

Appendix 5: Intensive Survey Branch SOP

Intensive Survey Branch Standard Operating Procedures Manual: Physical and Chemical Monitoring, version 2.1, December 2013

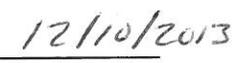
**INTENSIVE SURVEY BRANCH
STANDARD OPERATING PROCEDURES MANUAL:
PHYSICAL AND CHEMICAL MONITORING**

Version 2.1
December 2013

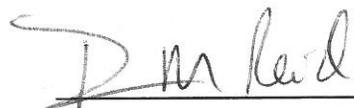
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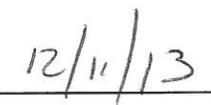
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DIVISION OF WATER RESOURCES
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INTRODUCTION

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

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

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

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

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

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INTENSIVE SURVEY BRANCH SOP REVISION LOG

Date	Version Edited	Chapter	Changes	Name
12-10-13	Ver 2.0	Cover page	Updated document date & version to Decemberr 2013 and Version 2.1	Jason Green
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08-19-08	Ver 8/2003	2 & 11	Put chapter 11 as a section in chapter 2	Danielle Mir

12-10-08	Ver 8/2003	Entire	Changed all Figure numbers	Danielle Mir
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I. CONSIDERATIONS FOR WATER QUALITY SAMPLING

The purpose for collecting water samples is to obtain a representative portion of the material or medium being evaluated. Valid results depend upon:

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

1. GENERAL WATER QUALITY SAMPLING CONSIDERATIONS

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

1.1. Selection of parameters to be measured

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

1.2. Dissolved and particulate sample fractions

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

1.3. Required sample volumes

The volume of sample obtained should be sufficient to perform all the required analyses with an additional amount collected to provide for any quality control needs such as split samples or repeat examinations. DWR Laboratory sample submitting guidance document can be found at: http://portal.ncdenr.org/c/document_library/get_file?uuid=92a278e5-f75a-4e42-9be5-282ac0216b2a&groupId=38364.

1.4. Sample handling

After collection, all samples should be handled as little as possible. All personnel should use extreme care to ensure that samples are not contaminated. If samples are placed in an ice chest, personnel should ensure that the ice does not submerge the sample containers, thereby preventing cross-contamination. This is extremely important, especially if the samples are to be used in an enforcement action. Alternatives that can be used to prevent contamination include the use of frozen water

containers instead of ice or double wrapping the sample containers in trash bags surrounded with ice.

1.5. Special precautions for sampling trace amounts of contaminants

Most contaminant compounds are detected in the range of parts per billion or parts per trillion; therefore, extreme care must be taken to prevent contamination of samples. The following precautions shall be taken when trace contaminants are of concern:

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

1.6. Procedures for identifying potentially hazardous samples.

- 1.6.1. Samples that are either **known** or **thought** to be **hazardous** should be identified **clearly** on both the sample tag and field sample sheet.
- 1.6.2. Information explaining the hazard, i.e., corrosive, flammable, poison, etc., shall also be listed.
- 1.6.3. If a sampling hazard is identified, only continue if a properly trained staff member is present and if appropriate safety equipment are available.
- 1.6.4. Follow procedures found on the ESS Fish Kill web page when sampling fish kill events: <http://portal.ncdenr.org/web/wq/ess/fishkills>

1.7. Collection of auxiliary data

All auxiliary data, such as flow measurements, photographs of sampling sites, meteorological conditions, and other observations, shall be entered into field records at the time samples are collected.

1.8. Time records

All records of time shall be kept utilizing local time in the military (2400 hour) time format and shall be recorded to the nearest five (5) minutes unless more precise measurements are dictated.

1.9. Transporting and shipping of samples

Samples may be hand delivered to the appropriate laboratory, or they may be shipped by common carrier. Chain of custody may be necessary during and after sample collection (Chapter II.3). All personnel must be aware that certain samples could be classified as hazardous materials and as such, could be regulated by the U.S. Department of Transportation under the Transportation Safety Act of 1974. These regulations are contained in Title 49, CFR, Parts II0-II9 (An example would be concentrated acid, azide, etc.). A copy of these regulations is available online at: <http://www.gpoaccess.gov/cfr/index.html>.

2. SURFACE WATER SAMPLE SITE SELECTION

Selection of a surface water sampling location for water quality studies is based on many factors. These include but are not limited to, study objective, water use, point source discharges, non point source discharges, tributaries, changes in stream characteristics, types of stream bed, stream depth, turbulence, presence of structures (weirs, dams), accessibility, safety concerns, and personnel. When such sampling locations are located in estuarine systems, tidal effects must be considered when determining sampling locations.

Before sampling is conducted, a site assessment should be conducted to locate suitable sampling locations. Bridges and piers are normally good choices as they provide ready access and permit water sampling at any point across the width of the water body. When sampling from bridges, samples should be taken from the upstream side; however, this may alter the nature of water flow and cause sediment deposition. Additionally, bridges and piers are not always located in desirable locations with reference to waste sources, tributaries, etc. Wading for water samples is not recommended in lakes, ponds, and slow-moving rivers and streams. However, when wading for sample collections in slow-moving water bodies, it is best to work from downstream stations to upstream sampling points, especially when samples are taken in close proximity. In slow-moving or deep water, a boat is usually required for sample collections and sampling should allow for the possible presence of stratification.

3. SAMPLE COLLECTION TYPES

3.1. Grab sample

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

3.1.1. *Conditions when a grab sample is conducted*

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

3.1.2 *Grab sample collection methods*

A grab sampler is collected at 0.15 m below the water surface. Gloves should be worn for personal safety and to prevent sample contamination.

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

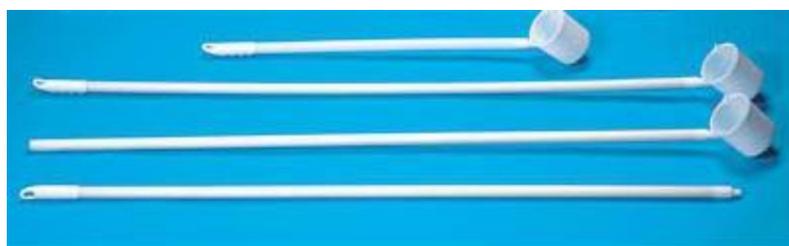


Figure 1. Polyethylene dipper typically used by DWR.

3.1.3. *Parameters that are always grab samples:*

metals	phenol
sulfide	oil and grease
volatile organics	bacterial
chlorine residual	other dissolved gases

3.2. Composite sample

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

3.2.1. *Timed integrated*

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

3.2.2. *Flow proportional integrated*

A flow proportional composite contains discrete samples, taken proportional to the flow rate over the compositing period. Proportional composites are collected with constant sample volume and constant time interval between samples proportional to stream flow.

3.2.3 *Area Integrated*

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

3.2.4. *Vertical spatial composite (Photic Zone Sampling)*

Vertical spatial samples are composite samples (a.k.a. photo zone, depth-intergraded samples) taken within the photic zone. The photic zone is found between the surface and twice the secchi reading (Chapter III.6). Samples are collected by lowering and raising an integrated depth sampling device such as a Labline water sampler (Figure 2) at a steady speed to obtain a representative water sample within the photic zone. Prior to sampling, the Labline should be rinsed 3 times with station water to avoid sample contamination.



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

3.3. Split sample

A split sample is a sample that has been portioned into two or more containers from a single collection device. Portioning assumes adequate mixing to assure the split samples are, for all practical purposes, identical. Devices such as churn splitters should be rinsed with ambient site water prior to field use for composite split samples, cleaned with phosphorous free cleaner after use and rinsed with deionized water before storage. Composite sample volume in the splitter should allow for $\frac{3}{4}$ of the total aliquot to be split with $\frac{1}{4}$ remainder. This prevents aeration of the sample during dispensing. Sample agitation should be performed for 2 minutes prior to sample split to ensure homogeneity of the composite. The spigot or valve should be purged prior to dispensing the first sample. As the composite volume in the churn is reduced, churning rate should increase.

3.4. Duplicate sample

Duplicate samples are collected simultaneously from the same source, under identical conditions but in separate containers.

3.5. Control sample

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

3.6. Background sample

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

3.7. Sample aliquot

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

3.8. Scoop sample

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

3.9. Physical Water Quality Measurements (In-Situ Field Measurements)

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

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

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

4. AUTOMATIC SAMPLERS

The Instrumentation Specialties Company (ISCO) model 2700 (Figure 3) and model 3700 wastewater samplers are portable devices designed to collect up to 24 separate sequential samples or can be programmed for composite sampling.

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

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

Example of an ISCO Sampler



Figure 3. ISCO automated samplers.

I. CONSIDERATIONS FOR WATER QUALITY SAMPLING

5. MANUAL SAMPLING

Manual sampling is usually employed when collecting grab samples and immediate *in-situ* field analyses samples. However, it may also be used, in lieu of automatic - equipment, over extended periods of time for composite sampling.

5.1. Manual Sampling Technique:

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

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



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

- 5.1.2. Collect the sample by lowering a properly cleaned collection vessel (bottle or intermediate sampling device) into the water or wastewater stream. If an intermediate sampling device is used, the container employed to collect the initial sample must be rinsed three times with sample water and must be constructed of a material that meets requirements of the parameter(s) being investigated. The collection vessel may be lowered by hand or attached to a pole or rope and then lowered into the stream.

- 5.1.3. Some types of analyses require the use of a pump when sampling. If a pump is used, it is imperative that it be pre-purged and all components of the pump that come into contact with the liquid be properly cleaned to ensure the integrity of the sample.
- 5.1.4. Tip the collection container into the water or wastewater stream so that the mouth of the container faces upstream.
- 5.1.5. Rinse out the container via this procedure at least twice before the sample is collected (exceptions to this rinsing procedure may exist if preservatives are present in the sampling container and for certain analyses such as oil and grease).

6. SPECIAL SAMPLE COLLECTION PROCEDURES

6.1. Priority pollutants

- 6.1.1. Priority pollutant detection limits are usually in the range of parts per billion, thus extreme care must be exercised to ensure sample integrity.
- 6.1.2. All containers, composite bottles, tubing, etc., used in priority pollutant sample collection should be cleaned as described in Chapter VI.
- 6.1.3. When possible, the sample should be collected directly into the appropriate sample container. If the material to be sampled cannot be physically reached, an intermediate collection device may be used. This device should be a Teflon, glass or stainless steel vessel or Teflon tubing via a peristaltic type pump. The device should be cleaned as described in Chapter VI.
- 6.1.4. When an automatic sampler is employed for priority pollutant collection, the procedures described in Chapter I concerning collection of organic and metal samples with automatic samplers should be used.

6.2. Bacterial sampling

Samples for bacterial analysis should always be collected directly into the prepared glass or plastic sample container. Everything possible must be done to avoid contamination through physical contact with the inside of the cap or bottle and mouth of the bottle.

- 6.2.1. Hold the bottle near the base.
- 6.2.2. With cap still on, plunge the bottle, neck downward, below the surface and turn until the neck points slightly upward. The mouth should be directed toward the current.
- 6.2.3. Uncap the bottle and fill to within one inch of the top without rinsing
- 6.2.4. Recap immediately while underwater.

6.3. Immiscible liquids/oil and grease

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

As it is very difficult to collect a representative oil and grease sample, the inspector must carefully evaluate the location of the sampling point. The most desirable sampling location is the point where greatest mixing occurs. Quiescent areas should be avoided. Because losses of oil and grease will occur onto the sampling equipment, the collection of a composite sample is impractical. Individual portions collected at prescribed time intervals must be analyzed separately to obtain the average concentrations over an extended period.

6.4. Volatile Organics Analyses (VOA)

Samples to be analyzed for volatile organics should be stored in the appropriate vials to prevent contamination and loss of sample. To verify proper sample container requirements, consult the DWR Chemistry Laboratory website (<http://portal.ncdenr.org/web/wq/lab/staffinfo>). The current methodology calls for 40 ml screw cap septum vials with a Teflon-silicone disk in the cap. The disks should be placed in the caps (Teflon side down) in the laboratory prior to the initiation of the sampling activities. Extra disks should be carried during field sampling in case of loss of the disks previously placed in the caps.

When there is no chlorine present in the sampled waterbody a 40ml VOA vial pre-preserved with 1:1 HCL by the Central Laboratory should be used for collection. A VOA sample should be preserved with ascorbic acid and 1:1 HCL whenever there is chlorine present or if it is not known if chlorine is present. Chapter 4 section 3.3.2 describes collection method used.

