtain help desk ticket for FTP site	866-637-8482	vccc@vita.virginia.gov

* Can perform manual download of WQM data to ship to DCLS.

4.12.10.4. Sample Collection Information & Scheduling With DCLS

This numbers are provided for non-routine sample collection and scheduling. Please make certain when scheduling bacteria samples that you confirm that one of the following persons know when and how many samples will be arriving and what services will be requested.

Order	Contact Name and Title	Phone Number(s)	E-Mail
1	Roger Stewart- CEDS Administrator	804-698-4449 804-370-8043 (C) 804-739-5995 (H)	Roger.Stewart@deq.virginia.gov
2	Cindy Johnson- DEQ & DCLS Liaison	804-698-4385	Cynthia.Johnson@deq.virginia.gov
3	James Beckley- DEQ QA Coordinator	804-698-4025 804-643-3139 (H)	James.Beckley@deq.virginia.gov
4	Ed Shaw – DCLS Asst. Dir. of Analytical Services & DEQ Coordinator	804-648-4480 x152 804-371-7973 (F)	Ed.Shaw@dgs.virginia.gov
5	Deborah Paul- DCLS Group Manger. Note: only for bacteria scheduling	804-648-4480 x310 804-997-3555 (P) 804-371-0666 (F)	Debbie.Paul@dgs.virginia.gov

4.12.10.5. Ordering Sample Kits and Containers

Melody Morton, DCLS Sample and Records Management 804-648-4480 x 140

Mattie Jones, DCLS Customer Service Support Technician 804-648-4480 x 104

ORDERING CLEAN METALS KITS

Norma Roadcap, Metals & Radiochemistry 804-648-4480 x 350
norma.roadcap@dgs.virginia.gov

4.12.10.6. Courier Service

Grier Mills, DCLS Customer Service – Planning Group 804-648-4480 x 154

4.12.10.7. Ordering Chain of Custody Equipment

Melody Morton, DCLS Sample and Records Management 804-648-4480 x 140

4.12.11. Directions to DCLS

DCLS is located at **600 North 5th Street, Richmond, VA 23219**

Temporary parking is available for sample delivery at sample receiving at 600 North 4th Street. To reach the DCLS lobby from sample receiving, walk around to the other side of the building to 600 North 5th Street.

From West of Richmond

1. Start out going East on I-64 E.
2. Take the I-64 E exit- exit 75- toward WILLIAMSBURG/NORFOLK. 0.17 miles
3. Take the 3RD STREET ramp toward COLISEUM/DOWNTOWN. 0.09 miles
4. Stay straight to go onto N 3RD ST. 0.13 miles
5. Turn LEFT onto E LEIGH ST. 0.06 miles
6. Turn LEFT onto N 4TH ST. 0.04 miles

Sample receiving is in the middle of the block on the right.

From South of Richmond

1. Start out going North on I-95 N.
2. Take the CHAMBERLAYNE AVE exit- exit number 76A. 0.16 miles
3. Turn LEFT onto CHAMBERLAYNE AVE/CHAMBERLAYNE PKWY. 0.20 miles

4: Turn SLIGHT LEFT onto W LEIGH ST. 0.30 miles

5. Turn LEFT onto N 4TH ST. 0.04 miles

Sample receiving is in the middle of the block on the right.

From East of Richmond

1. Start out going West on I-64 W toward RICHMOND.
2. Take I-95 S/5TH STREET exit- exit number 190- on the left toward PETERSBURG/DOWNTOWN/COLISEUM. 0.29 miles
3. Stay straight to go onto N 5TH ST. 0.12 miles
4. Turn RIGHT onto E JACKSON ST. 0.12 miles
5. Turn LEFT onto N 3RD ST. 0.07 miles
6. Turn LEFT onto E LEIGH ST. 0.06 miles
7. Turn LEFT onto N 4TH ST. 0.04 miles

Sample receiving is in the middle of the block on the right.

From North of Richmond

1: Start out going South on I-95 S toward RICHMOND.

2: Take I-64 E exit- exit number 75- toward WILLIAMSBURG/NORFOLK. 0.17 miles

3: Take the 3RD STREET ramp toward COLISEUM/DOWNTOWN. 0.09 miles

4: Stay straight to go onto N 3RD ST. 0.13 miles

5: Turn LEFT onto E LEIGH ST. 0.06 miles

6: Turn LEFT onto N 4TH ST. 0.04 miles

Sample receiving is in the middle of the block on the right.

Figure 4.7 COCR

VIRGINIA DEPARTMENT OF ENVIRONMENTAL QUALITY
629 EAST MAIN STREET, RICHMOND, VIRGINIA 23240

CHAIN OF CUSTODY RECORD

2801

Shipping Seal Number _____

SAMPLERS: (Signatures) _____
Region or Unit _____
Address: _____
Phone No.: _____ Fax No.: _____
E-Mail: _____
If this is an Emergency (Priority1) sample, please print 24-hour contact name and number. _____

Identifying No.(s) _____ Case No. or PC No. or VPDES No. or other _____ Lat. & Long. (if known) _____

STATION ID	DATE	TIME	BASIC STATION DESCRIPTION or CONTAINER TYPE	COMP. / GRAB	TESTS TO BE RUN IN LAB	Observations & Field Tests (optional for water & air media)

Relinquished by: (Signature)	Date/ Time	Received by: (Signature)	Relinquished by: (Signature)	Date/ Time	Received by: (Signature)
Relinquished by: (Signature)	Date/ Time	Received by: (Signature)	Relinquished by: (Signature)	Date/ Time	Received by: (Signature)
Relinquished by: (Signature)	Date/ Time	Received in Laboratory by: (Signature)	Date/ Time	Shipping seal received in place? ____ Seal No.____ Lab Remarks:	

Original to Accompany Shipment; copy attached to shipping label located outside of cooler; copy to Sampler

CHAPTER 5: QUALITY ASSURANCE AND QUALITY CONTROL

5.1. Quality Assurance of Field Parameters

As outlined in sections 3.1 and 3.2 of this manual, it is important for field teams to properly calibrate and perform quality assurance checks of field parameter instruments. Table 3.1 and 3.2 lists the acceptable calibration and end of day check ranges. If a unit fails a quality assurance check, it should undergo maintenance/repair and the affected parameters collected by the unit on that day should not be entered into CEDS.

5.2. Quality Control Samples for Ambient Samples

5.2.1. General

For specific instructions to enter QA/QC samples into the WQM module **of CEDS, see Appendix D for details.**

Analyte-free water (e.g. DI or lab grade water) is water in which all analytes of interest and all interferences are below method detection limits.

Always use analyte-free water to prepare sample or equipment blanks and for the final in-house decontamination rinse.

Transport sufficient analyte-free water to the field in clean containers of suitable construction, such as cubitainers or large plastic jars.

Whenever possible, collect equipment blanks and field split samples on the same sample run.

5.2.2. Equipment Blanks for General Water Quality Parameters

Equipment blanks check for carryover contamination between sampling sites or for the effectiveness of cleaning procedures. For WQM programs using a bucket to obtain samples, the equipment blank needs to check for carryover contamination. Equipment blanks are samples generated from the sampling equipment in use.

Collect equipment blanks before collecting a sample at a sample site. Whenever possible, perform an equipment blank at the site designated for a field split sample.

For general ambient water quality sampling, an equipment blank needs to be collected at a minimum of once for each 25 sites sampled (4%). The only exception to this is for bacteria samples, as the bottles are certified sterile. Each regular field sampler should meet this 4% equipment blank check. The Quality Assurance Coordinator will provide quarterly reports of performance will help ensure field teams are meeting this objective.

1. Flush or rinse the sampling device with analyte-free water at least once prior to collecting the equipment blank. Rinsing of sampler must follow the same procedure (number of

rinses, equivalent volume, and manner of rinsing) as when performing routine rising with sample water. Discard the rinse water.

a. For the pump and hose method, first rinse the outside of the pump and hose intake assembly with DI water then rinse the pump and hose as using the same procedure when rinsing with sample water. Typically, this will require at least 5 gallons of analyte-free water.

2. Rinse sample bottles with analyte-free water using the same number of rinses, volume, and manner as if rinsing with sample water. Discard the rinse water.

3. Run analyte-free water through the sampling equipment (pump and hose, bucket etc.) and then pour or transfer the water into the respective sampling containers, preserve equipment blank samples identically as samples normally collected. Transport equipment blank samples the same way as transporting regular samples to the lab.

To prevent the possibility of confusing equipment blank sample with regular or duplicated samples, place the sample tags on the equipment blank containers prior to collecting split samples. **Mark sample tags with EB under the sample type field**. Be sure to enter this sample designation in CEDS upon return to the office.

If the equipment blank result is three times higher than the method detection limit, data collected by the sampler on that particular date and parameter are considered suspect and will be flagged in the database by the QA Coordinator. If on more then three occasions per year/per analyte equipment blanks come back unacceptable for a field team member, a special audit of field performance and/or training may be necessary.

5.2.3. Field Split Samples for General Water Quality Parameters

Split samples are two or more samples collected to determine sampling technique precision and/or laboratory precision. Sampling technique precision is determined by the collection of field split samples.

Field splits should occur, whenever possible, at the same stations selected for field blank samples.

For general ambient water quality sampling, a field split sample of each routinely sampled analyte needs to be collected once for each 25 sites sampled (4%). Each regular field sampler should meet this 4% field split check. The Quality Assurance Coordinator will provide quarterly reports of performance will help ensure field teams are meeting this objective.

Stations designated for split samples will be chosen randomly. Split samples must follow the same collection, preservation, and handling in accordance with the procedures in this manual.

5.2.3.1. Split Sampling Using a Bucket

If performing split samples using a bucket, use the appropriate sampling technique and obtain one bucket of water and fill two identical containers sequentially.