7. WASTEWATER SAMPLING

7.1 General considerations

Important procedures for obtaining a representative wastewater sample include:

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

I. CONSIDERATIONS FOR WATER QUALITY SAMPLING

7.1.1. Site selection

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

- a. Influent - Influent wastewaters are preferably sampled at points of highly turbulent flow in order to ensure adequate mixing.
- b. Effluent - Effluent samples should be collected at the site specified in the permit, or if no site is specified, below all treatment units including post aeration.
- c. Pond and lagoon sampling - Generally, composite samples should be employed for the collection of wastewater samples from ponds and lagoons. Even if the ponds and lagoons have a long retention time, composite sampling is necessary because of the tendency of ponds and lagoons to short circuit. However, if dye studies or past experience indicate a homogenous discharge, a grab sample may be taken as representative of the waste stream; but in all cases, sampling should be consistent with permit requirements.

7.1.2 Sampling techniques

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

<http://www.epa.gov/compliance/resources/publications/monitoring/cwa/inspections/npdesinspect/npdesmanual.html>

II. FIELD MONITORING

1. DATA SHEETS

There are two types of sheets needed for sample collection. A Field Sheet is used to document sample location and field parameters such as dissolved oxygen, temperature, pH, and secchi depth. A Lab Form is used to submit a sample(s) to the DWR Chemistry Laboratory.

1.1. Field Data Sheets (Figure 5)

These sheets have spaces for the information that identifies the station (station number, station name, date, and comments), sampler, lake observation (wind direction, rain, percent as well as providing spaces for conducting a depth profile by parameter. Data sheets can be found with the project manager (*i.e.* Ambient Lakes Coordinator).

- a. Use a pen to mark on the sheets. Make sure that whatever is used is waterproof.
- b. Write legibly and within the allotted space.
- c. These forms are retained by the sampler for use in writing up the results or may be filed for later use.

1.2. Lab Sheets (Figure 6)

- a. These forms are obtained by accessing the DWR's Chemistry Lab website:
<http://portal.ncdenr.org/web/wg/lab/staffinfo/samplesubmit/forms>
- b. A separate form is used for sediment, soil and tissue. Access the DWR Chemistry website to acquire the appropriate lab form. Contract labs will have their own; consult lab prior to sampling for any special requirements.
- c. Lab sheets have spaces for all the information that identifies the station and sampler as well as boxes to check indicating the types of analyses to be conducted on the samples from the station.
- d. The sample number used on the tags should be entered into the matching Lab Sheet. There is only one sample number per station . it should be recorded on the Lab Sheet and all the samples related to that Lab Sheet. There is only one lab sheet per station.
- e. Be sure to secure lab sheets(s) in a watertight container before shipping.
- f. After analysis is complete and the information is transcribed to the lab sheet, it will be returned to the sampler.

DIVISION OF WATER RESOURCES
Surface Water Fieldsheet

COUNTY : _____
RIVER BASIN : _____
REPORT TO : _____ Regional Office
Other : _____
COLLECTOR(S) : _____

AMBIENT QA
 COMPLIANCE CHAIN OF CUSTODY
 EMERGENCY VISIT ID: _____

SAMPLE TYPE
 STREAM EFFLUENT
 LAKE INFLUENT
 ESTUARY

Lab Number : _____
 Date Received : _____
 Time Received : _____
 Received By : _____
 Date Released : _____
 Date Reported : _____

Estimated BOD Range: _____ Station Location: _____
 Seeds: _____ Chlorinated: _____ Remarks: _____

Station #/Location Code	Date Begin (yy/mm/dd)	Date End (yy/mm/dd)	Time Begin	Time End	Depth - DM, DB, DBM	Value Type - A, H, L	Composite - T, S, B	Sample Type
BOD 310	mg/L	Chloride 940	mg/L	NH as N 610	mg/L	Li-Lithium 1132	ug/L	
COD High 340	mg/L			TRN as N 625	mg/L	Mg-Magnesium 927	mg/L	
COD Low 335	mg/L	Chlorophyll a EPA 445.0 modified option	ug/L	NO2 plus NO3 as N 630	mg/L	Mn-Manganese 1055	ug/L	
Coliform MF Fecal 31616	/100ml	Color: True 80	c.u.	P. Total as P 665	mg/L	Na-Sodium 929	mg/L	
Coliform MF Total 31504	/100ml	Color: (pH) 83 pH=	c.u.	PO4 as P 70507	mg/L	Arsenic: Total 1002	ug/L	
Coliform tube Fecal 31615	/100ml	Color: pH 7.6 82	c.u.	P. Dissolved as P 666	mg/L	Se-Selenium 1147	ug/L	
Coliform Fecal Strept 31673	/100ml	Cyanide 720	mg/L	K-Potassium	mg/L	Hg-Mercury 71900	ug/L	
Residue: Total 500	mg/L	Fluoride 951	mg/L	Cd-Cadmium 1027	ug/L	Ba-Barium	ug/L	
Volatile 505	mg/L	Formaldehyde 71880	mg/L	Cr-Chromium Total 1034	ug/L	Organochlorine Pesticides		
Fixed 510	mg/L	Grease and Oils 556	mg/L	Cu-Copper 1042	ug/L	Organophosphorus Pesticides		
Residue: Suspended 530	mg/L	Hardness Total 900	mg/L	Ni-Nickel 1067	ug/L	Organonitrogen Pesticides		
Volatile 535	mg/L	Specific Cond 95	unhos/cm	Pb-Lead 1051	ug/L	Acid Herbicides		
Fixed 540	mg/L	MBAS 38260	mg/L	Zn-Zinc 1092	ug/L			
pH 403	units	Phenols 32730	ug/L	V-Vanadium	ug/L	Base/Neutral & Acid Extractable Organics		
Acidity to pH 4.5 436	mg/L	Sulfate 945	mg/L	Ag-Silver 1077	ug/L	TPH Diesel Range		
Acidity to pH 8.3 435	mg/L	Sulfide 745	mg/L	Al-Aluminum 1105	ug/L			
Alkalinity to pH 8.3 415	mg/L	Boron	ug/L	Be-Beryllium 1012	ug/L	Purgeable Organics (VOA bottle req'd)		
Alkalinity to pH 4.5 410	mg/L	Tannin & Lignin	ug/L	Ca-Calcium 916	mg/L	TPH Gasoline Range		
TOC 680	mg/L	Hexavalent Chromium	ug/L	Co-Cobalt 1037	ug/L	TPH/TEX Gasoline Range		
Turbidity 76	NTU	Bicarbonate	mg/L	Fe-Iron 1045	ug/L	Phytoplankton		
Coliform Total Tube	/100 ml	Carbonate	mg/L	Mo-Molybdenum	ug/L			
		Total Dissolved Solids	mg/L	Sb-Antimony	ug/L			
				Sn-Tin	ug/L			
				Tl-Thallium	ug/L			
				Ti-Titanium	ug/L			
				Hg-1631	mg/L	Temperature on arrival (°C):		

COMMENTS : _____

 Revision: 08/23/13

Figure 6. Surface Water Lab Sheet

2. SAMPLE TAGS

A sample tag is used for most samples returned to the laboratory for analysis (Figure 7). These tags are usually attached to the sample container by a rubber band. In some cases, particularly with biological samples, the sample tag may be included with or wrapped around the sample. Sample tags should be of material that is waterproof and should be written on with indelible ink. It is very important that these tags are legible.

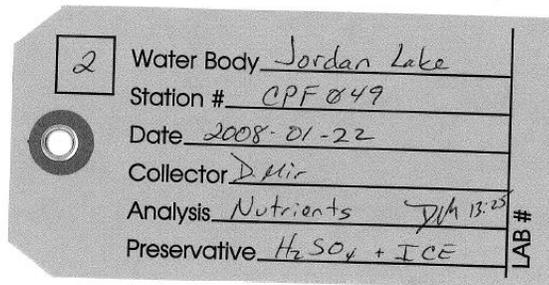


Figure 7. Completed Sample Tag

2.1 Information included on a sample tag:

- Sample number - determined based on number of stations to be sampled that day . All samples from a station will have the same sample number. Figure 7 shows the sample number for the tag as 2.
- Water Body
- Station number
- Date(s) & time(s)
- Name of the person collecting the sample
- Types of analyses to be conducted (such as Nutrients)
- Types of preservatives used
- Sampler initial after preserving with acid

2.2 Responsibility of project leader or field investigator

The project leader or field investigator assigns the station number to be used for that location. If previous sampling has occurred at a site, that station number should be used again. This number is ordinarily a numeric code, designed for a particular study, inspection, or investigation. Ambient stations have a special numbering system. New ambient stations are identified by the Ambient Monitoring Coordinator.

The project leader or field investigator must exercise due caution to ensure that duplicate station numbers are not used during the same study. The project leader or field investigator will also always specify the type of sample collected since the same station number is used when a water and sediment sample is collected at the same location. The exact description of all stations associated with field identification or sample station numbers is documented on the field sheet.

If a sample is split with a facility, state regulatory agency, or other party, sample tags with identical information are to be attached to each of the sample containers; the facility, state regulatory agency, etc., tag shall be marked facility (actual name), state regulatory agency (actual name), etc.

3. CHAIN-OF-CUSTODY PROCEDURES

This procedure is used for samples collected as part of an investigation for legal proceedings or where it is required under the study plan. The possession of samples or other evidence shall be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings.

3.1. Sample Custody

A sample or other physical evidence is under custody if it is in:

- The field investigator's actual possession, or
- The field investigator's view, after being in his/her physical possession, or
- The field investigator's physical possession and then he/she secures it to prevent tampering, or
- A designated secure area.

To simplify the chain-of-custody record and eliminate future litigation problems, as few people as possible should handle the samples or physical evidence. The field investigator is responsible for the care and custody of the samples collected until they are properly transferred to another person or facility.

3.2 Field Custody Procedures

3.2.1 *Security Seal*

- a. Complete sample tags for each sample.
- b. Place the lab sheets and chain of custody sheets in a Zip-loc bag and place in a cooler along with the samples.
- c. Seal the coolers with filament tape and a DWR custody seal similar to the one shown in Figure 8.
- d. The field investigator writes the date and their name on the seal. This requirement shall be waived if the field investigator keeps the samples in his custody from the time of collection until they are delivered to the laboratory.

3.2.2 *Chain of Custody Form*

- a. Record all samples on the field form or in field logbooks and using the Chain of Custody Record (Figure 9.) available from the DWR Chemistry Lab:
<http://portal.ncdenr.org/web/wq/lab/staffinfo/samplesubmit/forms>
- b. For documents received during investigations, place them in large envelopes, seal with a DWR seal such that the envelopes cannot be open without breaking the seal and note the contents on the envelope. If at any time the DWR seal is broken, that fact and the reason should be noted on the chain-of-custody record and a new seal affixed. The information on the seal should include the field investigator's signature, as well as the date and time of sealing.
- c. Place other physical evidence such as videotapes or other small items in zip-lock bags and affix a DWR seal so that the bag cannot be opened without breaking the seal. A chain-of-custody record should be kept with the items in the bag. Any time the seal is broken, note reason on the chain of custody record and affix a new seal.

- d. Personnel shall not accept samples from other sources unless the sample collection procedures used are known to be legally defensible, can be documented, and the sample chain-of-custody can be established. If such samples are accepted, a sample tag and a DWR form, containing all relevant information and the chain-of-custody record, shall be completed for each sample.



Figure 8. DWR Chain of Custody Security Seal

Report to: _____		SURFACE WATER SECTION			Page ____ of ____		
CHAIN OF CUSTODY (COC) RECORD							
NC DENR/DWR LABORATORY (check one): <input type="checkbox"/> CENTRAL <input type="checkbox"/> ARO							
For Investigation of:							
Sample collector (print name) _____ and DM-1 forms completed by: _____ Sample collector's signature: _____							
Field storage conditions and location (when applicable): _____							
Lab Use Only							
LAB NO.	STATION NO.	STATION LOCATION	DATE SAMPLED	TIME SAMPLED	NUMBER OF CONTAINERS		
Relinquished by (signature): _____			Date	Time	Received by (signature): _____		
Relinquished by (signature): _____			Date	Time	Received by (signature): _____		
Relinquished by (signature): _____			Date	Time	Received by (signature): _____		
Method of Shipment (circle one): State Courier Hand-delivered Federal Express UPS Other: _____							
Security Type and Conditions:		Sealed by: _____		Broken by: _____			
INTRALABORATORY CHAIN OF CUSTODY - Lab Use Only							
LAB NUMBERS FROM	THROUGH	NUMBER BOTTLES	ANALYSES REQUESTED	RELINQUISHED BY:	RECEIVED BY:	DATE	TIME

Figure 9. Surface Water Section Chain of Custody Form

II. FIELD MONITORING

3.3 Transfer of Custody and Shipment

- 3.3.1. When transferring the possession of chain of custody samples, the individuals receiving the samples shall sign, date, and note the time that they received the samples on the field form or in the field log book. This action documents transfer of custody of samples from the field investigator to another person (e.g. to the laboratory).
- 3.3.2. After properly packing samples for shipment to the appropriate laboratory for analysis, secure the shipping containers using nylon strapping tape and custody seals. The seal shall be placed under the point on the tape where the ends are located and wrapped over the top of it. The seal shall be signed, dated, and the time recorded by the field investigator.
- 3.3.3. Samples split with a facility, state regulatory agency, or other government agency must be signed for on the Chain of Custody Form by the facility, state regulatory agency, or other government agency representative receiving the samples.
- 3.3.4. All samples shipped shall be accompanied by the DWR chain-of-custody form(s). The original and one copy of the form will be placed in a plastic bag inside the secured shipping container. One copy of the form will be retained by the field investigator or project leader. The original of the form will be transmitted to the field investigator or project leader after samples are accepted by the laboratory.
- 3.3.5. If sent by mail, the package shall be registered with return receipt requested. If sent by common carrier, a government bill of lading or air bill should be used. Receipts from post offices, copies of bills of lading, and air bills shall be retained as part of the documentation of the chain-of-custody.