1. Split samples for each analyte should be filled in sets (i.e. fill total nitrogen S1 then total nitrogen S2). This will minimize the potential of sample water in the bucket from significantly changing in physical or chemical characteristics.
    a. If the sample water in the bucket appears to have significantly changed physical characteristics while filling split samples (i.e. solids are settling to the bottom), it is acceptable to either use a clean, stainless steel spoon to mix sample bucket contents or swirling the bucket to resuspend materials.

2. For bacteria split samples, lowering both bottles at the same time into the water is preferred when direct access to the water is not feasible.

5.2.3.2. Split Sampling Using the Pump and Hose Method

If using the pump and hose method, containers can be filled using one of two methods:

1. Place the identical split sample containers side by side and rapidly move the hose discharge back and forth across the tops of the containers until both containers are filled. Repeat the process for each required sample container.

2. Use a Y-fitting at the end of the discharge hose to fill both sample containers simultaneously. When using the Y-fitting, keep the discharge as level as possible so that discharge stream is divided as equally into each of the containers and the containers fill at the same rate.

Collect bacteriological split samples side by side directly from the source.

5.2.3.3. Labeling and Recording Split Samples

Designate the split sample bottles for each analyte as S1 and S2. If triplicate or additional split samples are needed for a particular analyte, use S3 and subsequent designations. **Mark sample tags with S1, S2, etc. for the designated sample bottle.** Be sure to enter this sample designation in CEDS upon return to the office.

If the split results are three times higher than the method detection limit and greater than 10% RPD (20% for total suspended solids or 10x difference between bacteria splits), the data collected on that particular date and analyte by the field team member are considered suspect and will be flagged in the database by the QA Coordinator. If on more then three occasions per year/per analyte sample splits come back unacceptable for a field team member, a special audit of field performance and/or training may be necessary.

5.3. Quality Control Samples for Sediments

Equipment blanks check for carryover contamination between sampling sites or for the effectiveness of cleaning procedures. Equipment blanks use the same procedures which are used in collecting samples.

To avoid having to clean equipment between the stations, getting enough equipment (i.e. pans, dredges) for the entire sampling run is encouraged. If staff can bring enough equipment for an

entire sampling run, it removes the possibility of carryover contamination. This would result in an equipment blank for sediment collection to verify cleaning procedures. In that instance, the equipment blank may be performed in the field at the first station prior to sampling or in the laboratory. An equipment blank is required whenever a batch of sediment sampling equipment is cleaned.

If it is not possible to have dedicated set of equipment at each sample site, the equipment must be cleaned in the field between each station. This will require two equipment blanks. One is prior to sampling at the first site to verify general cleaning procedures. The other equipment blank must be done between two sediment stations in the field. This second blank will confirm if there is carryover contamination between sample sites.

5.3.1.1. Collecting a Equipment Blank Sample

1. Run analyte-free water through all the sampling equipment that contacts with sediment samples and collect all the rinsate in the cleaned stainless steel or Teflon tray used to prepare sediment samples.

2. Transfer the rinsate from the tray to a sediment container and submit it for analysis.

3. Label the sediment container containing the rinsate water with the required information and **use the sample code EB**.

4. Upon returning from the field, enter the information into CEDS and **be sure to use the EB designation in the sample code field**.

If the equipment blank results are three times higher than the method detection limits, the sediment data collected on that particular date and sample run are suspect and will be flagged in the database by the QA Coordinator. If a region has more than two unacceptable sediment equipment blanks per year, an audit and/or a training session for the region on proper cleaning techniques may be necessary.

5.3.2. Field split sediment samples

One of every ten (10%) of sediment samples collected will be field split samples. The split sample station must be randomly selected.

The split samples will be collected from a single site as one large sample with sufficient volume to be halved into two separate samples of equal volume. Homogenize the sample using a clean spatula prior and splitting the sample into two jars for analysis.

Split sediment sampling and handling must follow the same procedures outlined in section 4.8 of this manual.

CHAPTER 6: SAMPLE IDENTIFICATION AND CORRECTIVE ACTION

6.1. Field data sheet

A field data sheet is required to be carried in the field by the sampler for each run. Make entries in the field data sheet for all the field parameters.

6.2. Sample label and tag

The sampler should print the label from the pre-print label file in the computer. The label should have the following information: station ID, date collected, time collected, depth, unit code, collector, group code, preservative, lab processing code, blank/dup designation, priority and container number. The preprinted label should have filled in all the information except the collected time based on the monthly run information in CEDS. The collector fills in the time in the field using an indelible ink pen. The Avery label should be placed on the plastic or glass sample bottles or placed on a label that is then attached to the sample containers. When using cubitainers, the label needs to be placed on the sample tag that is then attached to the containers.

6.3. Corrective Action

For the corrective action plan to be operative, all personnel associated with the program must report any suspected deficiencies in procedures or equipment. This is especially important for DEQ field personnel and DCLS lab personnel. Identification and correction of the problems in sample collection, preservation, handling and analysis is essential for an effective program.

The corrective action request (CAR) form (see Appendix C) is used to document the problem and steps taken for correction. CAR forms may originate in regions, central office or DCLS.

The originator:

- Identifies the problem.
- Lists possible causes (if known)
- Notes the date the problem was identified
- Identifies samples or field data that may be invalid as a result of the problem
- May recommend corrective action

CAR forms that originate in the region or central office are forwarded to the appropriate QA officer for review and recommendations. The QA officer forwards the form to the appropriate supervisor for review, recommendations and a final decision on appropriate corrective action. After resolution of the problem, the supervisor provides copies of the completed form to appropriate personnel on his staff and central office QA/QC officer. The supervisor has the responsibility for implementation of this corrective action. The QA/QC staff in central office

may provide additional comments or recommendations to the supervisor for his review if requested.

CAR forms that originate in DCLS are forwarded to the central office QA officer for review, recommendations or concurrence. Then, if appropriate, these forms are forwarded to the appropriate supervisor for a final decision and implementation.
It is the responsibility of the originator to notify management and the QA officer in central office if the corrective action system is not operating effective. In this situation, the originator may elect to call or send a CAR form directly to QA/QC central.

CHAPTER 7: SAFETY

This chapter is intended to assist field personnel in the safe performance and collection of water quality data. Fieldwork requires an awareness of potential hazards and knowledge of basic safety procedures. Field personnel routinely come in direct and indirect contact with waterborne pathogens, chemicals, and potentially hazardous plants and animals. In addition, work from bridges and stream banks present physical hazards, such as vehicles, drowning, and slip/trip/fall for which we should be aware. Safety is often nothing more than using common sense and being aware of your surroundings. Advanced planning can eliminate or reduce many safety hazards.

This chapter is intended to provide a general overview on safety, the entire Boat & Water Safety Policy adopted by the agency and for which staff are accountable can be found at: http://deqnet/docs/admin/admin_policy/Safety/Boating%20and%20Water%20Safety%20Procedures.pdf

7.1. Basic Safety Preparation

Basic preparations should be routine before every sampling run. Per the agency health and safety manual found at: http://cntrldeqnet/programs/healthsafety/documents/PolicyManualFINAL.doc , each employee shall be responsible for keeping their calendars current and for sharing their schedules with their immediate supervisor. At a minimum, complete a plan for each field trip, and leave it at a designated location in the office. The trip plan should include the following information:

- Names of participants, including guests and observers, with emergency contact information
- A basic itinerary, including where and when sampling will occur, along with departure and expected return time(s) and date(s)
- Hotel information and contact phone numbers (for overnight trips)
- Cell-phone numbers or radio frequencies

Here are important tips to remember:

- Staff should perform a pre-trip safety inspection of their vehicles and gear.
- Staff should have current certification in both Basic First Aid and CPR in order to provide emergency medical services.
- Whenever possible, or when conditions merit, fieldwork should be carried out in pairs. Have a cell phone, radio, or other emergency communication available when in the field.
- Carry basic safety equipment: first-aid kit, flashlight, boots, rain gear, and antibacterial soap or hand cleaner.
- Before leaving, check the weather forecast at the site. While in the field, be aware of changing weather conditions and the potential for flash floods, storms, or tornadoes.

- Be aware of potential hazards at a monitoring site. Examples include unsafe entry or exit locations, presence of unusual materials near the site or in the water, and similar hazards.

- Make a habit of carrying a packet of general safety information in each vehicle or boat—

  - ✓ Material-safety data sheets (MSDS) for chemicals used at the site
  - ✓ Basic first-aid manual
  - ✓ Emergency phone numbers and radio frequencies
  - ✓ Locations of emergency facilities (hospitals, police and fire departments, U.S. Coast Guard)

The checklist in Table 7.1 provides common safety equipment to have during a sampling trip.

Table 7.1. Basic Safety-Equipment Checklist

Yes	No	Safety Items
		General Items
		First-aid kit
		Flashlight and spare batteries
		Cell phone and/or marine radio
		Rain gear
		Hat, sun screen, and sunglasses
		Drinking water or sports drinks
		Safety cones, ANSI Class 2 or 3 garments (for working on bridges, ROW)
		Toolbox with basic tools (for vehicles)
		Antibacterial soap or hand cleaner
		Spill kits (if carrying >100 ml of preservatives)
		MSDS for preservatives (if carrying >100 ml)
		Hand-held eyewash unit
		Protective goggles
		Container to carry preservatives
		Fist of emergency phone numbers and office contacts
		Fire extinguisher (for vehicles)
		Boating or Wading
		Waders, hip boots, rubber knee boots per team member (if electrofishing or wading)
		Personal flotation device (PFD) per team member

7.2. General Laboratory and Warehouse Safety

When working in the field preparation room or warehouse, keep the following in mind.

1. Turn on and use the fume hood and wear appropriate personal protection equipment (goggles, apron, gloves, etc.) when pouring reagent grade chemicals into beakers or when preparing standards. Section 7.3 contains additional information on handing reagents.

2. When not in use, store reagent bottles in the appropriate chemical storage cabinet. Table 2.1 in section 2.6.1 contains a list of commonly used reagents and recommended storage areas. Never place incompatible materials in the same storage cabinet. (I.e. oxidizers with acids, safety flares with flammable liquids, etc.)

3. Maintain sufficient clearance for storage cabinets, electrical panels, and exits to ensure safe access. Typically, this is three feet.

4. Ensure emergency equipment such as fire extinguishers, eye wash and shower stations, and related items are charged or otherwise in good working order.

5. When disposing of significant quantities of hazardous materials, use a certified hazardous waste contractor.

Contact the regional or agency safety officer if there are questions about procedures. A contact list is available at http://deqnet/programs/healthsafety/contactus.asp.

7.3. Reagent Chemical Safety

Ensure that Material Data Safety Sheet (MSDS) are available for all chemicals used. These reports describe signs and symptoms of exposure, list first-aid procedures, and give details on cleaning up spills. Here are some general tips to remember:

- Protect field personnel by securing all chemicals using a container that will resist and contain the material in the event of an accident or spill such as an ice chest.

- If transporting large (>100 ml) quantities of chemicals to the field. Carry a spill kit containing neutralizing or absorbing agents. The most common and dangerous chemicals carried during sampling trips are sulfuric and nitric acids. For acid preservatives, a container of baking soda is a low cost and effective acid spill neutralizing agent.

- Label all chemical containers clearly. Include handheld eyewash in a chemical-safety kit whenever chemicals are part of the sampling plan.

- Use premeasured vials or sample bottles filled with sufficient volume of preservative instead of transporting large containers of preservatives. Although there is always a chance of a spill, the risk is significantly reduced by handling smaller volumes.

- Do not pipette by mouth. Always use mechanical pipettes or pipette bulbs.

When transporting gasoline, keep the following items in mind:

- If extra gasoline is carried, ensure it is transported and stored in approved containers.
- Remove portable tanks from vehicles or boats before filling them with fuel. Touch fuel pipes or tanks with the spout to prevent buildup of static electricity.
- Do not fill tanks completely full; leave room for the gasoline to expand.
- Cap tanks tightly to prevent vapors from escaping.
- Clean up spills immediately and air used rags before storing them. Store containers in a well ventilated area away from the engine and passenger compartments.

7.3.1. General Safety Procedures for Handling Acids

1. All acid fumes are harmful to lungs and unprotected parts of the body. When using any concentrated acids in a laboratory setting, use the fume hood located in the lab and set the fume hood to exhaust the gas. Be sure the hood is set at a high enough setting to whisk fumes away from the technician.

2. Concentrated acids will quickly react against organic and metal surfaces. Protect your hands by using Nitrile or acid grade gloves. Latex gloves are not sufficient as the acid can penetrate though the latex and contact with your hands. Wear a lab coat and chemical apron to protect you long enough to remove the articles of clothing if acid spills on you. Wear goggles or, at the very minimum, glasses with splash shields. A face shield is ideal, but goggles or safety glasses will protect your eyes against most spills.

3. Know where the safety shower and eyewash station is located at and ensure that they are properly working. If acid or any other chemical comes into contact with your eyes or on your body, wash the affected area for at least 15 minutes. Pull back eyelids to allow water to wash away any foreign material splashed in the eye. Report all incidents to the safety officer and seek immediate medical attention.

4. All acids react to water. When mixing acid with water, it creates heat. Rapidly mixing large quantities of concentrated acid with water can create enough heat to cause a thermal burn of unprotected hands and break glassware.

5. Whenever mixing a concentrated acid to water, always remember to ADD ACID TO WATER and not the other way around. If you add water to acid, it can cause excessive splattering due to the exothermic reaction explained above.

6. Do not discard high strength acids down the drain. This will result in corrosion of metal pipes in the drain or damage the sewer system. Always discard used acid into appropriate acid waste containers or by neutralizing using an aqueous solution of sodium hydroxide or baking soda. If neutralizing acids, do so in the fume hood and check the pH of the

solution at a regular basis using litmus paper. Adjust pH to at least 4.00 before discarding down the drain with plenty of water.

a. For disposing of large quantities (>100 ml) of concentrated acid, use of an approved hazardous waste disposal company. Contact your local safety officer if a disposal company is not already under contract.

7.4. Wading

1. Do not attempt to wade in a stream where the depth multiplied by the velocity is greater than or equal to 10 ft^2/s. For example, a stream 2 ft deep with velocities of 5 fps or more can be dangerous (Lane and Fay 1997).

2. When wading in deep or fast flowing water, wear a Coast Guard–approved personal flotation device (PFD). Use of a PFD is encouraged when doing any type of wading as a stream may have depressions, holes, or loose footing which may cause a fall.

3. Wear hip boots or chest waders. Boots and waders protect against cold, contaminants, and underwater objects. Be aware of the possibility of slipping and going under water (feet up, head down) while wearing them.

4. Avoid hip boots with tight ankles and waders with tight-fitting tops. They are difficult to remove in an emergency. When using chest waders, it is a good idea to wear a Coast Guard–approved PFD as if the waders fill with water, it can pull someone underwater.

5. Be aware of surrounding conditions. Watch for floating debris, areas of quicksand, and deep pools. Watch the stream stage, especially if there is a chance it could raise rapidly.

7.5. Working from Bridges

Samples are often collected from bridges. Such work is very dangerous, so take steps to minimize the risks. Use basic safety equipment for bridge sampling. Equipment may include reflective vests, orange safety cones, and four way flashers on vehicles. Sites where staff must climb over the side of a bridge such as accessing USGS gauges, must wear a Coast Guard approved PFD

NOTE: Staff working or stopped in the right of way (ROW) adjacent to a public roadway in order to access sample stations, gauging stations, or unload equipment shall wear ANSI 107-2004 class 2 or 3 compliant garments while in the ROW

7.5.1. Selecting Bridges for Sampling

When reconnoitering possible bridge sample sites, staff should keep the following items in mind.

1. Does the bridge have a shoulder/sidewalk to sample from? If not, is there sufficient room to perform sampling while not obstructing traffic?

2. Will traffic volume/speed allow safe access for monitoring?

3. Are there sharp curves at one or both ends of the bridge? If there is a sharp curve, will drivers have enough time to respond and slow down?

4. Park the vehicle in a location such that it does not block or obstruct the flow of traffic and apply the use of four way flashers. Preplan/prepare so that the vehicle is located in the right of way, no more then 15 minutes.

5. Does the bridge have a posted sign for no fishing or loitering? If so, sampling on the bridge is not encouraged as it is determined unsafe for pedestrians and may be illegal.

7.5.2. Vehicle Parking Procedures

- Park in a location away from sharp curves to allow time for drivers to slow down.
- Pull completely off the roadway and onto the shoulder. Be sure to leave sufficient room to exit and enter the vehicle without interfering with traffic flow.
- If the vehicle is parked along the road, turn on the vehicle hazard signal.
- Set up sampling away from the vehicle to allow observing of traffic from both directions.

7.6. Working from Boats

Staff must provide their immediate manager/supervisor a complete float/task plan, including a cell phone number (unless staff does not have a personal/agency phone). The complete information can be conveyed via email or by using the example in Table 7.2

Use a boating-safety checklist when inspecting boats and tow vehicles before leaving. (Example checklist is in Table 7.3).

When operating a boat:

- Staff shall work in pairs when conducting operations from any watercraft.

- Boat operators must be certified through a VDGIF approved boat safety course (www.dgif.virginia.gov/boating/education/) and undergo reexamination as required.

- All staff assigned to perform work on a boat must successfully complete a course in basic boat operations (http://boatingbasicsonline.com/content/va/)

- Know its capacity. Look for a capacity plate near the operator's position or on the transom. This plate indicates the maximum weight capacity or the maximum number of persons the boat can carry safely. The maximum weight includes the combined weight of passengers and gear.

- On outboard powerboats, check the capacity plate for the maximum horsepower rating; do not exceed the horsepower rating.

- While a boat is underway, all personnel should be seating in designated areas. When moving to another part of the boat, use secure handholds to prevent falling

- Use caution when refueling a boat. Check the entire fuel system for leaks, and tighten connections frequently. Turn off the engine and all electrical equipment before adding fuel to the tanks. If the boat is equipped with a power ventilation system, turn it on for at least four minutes before starting the engine to clear gasoline vapors from the bilge. Never smoke or strike a match while fueling or near a fueling dock.

- Make sure the boat is in good operating condition and full of gas before taking it out on the water. The checklist in Table 7.2 may be used to ensure the boat is ready for use.

- Check weather conditions before departure. If a storm comes up while on the water, head for shore. Always carry a marine radio or cell phone. Never go boating alone.

- Unless electrofishing is being performed, do not wear waders and hip boots in a boat because they could be a safety hazard if the boat should tip or a person is thrown out.

- While functioning in an official capacity, staff (and DEQ-sponsored guests) shall wear approved Personal Flotation Devices (PFDs) while aboard boats on any body of water.

- Report any collision, accident, or other incident that results in death or injury to any person or property damage involving a state vehicle, to the state police and the Virginia Management Control Center. If emergency services are needed, call 911 or the State Police before contacting VMCC. Accident reporting materials are found in the glove box of all fleet automobiles. If an incident involving injury, death, or property damage greater than $500 while operating a boat, contact the Virgina Game and Inland Fisheries.

  The driver involved in the accident MUST contact the VMCC. A State Police officer must investigate all accidents involving a state vehicle, regardless of the amount of damage. The employee involved in an accident must complete all accident forms and notify their supervisor, HRO, DEQ's Office of General Service, and the Commonwealth's insurance company.