4. FIELD INSTRUMENTS

Intensive Survey Branch uses a wide array of instrumentation for recording in-situ water quality parameters. Currently, Hydrolab (Hach Environmental) and YSI (Yellow Springs Instrument Co.) are the main manufacturers used. Instructions for use, calibration, and maintenance as written by the manufacturer should always be followed. Manufacturers manuals for all meters can be found in the ESS Calibration Lab. DWR produced a guidance sheet that outlines basic calibration, maintenance, and acceptance criteria for meters commonly used by DWR (Appendices 1-4). **All meter guidelines and guidance sheets found in this document are supplementary to and not a replacement for the manufacturer's directions.**

- 4.1. All field meters should be calibrated before and checked after sampling activities daily. Calibration data should be documented on a Water Quality Monitoring Field Meter Calibration Sheet (Figure 10).

- 4.2 In-situ field parameter measurements

- 4.2.1. *Parameters typically measured:*

- a. **Conductivity** (S/cm @ 25 °C)
 - b. **Dissolved Oxygen** (DO- mg/L)
 - c. **pH** (Standard Units)
 - d. **Temperature** (°C)
 - e. Light Attenuation (E/m²/s)

- Additional Calibrations and Use of multiparameter Meters

- 4.2.2. *Battery Voltage*

- a. Use the correct battery source for the particular instrument in use.
 - b. Battery voltage must be in an acceptable range before calibrating and using the meter (see respective manual).
 - c. Record both initial and terminal battery voltage on the Meter Calibration sheet (Figure 10).

- 4.2.3. *Depth*

- a. Some meters can be calibrated to read depth by entering the number zero on the keypad while the sonde sensors are at the surface during field measurements.
 - b. Record all field depth measurements to the nearest tenth of a meter (if needed).

- 4.3 Calibrated Backup Field Meters

Although meters are maintained, failure can occur at anytime. Calibrated backup meters, meter manuals, batteries and calibration buffers/ standards are required during sampling. Inability to collect data due to a meter failure is unacceptable. See Appendices 1 . 4 for detailed guidance on using, maintaining, and storage field meters commonly used by DWR.

Water Quality Monitoring Field Meter Calibration Sheet

Collector(s): _____
 Study: _____
 Sampling Location: _____
 Meter Model: _____
 Meter / Sonde Serial No: _____

	Date yy/mm/dd	Time 24hr hh:mm	Initials
Pre-Sampling Calibration			
Post-Sampling Check			

Miscellaneous (Does not apply to YSI or Accumet Meters)

	Battery Level (V)	Stirrer Working?
Pre-Sampling Calibration		Y / N
Post-Sampling Check		Y / N

Battery Ranges = Surveyor: Internal- 7.2-7.5V, external- 11-13V; Quanta: 4.0-4.5V

Barometer Calibration (mmHg)
*YSI Pro Plus Meters Only

Initial Reading	Calibrated Value

Dissolved Oxygen (mg/L)

	Temp. °C	Initial % Saturation	Barometric Pressure (mmHg)	Altitude (ft.)	D.O. Table Value	Initial Meter Reading (mg/L)	Calibrated Meter Reading (mg/L)	Calibrated % Saturation
Pre-Sampling Calibration								
Post-Sampling Check					Within ± 0.5?	Y / N		

Specific Conductance (µS/cm at 25°C)

	Dry Air ^{1,2} Zero (0)		Conductivity Standard ³ Value: _____		Calibration Check Value: _____	
	Initial Meter Reading	Calibrated ⁴ Meter Reading	Initial Meter Reading	Calibrated ⁴ Meter Reading	Initial Meter Reading	Calibrated ⁴ Meter Reading
Pre-Sampling Calibration						
Post-Sampling Check	Within ± 2? Y / N		Within ± 10%? Y / N		Within ± 10% Y / N	

NOTE: Quanta reads in mS/cm; move decimal 3 places right for µS/cm.
¹ Dry Air CALIBRATIONS are conducted for 4a and MS5 Hydrolabs only.
² Dry Air CHECKS (confirmation of zero in dry air) are conducted for YSI 85, YSI 6920, YSI Pro Plus & Quanta meters.
³ Conductivity standards are used to CHECK the YSI 85 meter and to CALIBRATE all Hydrolab meters and the YSI 6920 & YSI Pro Plus.
⁴ Does not apply to Dry Air CHECKS or Conductivity Standard CHECKS (leave blank).

±10% Ranges for Sp. Cond.	
Standard	Range
100	90 to 110
500	450 to 550
1,000	900 to 1,100
10,000	9,000 to 11,000
15,000	13,500 to 16,500
50,000	45,000 to 55,000

pH (SU)

	Lot #: _____		Lot #: _____		Slope Efficiency ⁵	Confirmation Buffer 7.0
	Buffer #1 7.0		Buffer #2 4.0 / 10.0			Meter Reading
	Buffer Temp: _____	Buffer Temp: _____	Initial Meter Reading	Calibrated Meter Reading		
Pre-Sampling Calibration						
Post-Sampling Check	Within ± 0.2? Y / N		Within ± 0.2? Y / N		Within ± 0.1? Y / N	

⁵ Slope efficiency applies to Accumet meters only (does not apply to Hydrolab or YSI meters).

Comments:

Keep original on file for 5 years Ver. 06/05/2012

Figure 10. Meter Calibration Sheet

III. FIELD PARAMETER MEASUREMENTS

1. WATER TEMPERATURE

Temperature measurements are taken by a multiparameter meter (Hydrolab or YSI) or dial Celsius-thermometer or a thermister. Below are some general considerations while collecting water temperature data.

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

2. AIR TEMPERATURE

Refer to previous procedure, except measure the ambient air temperature above the water surface to be sampled. Do not use immersion thermometers to measure air temperature.

3. DISSOLVED OXYGEN

Dissolved oxygen analysis measures the amount of gaseous oxygen dissolved in an aqueous solution. Dissolved oxygen may be measured by electrometric methods (e.g. Hydrolab or YSI) or by chemical methods (Winkler Method).

Testing must be done immediately at the sampling location, as a grab sample, which is why electrometric methods are favored.

See Appendices 1 - 4 for detailed guidance on using, maintaining, and storage of meters and probes commonly used by DWR. **This SOP and the attached meter guidance sheets are supplementary to and not a replacement for the manufacturer's instructions manual.** Manufacturer's operations manuals for all meters are kept in the ESS Calibration Lab.

3.1 Electrometric Method Calibration

All field meters should be calibrated before and checked after sampling activities (at least daily). The calibration data should be entered on a meter calibration sheet (Figure 10). Detailed guidance for calibrating dissolved oxygen is provided in Appendices 1 . 4.

3.1.1 Acceptance Criteria For DO calibration

- Calibrated meters should be compared to the DO table to ensure calibration was done correctly.
- Appendix 5 describes the calculations needed to correct for elevation and a table used at sea-level.
- Dissolved oxygen concentrations need to be calibrated within 0.5mg/L of the elevation corrected table concentration for a given temperature.

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

The azide modification effectively removes interference caused by nitrite, which is the most common interference in biologically treated effluents and incubated BOD samples. The azide modification is not applicable under the following conditions:

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

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

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

- Collect surface water samples in narrow-mouth glass-stoppered BOD bottles of 300 ml capacity with tapered and pointed ground-glass stoppers and flared mouths. Once analysis is complete and the information is transcribed to the lab sheet, it will be returned to the sampler.
- Avoid entrapping or dissolving atmospheric oxygen. Do not allow the sample to remain in contact with air or be agitated, because either condition may result in a change to its gaseous content.

- Where samples are collected from shallow depths (less than 5 feet) use of an APHA-type sampler is recommended. Use of a Kemmerer type sampler is recommended for samples from depths greater than 5 feet. Bleed sample from bottom of samplers through a tube extending to the bottom of a BOD bottle. Fill bottle to overflowing.
- Record sample temperature to nearest degree Celsius or more precisely.
- Reagents
 - Manganous sulfate solution
 - Alkaline iodide-sodium azide solution.
 - Sulfuric acid (H_2SO_4) concentration
 - Sodium thiosulfate solution 0.025 N
 - Starch solution
- Analysis Steps:
 1. Add 2 mL of manganous sulfate solution to sample container by holding the tip of the pipette below the surface of the liquid.
 2. Add 2 mL of alkaline iodide-sodium azide solution by holding the tip of the pipette below the surface of the liquid.
 3. Replace BOD bottle stopper, avoid trapping air bubbles, and shake well by inversion.
 4. When the precipitate settles, leaving a clear supernatant above the manganese hydroxide floc, shake again.
 5. Allow floc to settle again, at least 200 mL of clear supernate should be above the floc.
 6. Remove the stopper and add 2 mL of concentrated sulfuric acid by holding the pipette above the surface of the liquid and allowing the acid to run down the neck of the bottle, re-stopper, and mix by inversion until no floc is visible.
 7. Withdraw 203 mL of the solution into an Erlenmeyer flask.
 8. Titrate with 0.025 N sodium thiosulfate solution to a pale straw color.
 9. Add 1 mL of starch solution and continue titration to the first disappearance of blue color.
 10. Record the # of mL of thiosulfate used; where 1 mL thiosulfate = 1 mg/L DO.

4. pH (ELECTROMETRIC METHOD)

4.1. Information on pH

- 4.1.1. *Precision and accuracy:* ± 0.2 pH unit represents the limit of accuracy under normal conditions for measurements of water and poorly buffered solutions. For this reason, report pH values to the nearest 0.1 pH unit. Calibrate instrument within 0.2 pH units of the standard pH buffer value.
- 4.1.2. *Calibration Reagents* - Calibrate the electrode system against standard buffer solutions of known pH. Always use fresh commercially made buffers to calibrate field meters. Buffer solution and samples should be stored in polyethylene bottles. Never pour decanted or used buffer solution back into the original bottle.
- 4.1.3 *Procedure* - Always follow the manufacturer's instructions for pH meter storage and preparation of electrodes. Recommended short-term storage of electrodes varies with type of electrode and manufacturer. See Appendices 1 - 4 for detailed guidance on using, maintaining, and storage of pH meters and probes commonly used by ISB. Never store probes in DI water; tap water or pH buffer 4.0 is preferred.

Note: All field meters should be calibrated before and checked after sampling activities daily. The calibration data should be entered on a meter calibration sheet (Figure 10).

4.2. Multiparameter YSI or Hydrolab Meters

The Hydrolab and YSI meters used by ISB all have the same basic method for calibration. A training outline for each meter used by ISB is listed in Appendices 1 - 4. Copies of the manufacturer's instruction manual are located in the ISB calibration room.

4.3. Accumet AP Series (Fisher Scientific) Handheld pH Meters

The Accumet handheld pH meter is a stand-alone pH meter (it does not measure any other parameters beyond pH). See Appendix 2 for detailed guidance on using, maintaining, and storage of the Accumet AP61 pH meter which is typically used in conjunction with the YSI 85 meter. A copy of the manufacturer's instruction manual is located in the ISB calibration room.

5. SPECIFIC CONDUCTIVITY/SALINITY

The specific conductance (conductivity) of a solution is a measure of its ability to carry an electrical current. This ability depends on the presence of ions, their total concentration, mobility, valence, and relative concentrations, and on the temperature of measurement. Specific conductance is the conductance afforded by 1 cc (ml) of a solution of electrolyte and is reported in micromhos per centimeter ($\mu\text{mhos/cm}$). Specific conductance measurements are used in water analysis to obtain a rapid estimate of the dissolved solids content of a water sample.

- 5.1 Specific Conductivity Meter Calibration - Detailed meter guidelines for calibrations are listed in Appendices 1 - 4. Copies of manufacturers' instruction manuals are found in the ISB calibration room.

Note: All field meters should be calibrated before and checked after sampling activities daily. The calibration data should be entered on a meter calibration sheet (Figure 10).

- 5.2 Additional Calibration Information
- *Acceptance Criteria:* Calibrate instrument within $\pm 10\%$ of the calibration standard's true value.
 - Always calibrate with fresh, certified conductivity standards.

6. SECCHI DISK TRANSPARENCY

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

6.1. Secchi disk use (Figure 11)

6.1.1. *Conditions for secchi disk readings*

- a. Shaded, protected side of boat.
- b. Minimal waves or ripples, if possible.
- c. Do not wear sunglasses while taking the secchi depth reading.

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

6.1.2. *Method*

- a. Rope should be accurately graduated in meters, 0.1 meter graduations for the first meter, 0.5 m graduations thereafter. At a minimum of annually verification of correct graduation is necessary as rope may stretch with continued use.
- b. Observer's eye should be 1 meter above the water surface.
- c. Lower the disk into the water to the depth at which the disk disappears.
- d. Lift the disk and record the depth at which it just reappears.
- e. Record the average reading from previous 2 steps on field sheet as Secchi depth reading to the nearest tenth of a meter.

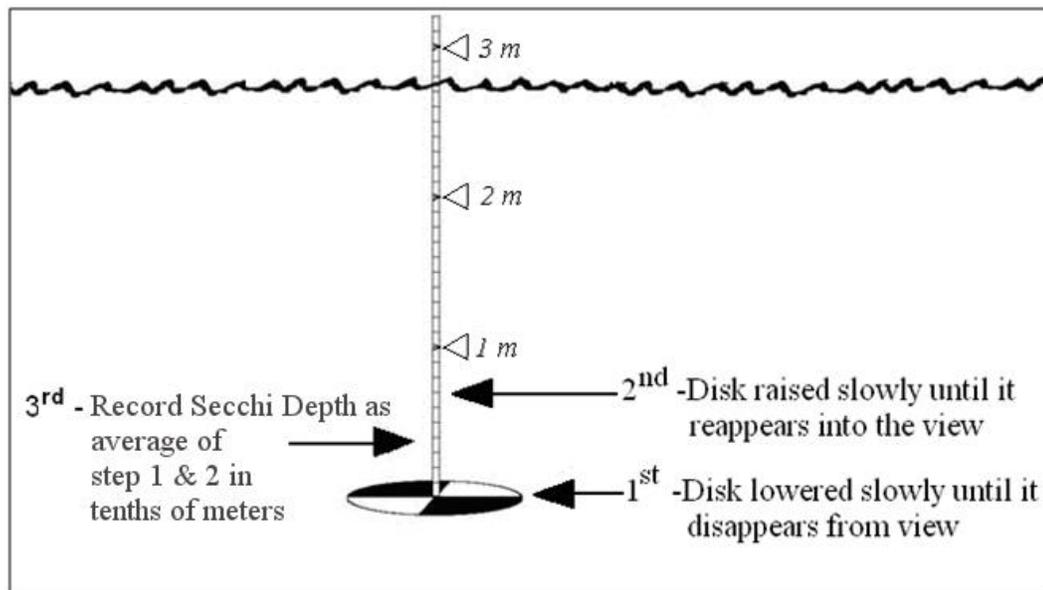


Figure 11. Secchi Disk

7. LIGHT ATTENUATION

The measurement of the decrease in light intensity through the water column as depth increases due to absorption and scattering effects of water molecules.

7.1. Light Attenuation is calculated by obtaining a vertical profile of light, using a PAR (photosynthetically active radiation) meter.

7.1.1. PAR Meter Preparation

- Obtain an independent datalogger such as LI-COR LI-1400.
- Connect a deck sensor and an underwater sensor to the LI-1400. Make sure the correct calibration factors are entered for each probe. All calibration factors are supplied by the manufacturer.
- Place the deck sensor on the boat where it will not be shaded.

7.1.2. Methods

- Lower the underwater sensor on the SUNNY, not shaded, side of the boat to a depth about 10 cm to represent the surface.
- Once readings stabilize, record the values from both sensors ($E/m^2/s$), along with the water depth of the underwater sensor. Log the values in the datalogger.
- Lower the underwater sensor to 0.5 m (6'), allow the values to stabilize and record the values from both sensors, along with the water depth of the underwater surface.

- d. Repeat at the following schedule:
- Shallow Sites (m \leq 2 m) . Every 0.5 m interval;
 - Nominal depths (>2 <10 m) . Every 0.5 m (near surface) and very 1 m interval to near bottom (0.5 m off-bottom)
 - Deep Sites (>10 m) . 0.5 m (near-surface) and every 1 m interval to 10 m, than at 5 m intervals, thereafter, to near-bottom (0.5 m off-bottom)
- NOTE:** Follow schedule, unless specified differently for the individual sampling project.
- e. If the meter impacts the bottom, allow 2-3 minutes for the disturbed conditions to settle before take the reading.
- f. If the light measurements become negative before reaching the bottom, terminate the profile readings at that depth.

8. REFERENCE POINT-TAPE-DOWN MEASUREMENT

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

8.1. Procedure for Reference Point-Tape-Down

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

9. STAGE MEASUREMENTS

These procedures are for use at U.S. Geological Survey permanent stream gauging stations.

9.1 Obtaining Stage Measurements

Follow instructions in the USGS publication Stage Measurements at Gauging Stations, Book 3, Chapter A7, United States Department of the Interior, Geological Survey, 1968. <http://pubs.usgs.gov/tm/tm3-a7/pdf/tm3-a7.pdf>

9.1.1 *Prior to Sampling*

- a. Obtain permission from the USGS district chief to read the stage measuring devices in the instrument shelters.
- b. Obtain on the job training by USGS personnel as to how to read the stage measuring devices.

9.1.2 *Stage Measuring Devices*

- Staff gage
- Wire weight gage
- Electric-tape instrument
- Automatic digital recorder
- Graphic recorder (bubble meters)

IV. WATER SAMPLE COLLECTION AND PRESERVATION

1. BOTTLES AND PRESERVATION

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

http://portal.ncdenr.org/c/document_library/get_file?uuid=719b475c-c4a7-44c7-86a7-1804bbd432c9&groupId=38364. Field staff are responsible for being familiar with the

Lab's procedures and following them accordingly. Preservatives can be added by pipette or pre-measured vials depending on the sensitivity of parameter being measured. If a parameter is not on the Lab's website, speak with the appropriate lab staff to determine how to proceed. Any samples submitted to the Lab must be accompanied by a Lab Sheet (Chapter 2-Figure 6). Immediately after sampling, labeling, and chemical preservation, samples are placed in coolers on ice, along with a temperature blank. Once samples arrive at the laboratory, support staff check the temperature blank (included in each cooler) to ensure that they are in appropriate temperature range ($4 \pm 2^{\circ}\text{C}$), assign lab tracking numbers, and distribute them to the appropriate analytical units. Any samples not meeting temperature, holding time, or preservation requirements or are otherwise not submitted in accordance with the SOP are subject to rejection as per Section 13: *Corrective Actions* of the Laboratory Section QAM. Laboratory staff will attempt to contact collector by phone or email before rejecting. If conditionally accepted, the laboratory will document the anomaly with a Sample Condition Upon Receipt (SCUR) and/or Sample Anomaly Report (SAR) form and include copies with the final analytical report. Results from anomalous samples will be reported using the appropriate qualification code(s).

2. COLLECTION METHODS FOR CONVENTIONAL PARAMETERS

Collection for majority of the conventional parameters can be done by the multiple methods introduced in Chapter 1, ~~Sample Collection Types~~. The following section is an overview of the types of parameters collected by DWR along with the required sample size, bottle type, preservative method and holding time.

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

- 2.1. BOD 5 . Day (Biochemical Oxygen Demand) - This test determines the amount of organic material in wastewater and surface waters by measuring the oxygen consumed by microorganisms in decomposing organic constituents. The test consists of the determination of dissolved oxygen prior to and following 5-day incubation of the sample at 20°C , thus establishing the amount of oxygen used.

2.1.1 *Collection method:*

- a. Collect sample in a 1-liter plastic bottle.
- b. Deliver within 48 hours
- c. Cool to 4°C
- d. For WWTP effluents, collect the sample ahead of disinfection when possible.

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

2.2.1 *Collection method:*

- a. Collect sample in a 200 ml plastic bottle
- b. Acidified the sample with H₂SO₄ to pH <2.
- c. Cool the sample to 4°C
- d. There is a 28 day holding period.

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

2.3.1. *General Collection Methods*

- a. Collect sample with a 250 ml wide-mouth sterile plastic bottle supplied by the DWR Laboratory. These bottles must contain sodium thiosulfate and EDTA reagents.
- b. Coliform sample is always collected as a surface grab sample. In no case should composite samples be collected for microbiological examination.
- c. Do not rinse bottle with sample, but fill it directly to within 1-2 inches from the top to allow mixing of the sample before analysis.
- d. Use caution to avoid contaminating the sample with fingers, gloves, or other materials.
- e. Cool to 4°C and return to lab **in less than 6 hours** from time of collection. The DWR Lab will analyze any coliform samples that are received in less than 24 hours; however, the data may not be acceptable for some uses due to extended holding time.

2.3.2. *Surface Sampling By-Hand*

- a. Grab sample should be collected directly into the sample bottle.
- b. Remove the bottle top to protect bottle and cap from contamination; avoid touching the inside of the bottle and cap.
- c. Grasp the bottle securely near the base with one hand and plunge the bottle mouth down into the water to avoid surface scum. Position the bottle towards the current flow and away from the hand of the collector, the shore, the side of the sampling platform, or boat. The sampling depth should be 0.15m below the water surface.
- d. If the water body is static, create an artificial current by moving the bottle away from the sampler while tipping the bottle slightly to allow water to enter.
- e. Tip the bottle slightly upwards to allow air to exit and the bottle. Fill the bottle to within 1-2 inches of the top.
- f. After removal of the bottle from the stream, tightly stopper and label the bottle.

2.3.3. *Surface Sampling by Weighted/Cage Bottle Frame* (Figure 4, pg. 19)

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

2.4. Residue (Solids) - Residue refers to solid matter suspended or dissolved in water or wastewater.

2.4.1. *Residue Types*

- a. Total Residue - is the term applied to the material left after evaporation of a water sample, and its subsequent drying in an oven at a defined temperature. Total residue includes nonfilterable residue and filterable residue. Also known as Total Solids.
- b. Nonfilterable Residue (Suspended) - the portion of total residue retained by a filter. The concentration of other water quality parameters is related to suspended solids since the

solid structure may contain biochemical and chemical oxygen demand materials, trace metals, nutrients, pesticides, and toxic or hazardous materials absorbed on the surface. Also, known as Total Suspended Solids.

- c. Filterable Residue (Dissolved) - the portion of total residue that passes through the filter. Dissolved solids consist mainly of inorganic salts, small amounts of organic matter, and dissolved gasses. Also called Total Dissolved Solids.
 - d. Volatile and Fixed Residue - the residue remaining after ignition for 1 hour at 550°C represents the ash or fixed solids, and the weight loss incurred is a reasonably accurate measure of organic matter or volatile solids.
- 2.4.2. *Collection method:* Use a 500 ml plastic bottle to collect **each** type of residue sample and cool to 4°C. The sample has a holding time of 7 days.
- 2.5. Alkalinity/Acidity - Alkalinity is a measure of the buffering capacity of water - the power of the water to neutralize hydrogen ions - and it is expressed in terms of an equivalent amount of calcium carbonate. Alkalinity is caused by the presence of carbonates, bicarbonates, and hydroxides. Acidity is the power of the water to neutralize hydroxy ions - and it is expressed in terms of an equivalent amount of calcium carbonate. Acidity is a result of the presence of free carbon dioxide, strong mineral acids, weakly dissociated acids, salts of strong acids, and weak bases.
- 2.5.1 *Collection method:* Collect sample with a 200 ml (for each parameter) plastic bottle, cool to 4°C. Holding time is 14 days.
- 2.6. TOC (Total Organic Carbon) - Measures the organic carbon present in water. When an empirical relationship can be established between TOC, BOD, and COD, the TOC provides a quick and convenient way of estimating the other parameters that express the degree of organic contamination.
- 2.6.1 *Collection method:* Collect sample with a 200 ml plastic bottle, add H_3PO_4 to pH <2 and cool to 4°C. Holding time is 28 days.
- 2.7. Turbidity (Clarity of Water) . measured in Nephelometric Turbidity Units (NTU). Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in straight lines. Turbidity in waters is a result of suspended matter such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, and plankton and other microscopic organisms.
- 2.7.1 *Collection method:* Collect sample in a 200 ml plastic bottle, cool to 4°C. The sample should be protected from light. The sample must be received by the lab in **less than 48 hours**.
- 2.8. Chloride - Chlorides are found in most natural waters. They may be of natural mineral origin or artificially introduced. Chloride concentrations are higher in wastewater than in raw water because sodium chloride (NaCl) is

a common article of diet and passes unchanged through the digestive system (American Public Health Association, 1992). Industrial processes also increase chlorides in wastewater effluents.