For convenience, numbers for State Police Division Headquarters are listed below:

Location	Toll Free Telephone	Local Telephone
Division 1- Richmond	1-800-552-9965	804-553-3444
Division 2- Culpeper	1-800-572-2260	540-829-7401
Division 3- Appomattox	1-800-552-0962	434-352-7128
Division 4- Wytheville	1-800-542-8716	276-228-3131
Division 5- Chesapeake Melfa Norfolk	1-800-582-8350	757-424-6820 757-787-5813 757-455-3983
Division 6- Salem	1-800-646-1273	540-375-9500
Division 7- Fairfax	1-800-572-4510	703-323-4500

Table 7.2 Boat Safety Float Plan

Virginia Department of Environmental Quality Boat Safety Float Plan

1. Region: _____ Date: _____ Purpose of Trip: _____

2. Name of Boat Operator: _____

3. Description of Boat: _____

   Type: _____ Color: _____ Trim: _____

   Registration Number: _____ Length: _____

   Name: _____ Make: _____ Other: _____

4. Engine Type: _____ Horsepower: _____

   Number of Engines: _____ Fuel Capacity: _____

5. Safety Equipment: ☐ PFDs ☐ Paddles ☐ Anchor

   ☐ Smoke Signals ☐ Drinking Water ☐ Food

   ☐ ANSI Safety Garments ☐ Other: _____

6. Radio: ☐ Yes / ☐ No Type: _____ Frequency: _____

   Cell Phone: ( ) - _____

7. Tow Vehicle License Number: _____ State Registered: ☐ Yes / ☐ No

   Make and Model of Vehicle: _____ Color: _____

   Trailer License: _____ Where Parked: _____

8. Persons on Board:

Name:	Age:	Address & Telephone Number:

9. Trip Leaving at: _____ ☐ AM/ ☐ PM Leaving From: _____

   Returning at: _____ ☐ AM/ ☐ PM If not returned by: _____ ☐ AM/ ☐ PM

   Call USCG: ( ) - Police: ( ) -

Table 7.3 Boating-Safety Checklist

TRAILER	**YES**	**NO**	**COMMENTS:**
Sizes of coupler and ball hitch match			
Tire pressures are at the maximum noted on the rim			
Tire treads are at least $^{3}/_{32}$"			
Tires are in good condition			Show no bulging, cracking, or tread separation
Brake lights and turn signals function			
Safety chains are attached in an *X* under the coupling			
All boat straps are tight			
License plate is present and firmly attached			
Trailer stand is secure			
BOAT	**YES**	**NO**	**COMMENTS:**
Boat plugs are present			
Battery is charged			
Gas tank is full			
Anchor and rope are aboard			
Navigation lights operational			Lights are appropriate for boat size; no other lights that may be mistaken for navigation lights are exhibited
Emergency paddles are aboard			
First-aid kit is available			
First extinguisher is charged and accessible			
Flashlight with working batteries is available			
An air horn or whistle is aboard			
Rain gear is aboard			
Personal flotation devices are available for every person on board			
The emergency kill switch for the boat motor is functioning			
Radio or cell phone is available and functioning			

7.6.1. Personal Flotation Devices

Approximately 90 percent of all boating fatalities are from drowning. Virtually all drowning victims are not wearing personal flotation devices (PFDs), or are wearing inadequate ones. All boats must be equipped with life jackets or PFDs approved by the U. S. Coast Guard (Table 7.4). The quantity and type depend on the length of the boat and the number of persons aboard.

Follow these guidelines:

- PFDs must be in good condition. Regularly test the buoyancy in shallow water.
- Inspect the PFDs for weakened material or insecure snaps or zippers.
- Replace spent cartridges in inflatable PFDs or tag used cartridges as out of service, so they are not used accidentally.
- Ensure all PFDs are the proper size for the intended wearer. Read the label to ensure that it is the right size for a person's weight and chest size.
- Keep all PFDs readily accessible.
- Make sure all sampling personnel wear PFDs when in boats and when wading deep or fast flowing water.
- For boats 16 feet long or longer, keep an extra Type IV PFD, in addition to those required for passengers, immediately available.
- Select PFDs that are appropriate for the area being sampled.

Table 7.4 Types of Personal Flotation Devices

Type	Conditions of Use	Positives	Negatives
I	Offshore work or remote areas where rescue may take a while.	Excellent for flotation and will turn most unconscious persons face up in the water.	None
II	Near-shore vests.	Good for calm waters and fast rescues.	Lacks the capacity to turn wearers face up.
III*	Vests or flotation aids.	Good for calm waters and fast rescues.	Will not turn an unconscious person face up and should not be used in rough waters.
IV	Throwable devices, cushions or buoy rings.	Designed to be thrown to someone in trouble.	Not good for long hours in the water, rough water, nonswimmers, or the unconscious.
V*	Type V, or special-use devices, are designed for specific activities. They are only appropriate for use in accordance with the specific instructions on the label of the device.		

*Some Type III and Type V PFDs are designed to inflate when the wearer enters the water. This type must be worn when under way to be acceptable.

7.7. Collecting Fish

7.7.1. Electrofishing

Electrofishing is hazardous work. The batteries and generators used provide more than enough current to electrocute a person. **Use extreme caution and never electrofish alone.** Ensure that everyone associated with the electrofishing efforts is aware of the hazards and safety requirements before beginning the project.

Below is a general list of safety items to be aware of during electrofishing. Additional items specific to electrofishing can be found in section 4.2 of the *Quality Assurance/Quality Control Project Plan for the Fish Tissue and Sediment Monitoring Program* available at http://www.deq.virginia.gov/export/sites/default/fishtissue/pdf/fishsop1998.pdf

- Be familiar with the equipment and inspect it before each use. Correct any equipment problems immediately. If equipment must wait to be repaired, tag it "Out of Service" so it won't be used accidentally.

- Evaluate the equipment annually during a preventive maintenance inspection.

- There should be no wiring splices in the equipment. If connections are necessary, ensure that the rating of the connector is the same or greater than that of the wire. Ensure all junction boxes are weatherproof or rain tight, depending on the use. Junction boxes with switching equipment must be weatherproof.

- Ensure that gel type batteries are used on backpack electrofishing units.

- Check hip and shoulder straps to make sure they are of the quick-release type, are not damaged, and are long enough for the person who will use them.

- Ensure that the backpack unit is equipped with a trip switch that breaks the circuit if the user falls. The switch must require a manual reset before reestablishing the circuit.

- Electrofishing should involve at least three people so that one person can administer CPR while the other seeks medical assistance. At least two members of the crew must have valid certifications in CPR and First Aid.

- Make sure all personnel wear a Coast Guard–approved PFD and rubber gloves rated for a voltage above that used by the electrofishing unit when operating in a boat. Hip or chest waders are required when electrofishing by boat.

- When backpack electrofishing, wear waders and rubber gloves rated for a voltage above that used by the electrofishing unit.

- Inspect all nets to ensure they are made of nonconductive material and that they are long enough to keep the user's hands out of the water.

7.7.2. Handling Fish

Do not pull fish out of the water with bare hands while electrofishing. Use nets or turn off the generator before reaching in the water. Take care when working with catfish or other fish with barbs. Barbs are usually located on the pectoral or dorsal fins and punctures from them can be very painful and lead to infection.

7.8. Contaminated Water

Always consider the possibility that the water being sampled may be contaminated with pathogens or hazardous chemicals. Use caution and extra protection when working in or around water with known or suspected contamination such as sewer overflows or an unusual odor or color in the water. Noting suspected contamination on sample tags will alert the laboratory to use appropriate precautions.

Waterborne, disease-causing organisms (pathogens) are found in nearly all surface water systems. Pathogens enter surface water through untreated sewage discharges and bypasses, storm and agricultural runoff, and direct contact. Bacteria, viruses, and other pathogens can occur in the most pristine environments. Never drink untreated water, no matter how pristine the environment appears. Regular use of antibacterial soap or hand cleaner carried in the vehicle or in the backpack greatly reduces the risk of infection.

7.9. Weather

Weather can change rapidly and create unexpected situations for sampling personnel, whether they are in a boat or in isolated sampling areas. Check local weather forecasts before going out into the field. While in the field, be alert to visual weather cues, such as developing clouds, wind shifts, and graying skies.

If you see these signs while in a boat, head for shore immediately. When encountering rough water, head the bow into the waves at a 45° angle and reduce speed while still maintaining headway. Seat passengers in the bottom of the boat, as close to the center line as possible.

Leave small creeks and rivers to avoid flash floods. Avoid using low-water crossings, as the integrity of the underlying roadway is uncertain. Floating debris may damage the vehicle, or even push it from the roadway.

7.9.1. Lightning Safety

When you first see lightning or hear thunder, seek shelter in a vehicle with the windows closed, or in a substantial building. If in a boat, immediately make way to the nearest dock and take shelter in a vehicle or building. Never stay in the boat during a storm. Avoid high ground, water, and open spaces. Unsafe shelter includes canopies, small picnic or rain shelters, or near trees. Activities should be suspended till 30 minutes after the last observed lightning or thunder.

7.9.2. Temperature Exposure

The ideal comfort range for humans is 10–32°C (60– 90°F). Hypothermia (cold) and hyperthermia (heat) usually occur outside this range.

7.9.2.1. Cold Emergencies

Hypothermia is a condition of reduced body temperature caused by exposure to cold, and aggravated by wet clothes, wind, hunger and exhaustion. Hypothermia can occur with air temperatures above 16°C (60°F) under wet or windy conditions.

7.9.2.1.1. Warning Signs

Symptoms of hypothermia include uncontrollable fits of shivering, incoherence, listlessness, fumbling hands, frequent stumbling, drowsiness, and the inability to get up after resting.

7.9.2.1.2. Treatment

Remove the victim from the cold and into a dry, warm place. Take the following temporary measures until medical help is available: Replace wet clothes with dry ones. Warm the body slowly. Give warm, nonalcoholic drinks.

7.9.2.1.3. Prevention

The best way to prevent hypothermia is to stay warm and dry. Put on rain gear before it rains. Dress in layers and add more before getting cold. Find shelter before conditions become severe. During colder weather, carry a complete change of dry clothes.

7.9.2.2. Heat Emergencies

Hyperthermia is caused by increasing body temperature due to exposure to extreme heat. Heat emergencies can be brought about by a combination of factors: physical exertion, clothing (e.g. waders), humidity, no breeze, air temperature, and the rate of fluid intake. Working in the extreme summer heat creates a very real threat of heat-related stress.