- 2.8.1. *Collection method:* Collect sample in a 500 ml plastic bottle, typically collected directly from the water body as a surface grab (0.15m deep). Cool to 4°C. Holding time is 28 days.

2.9. Chlorophyll *a* and Algal Biomass

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

Collection method: Use a 500 ml wide-mouth opaque plastic bottle to collect the sample. Cool to 4°C. Sample **must** be received by the laboratory in **less than 24 hours**.

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

Collection method: Samples for phytoplankton should be taken with an integrated depth sampling device (Labline water sampler).

- a) This device should be lowered to twice the secchi depth (approximately 1% light penetration) and slowly raised to the surface.
- b) Pour sample into a 500 ml plastic disposable bottle and preserve with approximately 2.5ml of modified Lugol's solution or until a dark straw color is reached.
- c) If a Labline is unavailable, a surface grab sample can be taken.
- d) Scoop samples are taken only when no quantitative methods are possible or as an additional sample for ease in identification. Live samples are taken as above (Labline preferred) but are not preserved. Cool to 4°C. Send to the Lab or EU lab in **less than 24 hours**.

2.9.3. *Chlorophyll a and Algal Sample Submittal Procedure*

- a. Samples should then be sent to the Central Laboratory along with nutrient and chemical samples.

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

2.9.4. *Parameters collected in conjunction with phytoplankton samples.*

- a. Physical Parameters- Are measured at the surface and at every meter or half meter from the surface to bottom according to depth. Parameters include:
 - 1. Temperature
 - 2. Dissolved Oxygen
 - 3. pH
 - 4. Secchi Depth
 - 5. Conductivity
 - 6. Salinity should be taken where appropriate.
- b. Chemical samples- Include ammonia/ammonium, nitrate/nitrite, total Kjeldahl nitrogen, orthophosphate, total phosphorous, and chlorophyll *a* are required to accompany phytoplankton samples.

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

- c. Map showing the location of the sampling site and/ or GPS coordinates.

- 2.10. Color - Color in water may result from the presence of natural metallic ions (iron and manganese), humus and peat materials, plankton, weeds, and industrial wastes. True color is the color of water from which turbidity has been removed by filtration or centrifugation. The term apparent color includes not only color due to substances in solution, but also that due to suspended matter. Apparent color is determined on the original sample without filtration or centrifugation. In stream samples, unaffected by industrial wastes, usually only true color is analyzed. In some highly colored industrial wastewaters, color is contributed principally by colloidal or suspended material. Therefore, apparent color may be a more appropriate measure for samples related to industrial wastewaters.

The color value of water is extremely pH dependent and increases as the pH of the water is raised. Therefore, always measure *in-situ* pH and specify the pH at which the color is determined.

2.10.1. *Accepted Methods to Determine Color*

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

- 2.10.2 *Collection method:* Use a 200 ml plastic bottle to collect a surface grab sample. Cool sample to 4°C. Sample must be submitted to the lab in **less than 48 hours**.

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

- 2.11.1 *Collection method:* Collect sample in a 200 ml plastic disposable bottle and cool to 4°C. Sample must be submitted to the lab in **less than 24 hours**.

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

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

2.12.1 *Collection method:*

- a. Use two 1 liter plastic bottles collect a surface grab sample directly from the water body.
 - b. Add NaOH to pH>12 and 0.6g of ascorbic acid if sample contains residual chlorine.
 - c. Cool sample to 4°C.
 - d. Sample has a holding time of 14 days.
- 2.13. Fluoride (F⁻) - Fluoride at 0.8 to 1.5 mg/l in drinking water aids in the reduction of dental decay, especially among children. Fluorides in high concentrations are not a common constituent of natural surface waters, but they may occur in detrimental concentrations in ground water. Fluorides are used as insecticides, for disinfecting brewery apparatus, as a flux in the manufacture of steel, for preserving wood and mucilage, for the manufacture of glass and enamels, in chemical industries, and water treatment. While not normally found in industrial wastes, they may be present in traces, or in higher concentrations resulting from spillage.

2.13.1 *Collection method:*

- a. Use a 500 ml plastic bottle to collect a surface grab sample directly from the water body.
- b. Sample must be cooled to 4°C.
- c. Holding time is 28 days.

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

2.14.1 *Collection method:*

- a. Collect surface grab sample in a 500 ml disposable plastic bottle
- b. Sample must be cooled to 4°C.
- c. Although no holding time is specified for this sample, it should be submitted to the lab as soon as possible.

2.15. HEM: Grease and Oil - For the grease and oil analysis; groups of substances with similar physical characteristics are determined quantitatively on the basis of their common solubility in trichlorotrifluoroethane (Freon). Grease and oils, either vegetable oil and animal fats or mineral hydrocarbons, when introduced to surface waters, are found floating on the surface, emulsified or solubilized in the water column, or settled on the bottom as a sludge. Potential contributors to oil pollution are all agencies engaged in productions, transportation, handling, and use of oil. Also, ships, railroads, civic dumps, salvage dumps, machining operations, and the most notable - garages and filling stations. Grease from animal and vegetable oils enters waterways from food processors and restaurants. Surface waters are at all times to be kept virtually free from oil or grease, not only for esthetic reasons and taste and odor problems for domestic water supply, but evidence has demonstrated both acute lethal toxicity and long term sublethal toxicity of oils to aquatic organisms.

2.15.1 *Collection method:*

- a. Collect 2 liters (two 1 liter glass wide mouth mason jars, Teflon-lined caps) of sample.
- b. A surface grab sample at 0.15 m deep is the only collection method.
- c. Acidify the sample with HCL or H₂SO₄ to pH <2.
- d. Sample must be cooled to 4°C Holding time for this sample is 28 days.

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

Classification of water by hardness content (Conc., mg/l CaCO₃) (USEPA, 1976).

Soft	0 - 75
Moderately Hard	75 - 150
Hard	150 - 300
Very Hard	300 and Up

The constituents that impart hardness to water are polyvalent cations, chiefly calcium (Ca⁺⁺) and magnesium (Mg⁺⁺). These form insoluble complexes with a variety of anions (HCO₃⁻, SO₄⁻, Cl⁻, NO₃⁻, SiO₃⁻). By convention, hardness is reported on the basis of equivalence as mg/l calcium carbonate (CaCO₃).

The DWR Lab is no longer analyzing samples for Total Hardness, therefore, when a total hardness sample is required, a nitric acid (HNO₃)-preserved sample must be submitted for Ca and Mg (see section 2.23). Once the Ca and Mg results are received from the lab, total hardness is calculated using the following formula:

$$\text{Total Hardness, mg/L} = 2.497[\text{Ca, mg/L}] + 4.118[\text{Mg, mg/L}]$$

2.16.1. *Collection method:*

- Sample must be collected as a surface grab (0.15 m from the surface) in a 500 ml plastic bottle.
- Acidify the sample with HNO₃ to pH <2 and cool to 4°C.
- Holding time is 6 months.

- 2.17. Specific conductance (Specific Electrical Conductance) - The specific conductance (conductivity) of a solution is a measure of its ability to carry an electrical current. This ability depends on the presence of ions, their total concentration, mobility, valence, and relative concentrations, and on the temperature of measurement. Specific conductance is the conductance afforded by 1 cc (ml) of a solution of electrolyte and is reported in micromhos per centimeter (µmhos/cm). Specific conductance measurements are used in water analysis to obtain a rapid estimate of the dissolved solids content of a water sample. This measurement is normally

made using a field meter; however, the following procedure can be used if necessary.

2.17.1. *Collection method:*

- a. Use a 200 ml plastic bottle collected as a surface grab (0.15 m from the surface).
- b. Sample should be cooled to 4°C.
- c. Holding time is 28 days.

2.18 MBAS - Methylene-Blue-Active Substances - This test determines

surfactants with no specificity, so the materials determined are designated as MBAS. This method depends on the formation of a blue salt or ion pair when methylene blue, a cationic dye, reacts with anionic surfactants. Surfactants are organic materials, which have the property of being surface active in aqueous solution. All surfactants have rather large polar molecules. One end of the molecule is particularly soluble in water and the other is readily soluble in oils. The surfactants include soaps, detergents, emulsifiers, wetting agents, and penetrants. Of these substances, the synthetic detergents are most important and are used in the greatest amounts. Presently, about 80 percent of all synthetic detergents are of the anionic type, and the MBAS method determines the presence of these surfactants. The most widely used anionic surfactant is linear alkylbenzene sulfonate (LAS). The detergent manufacturing industry changed to the production of LAS because it is more readily biodegradable than the older ABS (alkyl benzene sulfonates).

2.18.1 *Collection method:*

- a. **The lab must be notified that this sample will be collected and submitted for analysis.**
- b. Use a 500 ml plastic bottle to collect a surface grab sample (0.15 m from the surface) and cool to 4°C.
- c. Sample must be returned to the lab in **less than 48 hours**.

2.19. Phenols (C₆H₅OH) - An aromatic compound known as carbolic acid. In concentrated solution, phenol is quite toxic to bacteria and is widely used as a germicide. Phenol is obtained from coal tar and manufactured synthetically. It is used extensively in the synthesis of organic products, particularly phenolic-type resins. Phenolic wastes arise from the distillation of wood, from gas works, coke ovens, oil refineries, chemical plants, and from human and animal refuse.

2.19.1. *Collection method:*

- a. Use two- 1 liter glass (phenol bottles) bottles to collect a surface grab (0.15 m from the surface)
- b. Acidified the sample with H₂SO₄ to pH <2.
- c. Cool the sample to 4°C
- d. There is a 28 day holding period.

2.20. Sulfate (SO₄) - The sulfate ion is one of the major anions occurring in natural waters. Sulfates occur as the final oxidized state of sulfides, sulfites, and thiosulfates. Sulfates may also occur as the oxidized stage of organic matter in the sulfur cycle. Sulfates may be discharged in numerous industrial wastes, tanneries, sulfate-pulp mills, textile mills, and other plants that use sulfates or sulfuric acid. Sulfate is important to public water supplies because of its cathartic effect upon humans when it is present in excessive amounts (upper limit-250 mg/l U.S.P.H.S.). Sulfates are of considerable concern to wastewater treatment plants because of odor and sewer corrosion problems resulting from the reduction of sulfates to hydrogen sulfide (H₂S or hydrosulfuric acid in an aqueous solution).

2.20.1. *Collection method:*

- a. Sample should only be collected as a surface grab sample.
- b. Collect a surface grab (0.15 m from the surface) in a 500 ml plastic bottle
- c. Cool the sample to 4°C.
- d. Sample has a hold time of 28 days

2.21. Sulfide (S⁻) - Sulfides are constituents of many industrial wastes tanneries, paper mills, chemical plants, and gas works. Sulfides are also generated in sewage and some natural waters by the anaerobic decomposition of organic matter. Sulfides react with hydrogen ions to form HS⁻ or H₂S. The toxicity of sulfides derives primarily from H₂S rather than from the hydrosulfide (HS⁻) or sulfide (S⁻²) ions. H₂S is very toxic and has claimed the lives of numerous workmen in sewers, but owing to the unpleasant taste and odor (rotten eggs), most persons or animals avoid consuming a harmful dose.

2.21.1. *Collection method:*

- a. Samples should only be collect as a surface grab.
- b. Collect three- 40 ml glass VOA vials with Teflon-lined septum directly as a surface grab (0.15 m from the surface)
- c. Allow the sample to overflow the vial.
- d. Add 0.1 ml of 2N zinc acetate plus 6N NaOH to pH >9.
- e. cap the vial when sample is overflowing ,leaving no air space
- f. Cool the sample to 4°
- g. Holding time is 7 days.