7.9.2.2.1. Warning Signs

Symptoms of hyperthermia include chilling, headache, unsteadiness, dizziness, nausea, dry skin (either hot and red, which indicates heatstroke, or cool and pale, which indicates heat exhaustion), rapid pulse, and muscle pain and spasms.

7.9.2.2.2. Treatment

General treatment for heat emergencies involves cooling down and drinking plenty of fluids. **Do not give salt tablets**. A common symptom of dehydration is a headache. Heat stroke is life threatening. Quickly cool down heat stroke victims and watch for signs of shock. Call 911 or, if in an isolated area, transport the victim to a hospital immediately.

7.9.2.2.3. Prevention

- Hydrate well before working outdoors. Drink water in moderate amounts every fifteen minutes. Do not rely on thirst to indicate dehydration.
- Do not drink sodas or caffeinated drinks. These liquids can increase dehydration.
- Wear lightweight, light-colored clothing and a wide-brimmed hat.
- Schedule the most strenuous activities during the early morning or late afternoon.
- Find some shade and take breaks during the day.

7.10. Hazardous Plants and Animals

Certain insects, reptiles, and plants are always potential hazards for field personnel (Tables 7.5 to 7.7). The following is a summary of general information on the most common plant and animal hazards encountered by field staff (ARC 1988, Lane and Fay 1997, Milne and Milne 1980, Behler and King 1979).

Table 7.5 Potential Animal and Plant Hazards: Spiders and Other Arthropods

Animal	Characteristics and Habitat	Procedure (ARC 1988; Lane and Fay 1997)
Black widow spider	Female (only one that bites) is black with almost spherical abdomen, with a red hourglass mark (or two transverse red marks separated by black) on underside of abdomen Inhabits fallen branches and under objects.	✓ Take care when reaching into small, dark spaces. ✓ If bitten by a black widow, seek medical attention as soon as possible.
Brown recluse spider	Orange-yellow thorax with dark violin pattern. Bases of legs orange-yellow, rest of legs grayish to dark brown. Abdomen grayish to dark brown. Prefers dark spaces. Found outdoors in sheltered corners, among loose debris, indoors on the floor and behind furniture.	✓ Take care when reaching into small dark spaces. ✓ If bitten by a brown recluse, seek medical attention as soon as possible.
Ticks	Small, usually less than 3 mm (< $^1/_8$ in) long. Clamp to hosts using a dart-like anchor located just below the mouth. Found in wooded areas and tall grass.	✓ Wear long pants and tuck pants legs into socks and use a repellent containing DEET (N, N-diethyl-*meta*-toluamide). ✓ Check for ticks during and after field work. ✓ Remove with tweezers within 24 hours. ✓ Wash and disinfect the bite.
Bees	Vary in size from 2 mm (0.08 in) to 4 cm (1.6 in) long. Locations vary from ground nests to trees and human-built structures.	✓ Avoid beehives and wasp nests ✓ Scrape off the stinger with a knife or other flat object (e.g., a credit card) ✓ Wash area with soap and water ✓ Use a cold pack to reduce swelling ✓ Use an over-the-counter sting ointment or solution of water and baking soda ✓ Members of a field team who are allergic to insect bites or stings should notify the rest of the team and carry a sting kit for use in emergencies. ✓ Symptoms of an allergic reaction include pain, swelling of the throat, redness or discoloration in the area of the sting, itching, hives, decreased consciousness, or difficult or noisy breathing.
Wasps	Vary in size from minute to 5 cm (2 in) long. Adults have a narrow waist between the first and second abdominal segments. Locations vary from ground nests to trees and manmade structures.	

Table 7.6 Potential Animal and Plant Hazards: Snakes

Animal	Characteristics and Habitat	Procedure (ARC 1988; Lane and Fay 1997)
Cottonmouth (water moccasin)	A dark, heavy-bodied water snake. Broad-based head noticeably wider than neck. Olive, brown, or black above, patternless or with jagged-edged dark crossbands. Top of head is flat; eyes not visible from directly above, as in other harmless water snakes. Unlike other water snakes, it swims with head well out of water. Rarely found far from water. Most active at night, although may be seen sunning during the day. Found in lowland swamps, lakes, rivers, bayheads, irrigation ditches, canals, rice fields, to small, clear mountain streams. Bite more serious than that of a copperhead and can be fatal. When disturbed, tend to stand its ground, exposing the light **cottony** lining of its mouth.	Do not disturb. If bitten, **do:** ✚ Reassure the victim. ✚ Treat for shock. Keep victim lying down; elevate feet 10 to 12 inches. ✚ Seek medical attention as soon as possible. **Do not:** ✖ Cut or suck the bite area. ✖ Apply ice or a tourniquet. ✖ Leave victim unattended.
Copperhead	Stout body, copper, orange, or pink tinged with bold chestnut or reddish brown crossbands narrowing in the middle of the back. Top of head unmarked. Found in wooded hillsides with rock outcrops above streams or ponds, edges of swamps and periodically flooded coastal plains. Seen basking during fall and winter months, but more nocturnal during warm weather. Favorite warm weather habitats include stone walls, piles of debris, rotting logs, and large flat stones near streams. Copperhead bites are painful but rarely life threatening.	Best defense is to avoid them. Most snakes will go the other way unless unusually agitated or disturbed. Take care when electrofishing and seining near logjams, fallen trees, and undercut banks.
Rattlesnakes	Heavy-bodied with head distinctly wider than the neck. Most have blotches or crossband patterns on the back. Rattlesnakes have a distinctive rattle on the tail. Found mostly in the mountains of Virginia and sparsely vegetated rocky foothills. Is capable of delivering a fatal bite. When disturbed it normally stands its ground, lifting its head well above the coils. The warning is a buzzing sound.	Do not disturb. Slowly back away if the rattlesnake is sounding a warning. If bitten, **do:** ✚ Reassure the victim ✚ Treat for shock. Keep victim lying down; elevate feet 10 to 12 inches. ✚ Seek medical attention as soon as possible. **Do not:** ✖ Cut or suck the bite area. ✖ Apply ice or a tourniquet. ✖ Leave victim unattended.

Table 7.7 Potential Plant and Animal Hazards: Plants

Plant	Characteristics and Habitat	Procedure (ARC 1988; Lane and Fay 1997)
Poison ivy	Climbing poison ivy has alternate, trifoliate leaves with aerial roots that grow straight and are fuzzy. Found in most environments. Nonclimbing poison ivy lacks aerial roots. The leaves are the same shape as the climbing poison ivy, but are larger and broader. Vines without leaves can still cause a case of poison ivy. If a piece of vine is used as firewood, the oily resins can be released into the air. The resin can also remain on unwashed clothing and equipment.	Consider using pre-exposure lotion, which creates a barrier against poison ivy, oak, and sumac oils. After skin contact, **do:** ✚ Flood the affected area with lots of cold water as soon as possible. Since the oily resin is only slightly soluble in water, a little water will only spread the poison. ✚ Use anti-itch cream. Persons allergic to poison ivy may require medical attention. ✚ Use poison oak and ivy cleansers are available that can be used up to 8 hours after exposure. **Do not:** ✖ Use hot water or soap. This will increase the effects of poison ivy.

7.11. References

1. Texas Commission of Environmental Quality Surface Water Quality Monitoring Procedures. Volume 1: Physical and Chemical Monitoring Methods for Water, Sediment and Tissue Chapter 11. http://www.tceq.state.tx.us/comm_exec/forms_pubs/pubs/rg/rg-415/

2. VADEQ Agency Policy Statement 7-2009. http://deqnet/documents/index.asp?path=%2Fdocs%2Fadmin%2Fadmin%5Fpolicy/Safety

3. VADEQ QAPP for the Fish Tissue and Sediment Monitoring Program (1998). www.deq.virginia.gov/export/sites/default/fishtissue/pdf/fishsop1998.pdf

Appendix A: Calibration Logsheet for Multiprobes

VADEQ Calibration Log Display #: _____ Sonde #: _____ Region:_____

	Calibration / EoD Check	Calibration / EoD Check	Calibration / EoD Check
Date	/	/	/
Time	/	/	/
Initials	/	/	/
Run ID			
Specific Conductance			
Cond. Standard (µS/cm)	/	/	/
Cond. Reading (µS/cm)	/	/	/
Calibrate to (µS/cm)			
Difference (±1% / ±10%)	/	/	/
Cond. Cell constant*			
Acceptable Cell Constant: YSI 4.55 to 5.45. In-Situ- 0.33 to 0.39 (3-50,000 uS/cm) 4.4 to 5.8 (70 to 200,000 uS/cm)			
Dissolved Oxygen			
Temp Lab (°C)			/
Temp Probe (°C)	/	/	/
Barometric Pressure	/	/	/
Initial DO (mg/L)	/	/	/
Chart DO (mg/L)	/	/	/
Cal DO (mg/L)			
Diff (≤ 0.2 / < 0.5 mg/L)	/	/	/
DO Charge (YSI ROx)*			
DO Gain or Scale*			
Acceptable ranges: YSI ROx Charge 25 to 75. YSI ROx Gain (-0.7 to +1.5). Hydrolab Scale Factor (0.5 to 1.5)			
pH			
7 pH Initial pH (SU)	/	/	/
Calibrate to (SU)			
Difference (± 0.2 SU)	/	/	/
pH mV*			
4 or 10 Initial pH (SU)	/	/	/
Calibrate to (SU)			
Difference (± 0.2 SU)	/	/	/
pH mV*			
3rd pH check pH (SU)	/	/	/
Difference (± 0.2 SU)	/	/	/
pH mV*			
Acceptable ranges: YSI: 4 (130 to 230mV) 7 (-50 to 50mV) 10 (-230 to -130mV). In-Situ : 7 (-20 to 20 mV)			
Comments:			

*These values should be recorded after calibration.