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

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

2.22.1 *Nutrient Types*

- a. NH₃ (Ammonia) - In surface or ground waters, ammonia results from the decomposition of nitrogenous organic matter. It may also result from the discharge of industrial wastes from chemical or gas plants, from ice plants, or from scouring and cleaning operations where ammonia water is used. The conversion of ammonia to nitrites and nitrates by bacteria requires oxygen, and so the discharge of ammonia nitrogen and its subsequent oxidation can seriously reduce the dissolved oxygen levels in rivers and estuaries.
- b. TKN (Total Kjeldahl Nitrogen) - Analytically, organic nitrogen and ammonia can be determined together and are referred to as Kjeldahl nitrogen, a term that reflects the technique used in their determination.
- c. NO₂ + NO₃ (Nitrites + Nitrates) - Nitrites are quickly oxidized to nitrates. Nitrates are the end product of the aerobic stabilization of organic nitrogen. Nitrates also occur in percolating ground waters as a result of excessive application of fertilizer or leaching from septic tanks. Nitrates are seldom abundant in natural surface waters because of uptake by plants.
- d. Total P (Phosphorus) - Phosphorus occurs in natural waters and in wastewater almost solely as phosphates. High phosphorus concentrations are associated with accelerated eutrophication of waters when other growth promoting factors are present.
- e. PO₄ (Orthophosphate) - Orthophosphate is used as fertilizer and is applied to agricultural and residential cultivated land where it is carried into surface waters with storm runoff.

2.22.2 *Collection Methods for Unfiltered Nutrients*
(NH₃, TKN, NO₂+NO₃, and Total P)

- a. Use a 500 ml plastic disposable bottle for sample collected.
- b. Acidify the sample with H₂SO₄ to pH<2 (to 500 ml sample add 2.0 ml 25% H₂SO₄ **Note:** Addition of an excessive amount of acid will interfere with the sample analysis)
- c. Cool the sample to 4°C.
- d. Holding time is 28 days.

2.22.3. *Collection Method for PO₄ and Dissolved P (Filtered Nutrients)*

This water sample must be filtered in the field. A detailed Standard Operating Procedure for field filtering using a vacuum pump can be found in Appendix 6. Be careful- **do not** allow filter residue to touch filter apparatus or forceps.

- a. Use a 200 ml plastic bottle for each sample.
- b. **Dissolved P sample is acidized to pH <2 by adding 25% H₂SO₄.**
- c. Dissolved P and PO₄ samples must be cooled to 4°C
- d. **Holding time for PO₄ is less than 48 hours**
- e. The holding time for Dissolved P is 28 days.

NOTE: For Turbid Samples - change filters during process.

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

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

Collection method:

- a. Collect 500 ml of sample in a plastic disposable bottle directly from the water body as a surface grab (0.15 from the surface).
- b. Add HNO₃ to pH <2.
- c. Cool the sample to 4°C.
- d. Metals have a 6 month holding time with the exception of Mercury (Hg) which is 28 days.

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

- 2.23.2. *Chromium (Total Cr)* - The principal chromium emissions into surface waters are from metal finishing processes such as electroplating, pickling, and bright dipping. Other smaller discharges of chromium are from the additive in circulating cooling waters, laundry chemicals and animal glue manufacture, leather tanning, and textile dyeing. Chromium is one of the least toxic of the trace elements. Chromium is not acutely toxic to humans.
- 2.23.3. *Copper (Cu)* - Copper salts in natural waters are generally the result of pollution attributable to the corrosive action of water on copper and brass tubing, to industrial effluents, and algaecide. Copper salts are used in textile processes, pigmentation, tanning, photography, engraving, electroplating, insecticides, and fungicides. Because copper in concentrations high enough to be dangerous to human beings renders water disagreeable to taste, it is believed that copper is probably not a hazard in domestic water supplies. However, copper in water may be disadvantageous or detrimental for certain industrial uses. In trace amounts, copper may be beneficial or even essential for the growth of living organisms. In excessive quantities it has been found toxic to a wide variety of aquatic forms, from bacteria to fish.
- 2.23.4. *Nickel (Ni)* - Nickel toxicity to man is believed to be very low. Systemic poisoning of human beings by nickel or nickel salts is almost unknown. Nickel does not merit serious consideration as a water pollutant, but nickel ions may be detrimental to beneficial uses. Nickel is toxic to some plants. Nickel is used in metal plating, batteries, as a catalyst in the preparation of edible oils, and in solar energy equipment.
- 2.23.5. *Lead (Pb)* - Lead is a cumulative poison. The poisoning usually results from the cumulative toxic effects of lead after continuous, long-term consumption rather than from occasional small doses. Lead exists in nature mainly as the sulfide (galena). Some natural waters contain lead in solution where mountain limestone and galena are found. Lead may also be introduced into water as a constituent of various industrial and mining effluents or as a result of the action of the water on lead in pipes. Atmospheric fallout and rainout of particulate lead are considered the most significant sources of lead input into natural surface waters, especially in urban areas. Storm runoff originating in urban areas will tend to be high in lead concentration. The low solubility of lead in the aqueous phase of natural systems and the formation of stable complexes with organic matter are manifested in the low uptake by some plants and animals. There are extremely low concentrations of lead in natural bodies of water in proportion to the concentration in the beds of lakes and streams. The net effect of these sluggish dynamics is a high degree of accumulation with prolonged exposure.

- 2.23.6. *Zinc (Zn)* - Zinc is used extensively for galvanizing, in alloys, for electrical purposes, in printing plates, for dye manufacture, and dyeing processes. Zinc salts are used in paint pigments, cosmetics, pharmaceuticals, dyes, and insecticides. Zinc is found in high concentrations in natural waters in zinc mining areas and in effluents from metal plating works. In most surface and ground waters it is present only in trace amounts. There is some evidence that zinc ions are absorbed strongly and permanently on silt with a resultant inactivation of the zinc. Zinc has no known adverse physiological effects upon man except at very high concentrations. For esthetic considerations, high concentrations of zinc in domestic water are undesirable. At 30 mg/l, zinc gives water a milky appearance and causes a greasy film on boiling. It is toward fish and aquatic organisms that zinc exhibits its greatest toxicity at much lower concentrations.
- 2.23.7. *Silver (Ag)* - Silver metal is used in jewelry and silverware, in alloys, for electroplating, and in the processing of food and beverages. Silver nitrate is used in photography, ink manufacture, electroplating, coloring porcelain, and as an antiseptic. Traces of silver can be expected to reach natural waters from such sources. Silver is bactericidal and toxic at low concentrations.
- 2.23.8. *Aluminum (Al)* - Aluminum is the third most abundant element of the earth's crust. Aluminum occurs in many rocks and ores and clays, but never as a pure metal in nature. The metal itself is insoluble, but many of its salts are readily soluble. Aluminum is not likely to occur for long in surface waters because it precipitates and settles, or is absorbed as aluminum hydroxide or aluminum carbonate. In streams the presence of aluminum ions may result from industrial wastes or more likely from wash water containing alum from water treatment plants.
- 2.23.9. *Beryllium (Be)* - A relatively rare element, found chiefly in the mineral beryl, this substance is not likely to occur in natural waters. Although the chloride and nitrate forms are very soluble in water and the sulfate form moderately so, the carbonate and hydroxide forms are almost insoluble in cold water. Beryllium is used primarily in metallurgy to produce special alloys, in the manufacture of X-ray diffraction tubes and electrodes for neon signs, and in nuclear reactors. Beryllium is not harmful when taken internally through the digestive tract but has been incriminated in pulmonary ailments of workers exposed to beryllium dusts.

- 2.23.10. *Calcium (Ca)* - Calcium is the most abundant dissolved cationic constituent of natural fresh waters. This element is widely distributed in the minerals of rocks and soils. Calcium carbonate is frequently found as a cementing agent between mineral particles of sandstone and other detrital rocks. Calcium is one of the constituents of hard water and is a scale former in hot water systems. Prevention of corrosion of cast iron water distribution systems may be obtained through controlled precipitation of calcium carbonate. Lime (CaOH_2), and dolomite [$\text{CaMg}(\text{CO}_3)_2$] are frequently employed as neutralizing agents in water and wastewater treatment.
- 2.23.11. *Cobalt (Co)* - Cobalt and its salts are used for making alloys, in nuclear technology, as pigment in the china and glass industry, and as binders in the tungsten-carbide tool industry. Cobalt has a relatively low toxicity to man, and traces of cobalt are essential to nutrition.
- 2.23.12. *Iron (Fe)* - Iron interferes with laundering operations, imparts objectionable stains to porcelain fixtures, and causes difficulties in distribution systems by supporting growths of iron bacteria. Iron also imparts a taste to water, which is detectable at very low concentrations. In addition to corrosion products, natural waters may be polluted by iron-bearing ground water.
- 2.23.13. *Lithium (Li)* - An alkali metal, it is not widely distributed in nature, being found in a few minerals and in certain spring waters. Lithium is used in metallurgy, medicinal waters, some types of glass, and, as lithium hydroxide, in storage batteries. Lithium is toxic at high concentrations.
- 2.23.14. *Magnesium (Mg)* - Magnesium ions are of particular importance in that they occur in significant concentration in natural waters, and along with calcium, form the bulk of the hardness reaction. Magnesium is considered relatively non-toxic to man and not a public health hazard because, before toxic concentrations are reached in water, the taste becomes quite unpleasant. At high concentrations, magnesium salts have a laxative effect, particularly upon new users, although the human body can develop a tolerance to magnesium over a period of time.
- 2.23.15. *Manganese (Mn)* - Manganese is essential for the nutrition of both plants and animals. Manganese is undesirable in domestic water supplies because it causes an unpleasant taste, deposits on food during cooking, stains, and discolors laundry and plumbing fixtures, and fosters the growth of some microorganisms in reservoirs, filters, and distribution systems. Manganese frequently appears in surface waters as the result of decaying vegetation, in waters with acid pH values, and acidic waters from coal mine drainage. In ground water subject to reducing conditions, manganese can be leached from the soil and occur in high concentrations.

- 2.23.16. *Sodium (Na)* - Sodium salts are extremely soluble in water; any sodium that is leached from soil or discharged to streams by industrial wastes will remain in solution. Sodium is the cation of many salts used in industry and as such is one of the most common ions in process wastes. Sodium in drinking water may be harmful to persons suffering from cardiac, renal, and circulatory diseases.
- 2.23.17. *Potassium (K)* - One of the more common elements, potassium is one of the most active metals, and for that reason it is not found free in nature but only in the ionized or molecular form. Potassium is used for fertilizers and some varieties of glass. It is an essential nutritional element, but in excessive quantities it acts as a cathartic.
- 2.23.18. *Barium (Ba)* - Barium ions are not normally present in natural surface or ground waters in measurable concentrations although they have been detected in a few springs and in effluents from areas where barytes, BaSO_4 , or witherite, BaCO_3 , are mined. Barium and its salts are used in the metallurgical industry for special alloys, in the paint industry, in cements designed to withstand salt water, and in the ceramic and glass industries. Because of possible toxic effects on the heart, blood vessels and nerves a surface water supply standard of 1.0 mg/l was established.
- 2.23.19. *Arsenic (As)* - Arsenic may occur in water as a result of mineral dissolution, industrial discharges, or the application of insecticides. Arsenic is toxic to humans and accumulates in the body.
- 2.23.20. *Selenium (Se)* - Elemental selenium is practically nontoxic, but hydrogen selenide and other selenium compounds are extremely toxic and resemble arsenic in their physiological reactions. Selenium poisoning occurs mostly among livestock, and the toxic effects appear to be associated with the consumption of high concentrations of selenium in food, such as locoweed or grains grown in soils with high concentrations of selenium, rather than from water consumption. Selenium occurs in sulfur deposits, sulfides of metals, volcanic emissions, sedimentary rocks, organic-rich soils, and coal. Selenium is used in the electronics industry, xerographic copying machines, photoelectric cells, glass and ceramics, pigment manufacture to color plastics, paints, enamels, inks, and rubber. It is also used as a component of plating solutions. It can also be found in discharges from coal-fired power plants.
- 2.23.21. *Mercury (Hg)* - Mercury and mercuric salts are considered to be highly toxic to humans and aquatic life. Elemental mercury is inert chemically and insoluble in water, and is not likely to occur as a water pollutant. Mercuric salts occur in nature chiefly as the sulfide HgS , known as cinnabar, but numerous synthetic organic and inorganic salts of mercury are used commercially and industrially. They are used in medicinal products, disinfectants, detonators,

pigments, and photoengraving. Many of the mercuric and mercurous salts are highly soluble in water.

3. PESTICIDES AND ORGANICS

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

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

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

3.2. Organics - All organic compounds contain carbon in combination with one or more elements.

3.3. Collection Methods

3.3.1. *Pesticides, semivolatile organics, and acid herbicides*

- a. Collect each sample (Pesticides, semivolatile organic & acid herbicides) into a separate 1 gallon amber glass jug with a Teflon-lined cap.
- b. The sample is collected directly from the water body as a surface grab (0.15m deep).
- c. Add sodium thiosulfate and cool to 4°C.
- d. Holding time is 7 days.