Multiprobe Calibration and EoD Check Form Instructions

This calibration log sheet is designed to work with most multiprobe units such as YSI and Hydrolab series units. If the probe being used does not display the same fields as referenced in this document, please contact the Quality Assurance Coordinator.

It is important to follow the directions stated by the probe manufacturer or contained in Chapter 5 of this SOP, and in this document to get the most accurate probe readings. If there are any questions about using a particular probe, please contact the Quality Assurance Coordinator.

Before calibrating or post calibrate checks, be sure the unit has reached a stable temperature. A stable temperature is when the unit changes less than 0.1^{o}C in 10 seconds.

General Fields

Display #: Serial number or DEQ tag number of handheld display
Sonde #: Serial number or DEQ tag number of sonde
Region: Regional office

Probe Calibration Fields

The column identified as **Calibration** is filled prior to going into the field to collect readings.

- **Date:** Record the date and time of calibration. Calibration should be done each day field samples are collected.
- **Time:** Record time the calibration process begins. Using military time will avoid am/pm errors.
- **Initials:** Initials of the person performing the calibration.
- **Run ID:** Run ID(s) the unit is being used for that day.

Specific Conductance-

- **Cond. Standard (uS/cm):** Strength of conductivity standard used in uS/cm
- **Cond. Reading (uS/cm):** Stable reading of unit immersed in the standard
- **Calibrate to (uS/cm):** After the unit is calibrated, the value displayed on the handheld
- **Difference (+/-**1%): Difference of cal vs. standard value. Readings should be 1% or less.
  (Calibrated value) – (Standard used) / (Standard used) x 100%.
  Example: 1231 uS/cm calibrated reading – 1214 uS/cm standard = 17 uS/cm difference
  (17 uS/cm difference / 1214 standard) x 100% = 1.4%
- **Cond Cell constant**: Value obtained following manufacturer instructions. Table contains acceptable constants.

Dissolved Oxygen-

- **Temp Lab C:** Temperature of the lab taken before beginning to calibrate the DO probe. Reading is from a laboratory thermometer which was verified to the DEQ master thermometer within one year. Check is performed once every three calibration sessions.

- **Temp Probe C:** Temperature of the probe while calibrating. Do not begin the calibration of the probe until the unit displays a stable temperature (≤0.1°C change in ten seconds).
- **Barometric Pressure:** Record the barometric pressure of the master certified barometer. Enter the units used for the pressure reading (mmHg, inHg, or mbar). If a certified barometer is not available, you may also use local weather station data available on www.wunderground.com or www.noaa.gov*.

***Note:** Refer to the *Dissolved Oxygen Calibration Sheet* available in Appendix B of the WQM SOP manual for additional information to use weather station barometric pressure data.

- **Initial DO mg/L: E**nter the stable dissolved oxygen value (≤0.1 mg/L change in ten seconds) prior to calibrating the unit.
- **Chart DO mg/L:** Prior to calibrating your probe, you should determine the theoretical DO value to confirm your probes readout. To determine the theoretical value, refer to the *Dissolved Oxygen Calibration Sheet* available in Appendix B of the WQM SOP manual.
- **Cal DO mg/L:** Record the mg/L reading after calibrating the DO sensor.
- **Diff (≤ 0.2 / <0.5 mg/L):** If using an optical DO sensor, the probe should now read within 0.1 mg/L of the Chart DO reading. Clark cells should be within 0.2 mg/L.
- **DO Charge (YSI ROx):** If using the YSI ROx optical DO sensor record the voltage of the unit. Exceeding the manufacturer stated range will indicate the probe may need a new sensing membrane or servicing.
- **DO Gain or Scale:** An additional field to help diagnose calibration issues with optical DO sensors. If the DO gain or scale is outside of manufacturer values, the probe may need a new membrane or servicing.

pH-

- **7 pH**- Rinse and place the probe in 7.00 buffer and allow it to equalize before calibrating.
- **Initial pH (SU):** Once the probe is equalized, record the reading prior to calibration.
- **Calibrate to (SU):** Calibrate and record the returned value. Most buffer solutions provide a table to ensure that a properly calibrated probe is recording the correct pH. Round up to the next tenth digit (example 7.11 should be written as 7.2)
- **Difference (±0.2 S.U.):** After calibration of the probe, the readings should be within 0.2 S.U. of the buffer value.
- **pH mV:** YSI and In-Situ can report the millivoltes of the pH probe. Record this reading after you calibrate the probes and the reading should be within the manufacturer specified values as noted on the sheet.
- **pH 4 and pH 10**- Repeat the above process used to calibrate the probe with 4.00 or 10.01 buffer and record the values in correct column.
- **3rd pH check pH (S.U.):** If the field team anticipates they will encounter pH values outside their calibrated buffer range, immerse the probe in a third standard (either 4 or 10 buffer) to verify the unit is accurate.

Comments-

- **Comments**- A space provided for any notes or comments regarding the probe such as maintenance or membrane changes.

End of Day (EOD) Check

The column identified as **EoD Check** is filled out after returning from the field and performing an end of day check. This is a necessary quality assurance step to verify that the probe has not drifted outside acceptable values during the course of sampling. The calibration check consists of placing the probe in the appropriate buffer/standard and letting it equalize. This may take up to 30 minutes depending on the condition of the unit.

Probe EoD Check Fields

- **Date:** Record the date and time of the EoD check. This should be done each day field samples are collected.
- **Time:** Record time the calibration process begins. Using military time will avoid am/pm errors.
- **Initials:** Initials of the person performing the EoD check.

Specific Conductance-

- **Cond. Standard (uS/cm):** Strength of conductivity standard used in uS/cm
- **Cond. Reading (uS/cm):** Stable reading of unit immersed in the standard
- **Difference (+/-10**%): Difference of the probe reading vs. standard value. Readings should be 10% or less.

  (Probe reading) – (Standard used) / (Standard used) x 100%.

  Example: 1260 uS/cm calibrated reading – 1214 uS/cm standard = 46 uS/cm difference
  (46 uS/cm difference / 1214 standard) x 100% = 3.8%

Dissolved Oxygen-

- **Temperature C:** Temperature of the probe while performing the EoD Check. Wait until the unit displays a stable temperature (≤0.1 C change in ten seconds).
- **Barometric Pressure:** Record the barometric pressure of the master certified barometer. Enter the units used for the pressure reading (mmHg, inHg, or mbar). If a certified barometer is not available, you may also use local weather station data available on www.wunderground.com or www.noaa.gov*.

***Note: *Note:** Refer to the *Dissolved Oxygen Calibration Sheet* available in Appendix B of the WQM SOP manual for additional information to use weather station barometric pressure data.

- **Initial DO mg/L: E**nter the stable dissolved oxygen value (≤0.1 mg/L change in ten seconds) prior to calibrating the unit.
- **Chart DO mg/L:** Prior to calibrating your probe, you should determine the theoretical DO value to confirm your probes readout. To determine the theoretical value, refer to the *Dissolved Oxygen Calibration Sheet* available in Appendix B of the WQM SOP manual.
- **Diff (<0.5 mg/L):** Do not enter dissolved oxygen data in CEDS if the difference between the probe DO reading and theoretical DO is 0.5 mg/L or greater. Service optical DO probes if the difference is greater than 0.2 mg/l. Service Clark DO probes if the difference is greater than 0.3 mg/L.

pH-

- **7 pH**- Rinse and place the probe in 7.00 buffer and allow it to equalize before calibrating.
- **Initial pH (SU):** Once the probe is equalized, record the reading prior to calibration.
- **Difference (±0.2 S.U.):** After the probe stabilizes, the reading should be within 0.2 S.U. of the buffer value.
- **pH 4 and pH 10**- Repeat the above process used to calibrate the probe with 4.00 or 10.01 buffer and record the values in correct column.
- **3rd pH check pH (S.U.):** If the field team encountered values outside of the range they calibrated the unit at (example pH 7.3 when unit was calibrated with 4 and 7 buffer), immerse the probe in third standard (in this case 10 buffer) to verify the unit is accurate.

Comments-

- **Comments**- A space provided for any notes or comments regarding the probe such as maintenance or membrane changes.

Appendix B: Theoretical Dissolved Oxygen Chart

How to Calculate Theoretical Dissolved Oxygen Values

Proper calibration of Dissolved Oxygen (DO) probes is important to collect accurate data. An easy way to see if a probe is calibrated correctly is to compare the probe's results against the theoretical DO value. This value is what the DO level should be according to temperature and barometric pressure.

DO Level Based on Temperature

The top table on the attached chart allows users to find the DO level based on temperature. The top and side axis of the table corresponds to the temperature that the probe is reporting. The intersection of the two axes displays the DO reading. Write this number down to start calculating the theoretical DO level.

Correction Factor for Barometric Pressure

Barometric pressure measures how much atmosphere is pressing down on a surface. Weather systems and elevation above (or below) sea level can change this value. The bottom table of the attached chart will compensate for these changes in pressure. Dissolved oxygen probes normally show pressure in millimeters of mercury (**mmHg**) or millibars (**mbar**).

Having a barometer on hand is a good way to get pressure data. A weather station can also provide this information. Websites such as www.wunderground.com are useful to find nearby stations. Please note that most barometers and weather stations report pressure in inches of mercury (**inHg**).

Note: Using Weather Station Barometric Pressure Readings

Weather stations standardize barometric pressure readings to make it appear as if the station was at sea level. To account for this, subtract the barometric pressure by 1.01 inHg per 1,000 feet in elevation of the weather station. This final value is known as **absolute barometric pressure (ABP)**.

Example: Find the absolute barometric pressure of a station located 222 feet above sea level that reported 30.12 inHg.

$$30.12 \text{ inHg} - \frac{1.01 \text{ inHg}}{1000/\ 222 \text{ feet}} \rightarrow 30.12 - \frac{1.01}{4.50} \rightarrow 30.12 - 0.22 = 29.90 \text{ inHg ABP}$$

Once finding the local pressure, use the bottom table to find the proper correction factor to use. The chart includes mmHg, inHg, and mbar to determine the pressure correction factor using different types of barometer readouts.

Example: A barometric pressure of 730 mmHg you would use a correction factor of 0.96 (second column, forth row down).

Theoretical DO Calculation

To find the theoretical DO value, use the following formula.

Theoretical DO = (DO level based on temperature) x (barometric pressure correction factor)

Example: If a probe had a temperature of 18.4 C and the barometric pressure was 730 mmHg, the theoretical DO value would be 9.00 mg/L (9.37 mg/L x 0.96 correction factor)

Dissolved Oxygen Calibration Sheet

Directions: Multiply the O2 concentration (top chart) by the pressure correction factor (bottom chart).

Temp in ^{O}C	O_2 concentrations in mg/l									
	.0	.1	.2	.3	.4	.5	.6	.7	.8	.9
0	**14.59**	**14.55**	**14.51**	**14.46**	**14.42**	**14.38**	**14.34**	**14.30**	**14.26**	**14.22**
1	**14.18**	**14.15**	**14.11**	**14.07**	**14.03**	**13.99**	**13.95**	**13.91**	**13.88**	**13.84**
2	**13.80**	**13.76**	**13.72**	**13.69**	**13.65**	**13.61**	**13.58**	**13.54**	**13.50**	**13.47**
3	**13.43**	**13.40**	**13.36**	**13.32**	**13.29**	**13.25**	**13.22**	**13.18**	**13.15**	**13.12**
4	**13.08**	**13.05**	**13.01**	**12.98**	**12.94**	**12.91**	**12.88**	**12.84**	**12.81**	**12.78**
5	**12.75**	**12.71**	**12.68**	**12.65**	**12.61**	**12.58**	**12.55**	**12.52**	**12.48**	**12.45**
6	**12.42**	**12.39**	**12.36**	**12.32**	**12.29**	**12.26**	**12.23**	**12.20**	**12.17**	**12.14**
7	**12.11**	**12.08**	**12.05**	**12.02**	**11.99**	**11.96**	**11.93**	**11.90**	**11.87**	**11.84**
8	**11.81**	**11.78**	**11.76**	**11.72**	**11.69**	**11.67**	**11.64**	**11.61**	**11.58**	**11.55**
9	**11.53**	**11.50**	**11.47**	**11.44**	**11.42**	**11.39**	**11.36**	**11.33**	**11.31**	**11.28**
10	**11.25**	**11.23**	**11.20**	**11.18**	**11.15**	**11.12**	**11.10**	**11.07**	**11.05**	**11.02**
11	**10.99**	**10.97**	**10.94**	**10.92**	**10.89**	**10.87**	**10.84**	**10.82**	**10.79**	**10.77**
12	**10.75**	**10.72**	**10.70**	**10.67**	**10.65**	**10.63**	**10.60**	**10.58**	**10.55**	**10.53**
13	**10.51**	**10.48**	**10.46**	**10.44**	**10.41**	**10.39**	**10.37**	**10.35**	**10.32**	**10.30**
14	**10.28**	**10.26**	**10.23**	**10.21**	**10.19**	**10.17**	**10.15**	**10.12**	**10.10**	**10.08**
15	**10.06**	**10.04**	**10.02**	**9.99**	**9.97**	**9.95**	**9.93**	**9.91**	**9.89**	**9.87**
16	**9.85**	**9.83**	**9.81**	**9.79**	**9.76**	**9.74**	**9.72**	**9.70**	**9.68**	**9.66**
17	**9.64**	**9.62**	**9.60**	**9.58**	**9.56**	**9.54**	**9.53**	**9.51**	**9.49**	**9.47**
18	**9.45**	**9.43**	**9.41**	**9.39**	**9.37**	**9.35**	**9.33**	**9.31**	**9.30**	**9.28**
19	**9.26**	**9.24**	**9.22**	**9.20**	**9.19**	**9.17**	**9.15**	**9.13**	**9.11**	**9.09**
20	**9.08**	**9.06**	**9.04**	**9.02**	**9.01**	**8.99**	**8.97**	**8.95**	**8.94**	**8.92**
21	**8.90**	**8.88**	**8.87**	**8.85**	**8.83**	**8.82**	**8.80**	**8.78**	**8.76**	**8.75**
22	**8.73**	**8.71**	**8.70**	**8.68**	**8.66**	**8.65**	**8.63**	**8.62**	**8.60**	**8.58**
23	**8.57**	**8.55**	**8.53**	**8.52**	**8.50**	**8.49**	**8.47**	**8.46**	**8.44**	**8.42**
24	**8.41**	**8.39**	**8.38**	**8.36**	**8.35**	**8.33**	**8.32**	**8.30**	**8.28**	**8.27**
25	**8.25**	**8.24**	**8.22**	**8.21**	**8.19**	**8.18**	**8.16**	**8.15**	**8.14**	**8.12**
26	**8.11**	**8.09**	**8.08**	**8.06**	**8.05**	**8.03**	**8.02**	**8.00**	**7.99**	**7.98**
27	**7.96**	**7.95**	**7.93**	**7.92**	**7.90**	**7.89**	**7.88**	**7.86**	**7.85**	**7.83**
28	**7.82**	**7.81**	**7.79**	**7.78**	**7.77**	**7.75**	**7.74**	**7.73**	**7.71**	**7.70**
29	**7.69**	**7.67**	**7.66**	**7.65**	**7.63**	**7.62**	**7.61**	**7.59**	**7.58**	**7.57**
30	**7.55**	**7.54**	**7.53**	**7.51**	**7.50**	**7.49**	**7.48**	**7.46**	**7.45**	**7.44**

Barometric Pressure Correction factor:

Units	Pressure	Correction	Pressure	Correction	Pressure	Correction	Pressure	Correction
mmHg inHg mbar	775 – 771 30.53 – 30.33 1033 – 1028	**1.020**	745 – 741 29.35 – 29.15 994 – 988	**0.980**	715 – 711 28.16 – 27.97 954 – 948	**0.940**	685 – 681 26.98 – 26.79 914 – 908	**0.900**
mmHg inHg mbar	770 – 766 30.32 – 30.14 1027 – 1021	**1.014**	740 – 736 29.14 – 28.96 987 – 981	**0.973**	710 – 706 27.96 – 27.78 947 – 941	**0.934**	680 – 676 26.78 – 26.60 907 – 901	**0.893**
mmHg inHg mbar	765 – 761 30.13 – 29.94 1020 – 1015	**1.007**	735 – 731 28.95 – 28.76 980 – 975	**0.967**	705 – 701 27.77 – 27.58 940 – 935	**0.927**	675 – 671 26.59 – 26.40 900 – 895	**0.887**
mmHg inHg mbar	760 – 756 29.93 – 29.74 1014 – 1008	**1.000**	730 – 726 28.75 – 28.56 974 – 968	**0.960**	700 – 696 27.57 – 27.38 934 – 928	**0.920**	670 – 666 26.39 – 26.20 894 – 888	**0.880**
mmHg inHg mbar	755 – 751 29.73 – 29.55 1007 – 1001	**0.993**	725 – 721 28.55 – 28.37 967 – 961	**0.953**	695 – 691 27.37 – 27.19 927 – 921	**0.914**	665 – 661 26.19 – 26.00 887 – 881	**0.874**
mmHg inHg mbar	750 – 746 29.54 – 29.36 1000 – 995	**0.987**	720 – 716 28.36 – 28.17 960 – 955	**0.947**	690 – 686 27.18 – 26.99 920 – 915	**0.907**	660 – 656 25.99 – 25.81 880 – 875	**0.867**

Appendix C: Corrective Action Request Form

Corrective Action Request Form

Section I: to be completed by originator
Submitted by: _____ Date: _____

A. Nature of Problem:

B. Possible Cause:

C. Date of Problem Identified: _____

D. Samples That May Be Invalid:

E. Recommended Corrective Action (Optional):

Continued on next page

Corrective Action Request Form- Continued

Section II: to be completed by program manager
Name: _____ Date: _____

A. Recommended Corrective Action:

B. Follow Up Action Required:

C. Implementation Will Begin On:

Section III: to be completed by QA Officer
Name: _____ Date: _____

A. Recommended Corrective Action:

B. Follow Up Action Required:

C. Implementation Will Begin On:

Appendix D: Entering QA/QC into CEDS

Entering QA/QC data into CEDS

QA/QC Run IDs:
QA/QC run IDs consists of the first letter of the region conducting the sampling followed by the letters QA and 2-letter program code under which the samples are collected (e.g. TQAAQ for Tidewater regional QA run for Ambient Monitoring Program).

Blank/Duplicates designations:

Code	Designation
R	Regular sample (default designation)
EB	Equipment Blank
S1	First subsample of a field split sample (these data are stored in the regular run ID)
S2	Second subsample of a field split sample (these data are stored in the QA/QC run IDs)

Container number designations:

Container #	Designation
1 - 9	**S1 sample containers**
11 - 19	**S2 sample containers** **Note:** The ones place of a S2 container is the same as the corresponding S1 container e.g. if S1 for NUT4 is container number 2 then S2 for NUT4 is container number 12.
21 - 29	**Equipment blanks** **Note:** The ones place for equipment blanks also correspond to the S1 containers for the sample types e.g. S1 for NUT4 is 2 then EB for NUT4 is 22.

Lab Process code designations:

D - Indicates to the lab to perform lab splits on the sample
M - Indicates to the lab to perform matrix spikes on the sample

Yearly run schedule:

1. In CEDS, click on applications /environmental monitoring/water/yearly run schedule.
2. In the yearly run schedule, set up a generic QA/QC run ID (e.g.TQAAQ).
3. Use QA as the station ID and survey program.
4. Enter the lab Proc Code for NUT4 and TNTUL containers as M.
5. Enter the appropriate depths (0 for EB), container IDs, parameter group codes and save. Once this is completed, the QA/QC run ID can be used for all QA sampling events.