3.3.2. *Purgeable Organics (VOA)*

- a. Collect sample into three-40 ml Teflon vials, remove cap underwater.
- b. When collecting in waters with no chlorine, use vials pre-preserved by the Central Laboratory with sodium bisulfate (NaHSO_4).
- c. The vial should be filled and capped underwater (0.15m deep) with no air space in the vial. While keeping lid and bottle under water gently rock the lid and bottle to remove air bubbles (unless bottle is pre-preserved). The volatile organics vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence that could also produce volatilization.
- d. When collecting in waters where chlorine is present, first preserve an empty vial with 0.6 g of ascorbic acid before filling and capping the vial underwater. After capping the vial, remove the vial above water, uncap, and add 0.25 g of sodium bisulfate leaving no air space before recapping the vial.

- e. The three vials should be placed in a Ziploc bag in the cooler. A trip blank is also required. This is a vial filled at the laboratory with appropriate bottled water and placed in a Ziploc bag in the same cooler with the other VOA vials.
- f. A separate laboratory sheet is filled out for the trip blank and this sample is used to determine if any contamination has occurred of the VOA samples.
- h. Cool to 4°C.

3.3. Instructions for requesting a pesticide or organic analysis

When a particular pesticide or organic is suspected or known to be present in a sample, its name should be entered on the laboratory form. With this information, the laboratory can focus immediately on the analytical methodology for determining the presence and concentration of the suspected pollutant and as a result possibly decrease the analysis time. A list of specific pesticides and organics currently analyzed at the Laboratory is available online: <http://portal.ncdenr.org/web/wq/lab/ops/org>

V. SEDIMENT COLLECTION AND PRESERVATION

1. COLLECTING SUSPENDED SEDIMENT

1.1. Samplers and Applications

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

1.1.1. *U.S. DH-48 – When in wadable streams*

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

1.1.2.1 Used When

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

1.1.2.2 Sampling Tips

- a. Set out safety equipment (cones, high visibility vests, etc.) as necessary and assemble sampling equipment. Note: Prior to using any sampler, it should be thoroughly cleaned and inspected.
- b. Rinse sampler with distilled water before the first station and between stations to wash away any contaminants.
- c. Use the upstream side of bridges if possible.
- d. Go to midstream or the area where most of the flow is occurring. First sampling point must be made where the flow is greatest.
- e. Lower and raise the sampler at a consistent rate with the nozzle oriented upstream to the bottom, immediately reverse it and raise to above the water surface. Repeat until jar is filled within approximately 3 inches of the top of the jar (350-440 cc). Rate must not exceed 0.4 times the mean velocity and must be fast enough to keep from overflowing.
- f. If bottle overfills - discard sample, rinse bottle, and collect again. Use a smaller nozzle or a faster transit rate.
- g. Raise the sampler and pour contents into a cleaned sample splitter. For cleaning instructions see USGS references. The sample splitter should be rinsed with distilled water before the first station and between stations.
- h. Sample at the next sampling point and place contents into a mixing churn.

- i. Ideally try for 3 sampling points, midstream and quarter points, but the situation might indicate otherwise (if maximum flow is not midstream). Sampling points should be equally spaced.
- j. If more sample is needed for the churn, take a second set of samples at the same transit rate at all verticals.
- k. Churn sample at a uniform rate of about nine inches per second. Disc should touch the bottom of the tank on every stroke and the stroke length should be as long as possible without breaking the water surface.
- l. After churning for about 10 strokes, withdraw sub-samples and place in ½ liter bottles. As sub-samples are withdrawn, maintain churning rate. If there is a break in withdrawals, the stirring rate must be re-established before withdrawals can continue.

1.2. Variations on Suspended Sediment Sampling

- 1.2.1. When suspended materials in the stream are uniformly distributed, a representative sample can be obtained by sampling vertically at one location near the center of the flow.
- 1.2.2. Use surface or dip sampling instead of depth integrated sampling when:
 - Stream velocity is too high.
 - Large floating and moving submerged debris is in the stream.
 - A depth-integrated sampler is not available.
 - The depth of the stream is very shallow.

2. COLLECTING BOTTOM SEDIMENT

2.1. Containers and Volumes

2.1.1. *Sample Containers*

- a. Use certified jars for sediment samples or as indicated by Chemistry Laboratory.
- b. Use Teflon lid or parafilm between jar and lid for nutrients and all metals.
- c. Tin foil can be used between jar and lid for all metals except aluminum.
- d. Use Teflon lid or tinfoil for pesticides.

2.1.2. *Required volume*

- One pint of sample must be obtained for analyses of metals, nutrients, and organics.

3. BOTTOM SEDIMENT SAMPLERS, APPLICATIONS, AND PROCEDURES

For more descriptive information about these samplers see references.

3.1. Ekman grab (Figure 12)

3.1.1. *Locations Suitable for Use:*

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

3.1.2. *Sampling Tips:*

- a. Make sure grab is operating correctly. The grab can cause severe injury. Do not activate unit while holding.
- b. If sampling from a low bridge, it may be advisable on wide streams to take 3 samples (midstream and quarter sections) and composite them to form 1 sample in a Nalgene mixing tub.
- c. Set in open position by locking open the spring operated jaws.
- d. Operating procedures are similar to those of the Petersen grab starting at step 5.2.6.

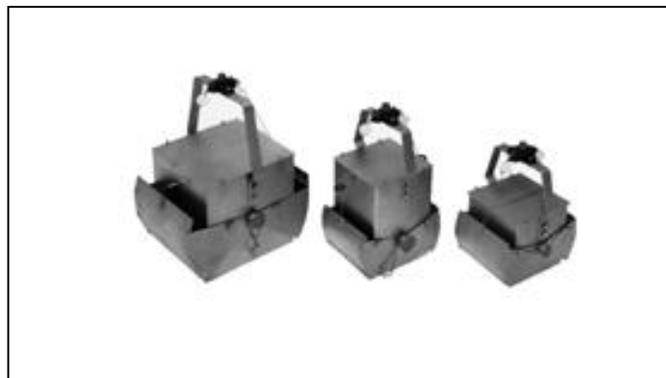


Figure 12. Ekman Grab Samplers

3.2. Petersen grab (Figure 13)

3.2.1. *Locations Suitable for Use:*

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

3.2.2. *Sampling Method and Tips*

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



Figure 13. Peterson Grab Sampler

3.3. Ponar grab (Figure 14)

3.3.1. *Locations Suitable for Use:*

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

3.3.2. *Sampling Method and Tips*

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



Figure 14. Ponar Grab Sampler

4. BOTTOM CORE SAMPLERS, APPLICATIONS, AND PROCEDURES

4.1. Phleger core sampler (Figure 15.)

4.1.1. *Locations Suitable for Use:*

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

4.1.2. *Phleger Core Sampler Methods*

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

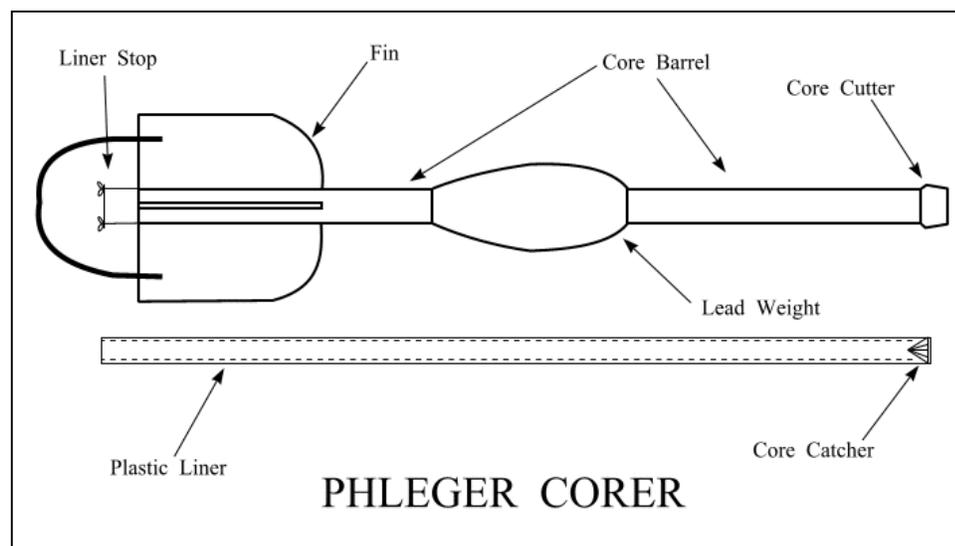


Figure 15. Phleger Corer Diagram

4.2. Wildco Light Duty Model 2414 Core Sampler

4.2.1. *Locations Suitable for Use:*

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

4.2.2. *Wildco Light Duty Model 2414 Core Sampler Methods*

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

4.3. Hand coring device. for shallow water use.

4.3.1 *Procedure for hand coring device:*

- a. Make sure sampler is clean before using. Rinse before first station and between stations.
- b. Take sample by turning sampler into sediment.
- c. Remove sampler and core.
- d. Measure out top two inches of core.
- e. Place this into jar.
- f. Repeat sampling until approximately one pint of sample is obtained.

4.4. Hand Sampling Method

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

VI. STANDARD CLEANING PROCEDURES

1. GENERAL

The procedures outlined in this section are to be used by all personnel to clean sampling equipment and sample containers prior to field use. These procedures assure the standard operating procedures (SOP) for the Section; any deviation from them must be documented in field records and investigative reports.

All equipment and sample containers that are cleaned using these procedures will be tagged, labeled or marked with the following information:

- Name of person cleaning equipment or containers
- Date equipment or containers were cleaned
- Any deviation from SOP that was employed

All equipment and reusable sample containers used to collect samples will be identified at the conclusion of sampling activities. Any problems encountered with the equipment or needed repairs will also be noted. Equipment or reusable sample containers needing cleaning or repairs should not be stored with clean equipment, sample tubing or sample containers. Equipment, reusable sample containers, disposable sample containers, and sample tubing that are not used during the course of an investigation may not be replaced in storage without being re-cleaned if these materials are transported to a facility or study site where herbicides, pesticides, organic or other toxic materials are present or suspected of being present. All portions of unused coils of tubing that are returned shall be re-cleaned before being restocked. If these materials are transported to a facility in connection with a routine inspection or study where toxic or organic materials are not known or not suspected of being present, they may be placed back in storage without being cleaned.

Sufficiently clean equipment and sample containers should be transported to the field so that an entire study can be conducted without the need for cleaning equipment in the field. However, this will not always be possible when using coring equipment, dredges, buckets, water samplers, pumps and other such equipment. Field cleaning procedures are included to cover these special problems. Emergency field sample container cleaning procedures are also included; however, they should not be used unless absolutely necessary. Specific cleaning procedures are included in the following paragraphs.

2. AUTOMATIC SAMPLING EQUIPMENT

2.1. General Cleaning

2.1.1. *For All Automatic Samplers*

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

2.2. ISCO Specific Cleaning Procedures

2.2.1. *Automatic sampler rotary funnel, distributor and metal tube*

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

2.2.2. *Automatic sampler headers*

- a. Rinse entire header with hot water, a bottle brush, and phosphate free laboratory detergent.
- b. Disassemble header and rinse thoroughly with hot tap water, using a brush to remove particulate matter and surface films.
- c. Rinse plastic portion of the header with 20 percent nitric acid. Do not use acid on metal parts.
- d. Rinse thoroughly with tap water.
- e. Reassemble header and rinse with distilled water.
- f. Let dry thoroughly and wrap with aluminum foil.

- g. Headers may not be used when collecting samples for organic analyses.
- 2.2.3. *Glass reusable composite containers (2 ½, 3 and 5 gallon capacities)*
- a. After using, rinse with water in the field, seal with aluminum foil to keep the interior of the container wet and return to the laboratory.
 - b. Wash thoroughly with hot tap water and phosphate free laboratory detergent, using a bottle brush to remove particulate matter and surface film.
 - c. Rinse thoroughly with hot tap water.
 - d. Wash with 10 percent nitric acid.
 - e. Rinse thoroughly with tap water (at least 3 times).
 - f. Rinse thoroughly with distilled water (at least 3 times).
 - g. Rinse thoroughly with acetone (pesticide grade). Caution: Acetone must be removed before using. Residual acetone will interfere with certain analyses.
 - h. Rinse twice with distilled H₂O. Allow to air dry,
 - i. Cap with aluminum foil or Teflon film.
 - j. Do not use composite containers used to collect samples at facilities manufacturing pesticides, herbicides or other toxic or noxious compounds. These are to be properly disposed of at the DWR Chemistry Laboratory.
 - k. Glass composite containers used to collect in-process wastewater samples at industrial facilities will be discarded after sampling.
 - l. Any bottles that have a visible film scale or discoloration remaining after this cleaning procedure are to be discarded.
- 2.2.4. *Glass sequential sample bottles (automatic sampler base for sequential mode)*
- a. Rinse bottles in the field after using with tap water and seal with aluminum foil or cap for return to laboratory.
 - b. Rinse thoroughly with hot tap water.
 - c. Wash with 20 percent nitric acid.
 - d. Rinse thoroughly with tap water.
 - e. Place in dishwasher - phosphate free detergent cycle followed by tap and distilled water rinse cycles.
 - f. Replace in covered, automatic sampler base; cover with aluminum foil for storage.
- 2.2.5. *Bottle siphons*
- a. Use a new siphon for each sampling location.
 - b. Pre-rinse the 3/8 inch Teflon tubing (used to make siphons for organic analyses) as in Teflon tubing cleaning instructions.