Monthly schedule:

1. Click on applications/environmental monitoring/water/monthly run schedule.
2. Click on the get yearly run data tab.

3. Query QA Station ID with appropriate sample collection date
4. Copy (press F4) the last line so you have a blank and S2 for each parameter. Fecal samples don't get blanks. For a regular AQ station you will have five more lines
5. For blanks, the 'Survey Program' is "QA" and depth is "0" and "%FRB=50"
6. Change Blank/Dup to S1, S2 and EB
7. Change Container ID to correspond to S1 number. S2=1, EB=2. All the same parameters should end in the same number
8. For 'Lab Proc Code' put an "M" for the S2 samples for (TPLL; TN; WAT; TNUTL; NUT4; BAYT; TOC; PROB3; and T2.
9. Click 'Change Parameter Group Codes'.
10. Save.
11. Enter the regular run ID and the date to be collected on the first line of the pop-up screen. On the next line enter the QA/QC run ID (e.g. TQAAQ and the date to be collected).
12. Click on the get yearly run data button. The database will be automatically updated with the runs chosen and will be displayed in the monthly run schedule screen. Save.
13. Click on the query button.
14. Enter the regular run ID, the station chosen for QA/QC and the date as scheduled in step 5.
15. Change the blank/dups designation for all containers to S1.
16. Save.
17. Click the query button.
18. Enter the QA/QC run ID (TQAAQ) and the date scheduled in step 3.
19. Change the station ID from QA to the name of the station chosen for QA/QC sampling for all samples.
20. Change the blank/dup designation for all containers numbered 11-16 to S2.
21. Change the blank/dup designation for all containers numbered 21-26 to EB.
22. Save the information and exit CEDS.

Appendix E: Dissolved Oxygen Using Modified Winkler Method

This procedure is applicable to the analysis of dissolved oxygen in fresh, estuarine and coastal water samples. The results are measured and reported as milligrams dissolved oxygen per liter of water.

Summary of method

The iodometric test is the most precise and reliable titrimetric procedure for DO analysis. It is based on the addition of divalent manganese solution, followed by a strong alkali, to the sample in a glass-stoppered bottle. Dissolved Oxygen (DO) rapidly oxidizes an equivalent amount of dispersed divalent manganous hydroxide precipitate to hydroxides of higher valence states. In the presence of iodide ions in an acidic solution, the oxidized manganese reverts to the divalent state, with the liberation of iodine equivalent to the original DO content. The iodine is then titrated with a standard solution of sodium thiosulfate or phenylarsine oxide (PAO).

Interference

Iron salts, organic matter, excessive suspended matter, sulfide, sulfur dioxide, residual chlorine, chromium, cyanide and certain oxidizing and reducing agents can effect the Azide modification of the Winkler titration method.

Apparatus and materials

Equipment

1. Digital Buret
2. 300 ml glass stopper BOD bottle
3. 250 ml wide-mouthed Erlenmeyer flasks
4. 200 ml volumetric flask
5. Stir bar and stir bar retriever (optional)
6. Magnetic stir plate (optional)

Chemicals

1. Hach Iodate-Iodide standard solution 0.00125 N (Catalog #40149)
2. Hach Manganese Sulfate powder pillow (Catalog #107166)
3. Hach Alkali-Iodide-Azide powder pillow (Catalog #107266)
4. Hach Sulfamic acid powder pillow (Catalog #107399)
5. Starch indicator solution (Catalog #34932)
6. Sodium Thiosulfate standard solution 0.025N (Catalog #2409353)

Sample bottle and equipment cleaning

- Sample containers must be clean, dry and free of contaminants. Using lab grade detergent, wash with a good cleaning brush and rinse thoroughly with tap water followed by analyte-free water. Allow the bottle to dry in an upside-down position.

- Clean all sample containers immediately after completing the test to prevent accumulation of residues in the containers, which will affect future samples.

Instrument or method calibration

The digital burette should be calibrated following the manufacturer's instructions.

Sample collection

Note: Winkler DO is normally taken to either verify DO probe accuracy in the field or to verify correct calibration of the probe in the laboratory.

Laboratory sample

Used to verify probe calibration accuracy and troubleshooting.

6. Using a clean 1/2 gallon wide mouth bottle or similar container, fill ¾ full with DI water. For most accurate results, fill with DI water stored in a carboy which is accumulated to laboratory temperature or allow the bottle to sit overnight before testing.

7. Close the container and vigorously shake for 2-5 minutes to add oxygen to the sample.

8. Open the container and slowly fill the BOD bottle to overflowing without introducing air bubbles. This is best achieved by tilting the BOD bottle 45° and filing down the side of the bottle.

9. Carefully pour the remaining water into a large beaker with a stir bar. Use a stir plate and stir bar or the probe stirrer to facilitate gentle mixing of the without forming air bubbles and lower the DO probe apparatus into the beaker to begin reading.

10. Proceed to step 1 of Handling and Preservation section for the filled BOD sample bottle.

Field sample

Used to verify field performance of DO probeware if a problem is suspected.

Collect the Winkler sample at the same time and place as the meter readings is taken. If the meter reading is taken on a bridge, take the sample from the bucket. If the meter reading is taken in-stream, collect the sample in-stream. In-stream samples are preferred.

1. Rinse the BOD bottle twice with sample water, discarding the rinse.

2. Fill the bottle by tilting the bottle and submerging it under the water surface at an angle. Avoid introducing air bubbles into the sample by slowly turning the bottle up until it is filled and in an upright position.

Handling and preservation

1. Immediately add 1 pillow of Manganese Chloride powder, followed by 1 pillow of alkali-iodide-azide powder. Allow the majority of the powder to settle below the neck of the bottle.

2. Place the glass stopper onto the sample bottle carefully to avoid introducing air bubbles. Twist the stopper to ensure a snug fit. There should be a small amount of water covering the top of the stopper.

3. Invert the sample bottle until the most of the chemicals appear to dissolve. If air bubble appears in the bottle, recollect the sample.

4. When the precipitate has settled to at least half the sample bottle volume (leaving clear supernatant above the manganese hydroxide floc), mix the sample again by inverting the bottle several times.

5. Once the precipitate has settled to half the sample bottle for the second time, add a pillow of Sulfamic Acid powder to the sample, place the stopper on the bottle and shake well until the precipitate has dissolved. Occasionally, a dark brown precipitate persists in the bottle after acidification. This precipitate will dissolve after continuing mixing for a few more minutes.

At this point, the sample is considered "fixed" and concern for additional oxygen being introduced into the sample is reduced. If transporting samples for testing, keep them away from direct sunlight. Fixed samples must be titrated within 8 hours.

Sample analysis

Titrant Strength Verification

Before titration (Ideally before collecting the sample), it is important to verify the strength of the titrant (sodium thiosulfate or PAO). Failure to perform this verification prior to titration may result in inaccurate results invalidating the test.

1. Add 200.0 ml of Iodate-Iodide Standard Solution, 0.00125 N, to an Erlenmeyer flask.

2. Add one Sulfamic Acid Powder Pillow and swirl to mix.

3. Prime the digital pipette before titrating to ensure there are no air bubbles in the pipette. Do this by drawing approximately 20 ml of 0.025N sodium thiosulfate (or PAO) into the digital pipette and then dispense all of solution into a beaker and discard. Next, pull approximately 20 ml of fresh titrant into the pipette for the analysis. Zero the digital pipette.

4. Titrate the sample with 0.025N Sodium Thiosulfate (or PAO) until the solution is a pale yellow (straw) color. Use a stir plate or gently swirl by hand after each titrant addition.

5. Add approximately 2 ml of starch indicator solution to turn the solution a blue color. Swirl to mix to a uniform color.

6. While continue to mix, slowly titrate with the thiosulfate (or PAO) until the blue color disappears. The titrated volume in ml is the concentration of dissolved oxygen in mg/L

If more less than 9.5 ml or more than 10.5 ml of titrant is used, check the cleanliness of the titrating glassware and retest. If you still use less than 9.5 ml or more than 10.5 ml, discard the titrant.

Titration of Sample

1. Prime the digital pipette before titrating to ensure there are no air bubbles in the pipette. Do this by drawing approximately 20 ml of 0.025N PAO into the digital pipette and then

dispense all of solution into a beaker. Next, pull approximately 20 ml of PAO into the pipette for the analysis. Zero the digital pipette.

2. Pour 200 ml of the sample from the BOD bottle into a 200 ml volumetric flask and transfer to an Erlenmeyer flask. Place the flask on the magnetic plate with stirrer in it and turn the stirrer on.

3. Prime the digital pipette before titrating to ensure there are no air bubbles in the pipette. Do this by drawing approximately 20 ml of 0.025N Sodium Thiosulfate (or PAO) into the digital pipette and then dispense all of solution into a beaker and discard. Next, pull approximately 20 ml of fresh titrant into the pipette for the analysis. Zero the digital pipette.

4. Titrate the sample with 0.025N Sodium Thiosulfate (or Phenylarsine Oxide) until the solution is a pale yellow (straw) color. Use a stir plate or gently swirl by hand after each titrant addition.

5. Add approximately 2 ml of starch indicator solution to turn the solution a blue color. Swirl to mix to a uniform color.

6. While continue to mix, slowly titrate with the thiosulfate (or PAO) until the blue color disappears. The titrated volume in ml is the concentration of dissolved oxygen in mg/L.

Data management and record management

All records must be maintained in the logsheet and keyed into CEDS.

Quality Control criteria

In the field, the difference between probe DO and Winkler DO should be within 0.6 mg/L. For laboratory checks, the difference between probe DO and Winkler DO should be within 0.2 mg/L.

Corrective Action

If the difference is ±0.6 mg/L or greater, most likely it is necessary to change the sensor membrane and electrolyte solution. Record the date of the membrane change on the log sheet. Collect a Winkler sample on the next sampling run using that probe. If the difference between Winkler and the probe is less than 0.6mg/l, the problem has been resolved.

Reference

Standard Methods for the Examination of Water and Wastewater, 18th edition.