- c. Flush the PVC 3/8 inch tubing used for samples other than those collected for organic analyses with sample before use.
- 2.2.6. *Teflon composite mixer rods*
- Use the sample cleaning procedure outlined for glass reusable composite containers above.
- 2.2.7. *Automatic sampler rubber pump tubing*
- Only new pre-rinsed tubing should be used for each automatic sampler set up
 - a. Rinse tubing with hot tap water for five minutes.
 - b. Rinse outside of tubing with hexane.
 - c. Install in automatic sampler.
 - d. Cap both ends of tubing with aluminum foil or Teflon film.
- 2.2.8. *Teflon sampler tubing (pure Teflon or Teflon lined)*
- a. If required length is known pre-cut Teflon tubing or clean 100 feet coil intact.
 - b. Rinse outside of tubing with hexane.
 - c. Flush interior of tubing with hexane.
 - d. Air dry.
 - e. Cap each end of tubing with aluminum foil or Teflon tape and completely wrap the coil of Teflon tubing with aluminum foil to prevent contamination.
- 2.2.9. *Polyvinyl chloride sample (PVC) tubing (1/18, 1/4, or 3/8 Inch)*
- a. Use only new tubing.
 - b. Use in selective sampling where organics are not of concern.
 - c. Flush the tube with sample immediately after the sampler is set up at the sampling site to remove any residues from the manufacturing or extruding process.
 - d. Store tubing in original container and do not removed from this container until needed.
- 2.2.10. *Stainless steel tubing*
- Tubing will be flushed in the field with tap water after use and cleaned as follows upon return to the laboratory:
- a. Wash with phosphate free laboratory detergent and a long bottle brush.
 - b. Rinse with hot water for 5 minutes.
 - c. Rinse with acetone.
 - d. Rinse with distilled water for one minute.
 - e. Air dry.
 - f. Rinse with hexane.
 - g. Completely wrap tubing, including ends, with aluminum foil to prevent contamination during storage.

3. MISCELLANEOUS SAMPLING AND FLOW MEASURING EQUIPMENT

Miscellaneous flow measuring and sampling equipment should be washed with phosphate free laboratory detergent and rinsed with hot tap water before being stored. For Lablines, rinse at least three times with distilled deionized water and cover the top of the Labline with foil to prevent contamination and to show that the Labline has been cleaned.

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

4. STAINLESS STEEL SAMPLING EQUIPMENT

For collecting samples for organic analyses:

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

5. OTHER FIELD INSTRUMENTATION

NOTE: Where available, always follow the manufacturer's recommendations for cleaning the device (see Appendices 1-4).

The exterior of sealed, watertight equipment such as Labline Samplers and field meters should be washed with a mild detergent (liquid dishwashing detergent, for example) and rinsed with tap water before storage. The interior of such equipment may be wiped with a damp cloth if necessary. Other field instrumentation should be wiped with a damp cloth. Probes for pH, conductivity, DO, etc. should be rinsed with distilled water before storage. The desiccant in flow meters and other equipment should be checked and replaced if necessary each time the equipment is cleaned.

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

6. ICE CHESTS AND SHIPPING CONTAINERS

All ice chests and reusable shipping containers will be washed with a mild detergent (interior and exterior) and rinsed with tap water and air dried before storage.

7. FIELD CLEANING PROCEDURES

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

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

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

8. VEHICLES

All vehicles used by staff should be washed on a routine basis. This routine maintenance should minimize any chance of contamination of equipment or samples due to contamination of vehicles. When vehicles are used in conjunction with hazardous waste site inspections, or on studies where pesticides, herbicides, organic materials or other toxic matter are known or suspected to be present, a thorough interior and exterior cleaning is mandatory at the conclusion of such investigations. All vehicles shall be equipped with trash bags and/or trash containers to facilitate vehicle cleaning. All contaminated trash and equipment must be kept separate from ordinary trash and must be disposed of properly on-site or on return to the facility.

9. DISPOSABLE SAMPLE CONTAINERS

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

VII. TIME-OF-TRAVEL & DYE TRACING

1. FLUORESCENT DYE

The preferred dye for use in time-of travel studies by the North Carolina Division of Water Resources is Rhodamine W. T. (20%) solution. This is a red fluorescent dye which mixes well with water and is easily detected through visual means under high concentrations and through the use of a fluorometer for concentrations to as low as 0.01 parts per billion. Rhodamine WT has properties essential for water tracing studies. Rhodamine WT is:

- water soluble,
- highly detectable-strongly fluorescent,
- fluorescent in a part of the spectrum not common to materials generally found in water, thereby reducing the problem of background fluorescence,
- harmless in low concentrations,
- inexpensive, and
- reasonably stable in a normal water environment (Wilson, Cobb & Kilpatrick, 1986).

Rhodamine dye can also be used to determine such things as short-circuiting in wastewater treatment plants, outlets from storm drains, septic tank leakage, etc.

Most of ISB's dye studies are performed as part of a waste-load allocation model. This model requires that a stream be segmented into different reaches based upon predicted stream velocities, stream morphology, total distance of the study area, and major inputs from dischargers and tributaries. A dye sampling station is required in each of these reaches.

1.1. Safety

(MSDS is kept with dye container)

1.1.1. *Personal Protection*

- a. Latex or vinyl gloves (in lab and field).
- b. Goggles
- c. Ventilated room
- d. Apron

1.1.2. *Emergency and First Aid Procedure*

a. Inhalation:

- move to fresh air.
- Give oxygen and medical help if breathing is difficult.

b. Eye contact:

- Flush eyes with flowing water for at least 15 minutes, holding eyelids apart to irrigate thoroughly.
- Get medical attention right away.

c. Skin contact:

- Wash affected skin areas thoroughly with soap and water.
- If irritation develops, consult a physician.

d. Ingestion:

- If swallowed, dilute with water and induce vomiting.
- Get immediate medical attention.
- Never give fluids or induce vomiting if patient is unconscious or has convulsions.

1.2. Equipment - Fluorometer

1.2.1. Turner Designs Model 110 - reads dye concentrations directly in ppb. Operating instructions are contained in the Turner Designs Model 10 Operators manual, section 3-operations.

1.2.2. Turner Model 10-AU - reads dye concentrations in ppb. Operating instructions are contained in the Turner Designs Model 10-AU operating manual.

2. PRE-SURVEY

2.1. Surface Water Supplies

2.1.1. Identify all surface water supplies in or downstream from the study area.

2.1.2. Notify each water supply operator that may be affected in the study area, the DENR - Division of Water Resources regional water quality supervisor that a dye study is scheduled to be performed. Explain the reason for the study and inform the water treatment operator that DWR personnel will monitor dye concentrations at their water intake. If dye concentrations in the river exceed 10 ppb, the facility will be informed to shutdown their operation until river dye concentrations fall below 10 ppb. All efforts should be made to calculate a dye dosage that will result in a dye concentration at a water supply significantly below the 10 ppb.

2.2. Field Reconnaissance

1. Select dye sampling stations. Stations are selected based upon access, distance from the dose, and model requirements.
2. Locate all USGS gage stations in the study area or sites at which flows can be performed.
3. Determine if any dam structures that can regulate flow exist in the study area. If there is such a dam structure, a station is usually set up just upstream of the dam and an additional dose is made below the dam.

3. **DYE REQUIREMENTS (ESTIMATING DOSAGE)**

For Rhodamine WT 20 percent dye the dosage formula is:

$$V = 3.4 \cdot 10^{-4} \cdot [(Qm \cdot L)/Vm]^{0.93} \cdot Cp$$

Where:

- V** is the volume of dye, in liters
- Qm** is the maximum discharge in the reach, in cfs
- L** is the distance from injection to sampling point, in miles
- Vm** is the mean velocity, in fps
- Cp** is the peak concentrations desired in g/l

The volume of Rhodamine WT 20 percent dye required to produce a peak concentration of 1 g/l (ppb) can be determined from the nomograph in Figure 16 for a range of flow-reach conditions.

Measurement of Time of Travel and Dispersion by Dye Tracking

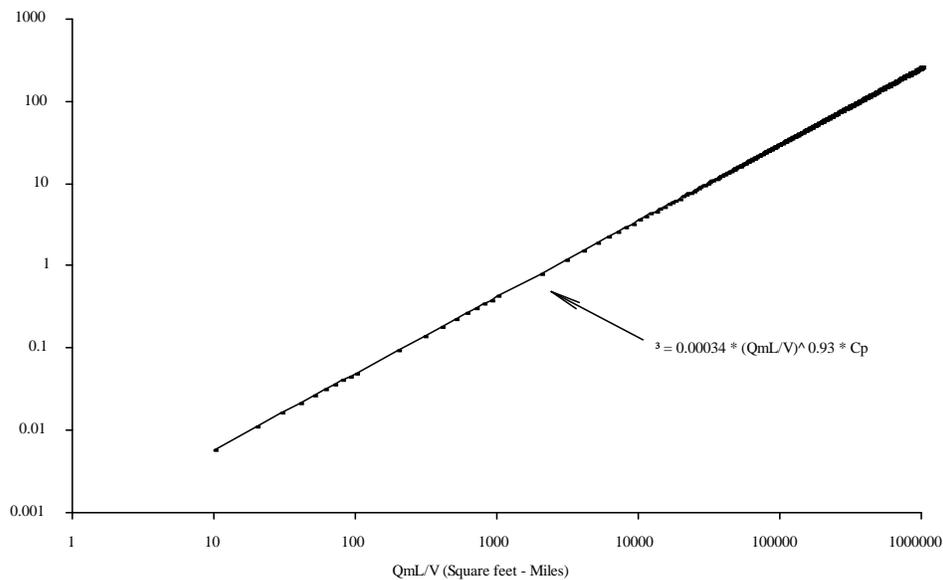


Figure 16. Nomograph for determining volume of dye necessary to produce peak concentration

4. INJECTION OF DYE

4.1. Injection Types

4.1.1. *Single Slug Injection*

- a. A single slug injection of dye is usually made in the center of the thread of flow.
- b. The desired quantity of dye as calculated in part 3 (above) can be poured into the stream from a container.

4.1.2. *Continuous injection*

- a. The desired quantity of dye is pumped into the water column at a fixed rate for a fixed time using an ISCO sampler or a peristaltic pump. Tubing is run from the pump to the desired dye injection point. Contaminated pump lines are first flushed with stream water and then placed in plastic bags for shipment back to the lab.

5. COLLECTION OF WATER SAMPLES

Samples should be taken in pre-numbered glass bottles by a hand sampler or by ISCO samplers. Care should be taken to collect samples in the peak concentration of the dye cloud, i.e. do not sample midstream if the dye cloud is along one stream bank.

5.1. Dye Sample Collection

5.1.1. *Data recorded on field sheets* (Figure 17)

- a. Station Location-Sampling Point
- b. Date
- c. Sample Bottle Number
- d. Time
- e. Name of Sampler

5.1.2. *Methods and Guidance*

- a. At least one background sample is needed for measurement of background fluorescence at each site in the study reach before the dye arrives.
- b. Sampling should begin early enough to determine the true dye peak.
- c. Sampling should continue until a peak has been determined; and until a decreasing trend has been clearly established.

5.5.1. *Sampling Schedule*

- a. The schedule for collecting samples at each sampling site is the most uncertain aspect of the plan.

- b. Estimates of the time to begin sampling, time intervals between samples, and the duration of sampling must be made, which will ensure adequate definition of the dye cloud passing each site. It is better to start with more frequent sampling and decrease frequency based on sampling results when travel times are unknown.
- c. An estimate of the time-of-travel between sampling sites is usually based on the cloud's movement to the first sampling site downstream of the injection site.

- a. Samples can be analyzed directly from the ISCO bottles, however, if the ISCO bottles are needed to continue sampling the samples can be transferred to numbered glass bottles.
- b. Label bottle racks.
- c. To reuse ISCO bottle, rinse three times using tap water or if tap water is unavailable uncontaminated stream water can be used.

6. FLUOROMETER USE

Refer to fluorometer manufacturer's operating instructions for specific procedures and service instructions. The Turner Designs Model 10 Fluorometer has two main scales; an X1 and an X100. When the fluorometer is in the X1 position, the sensitivity of the instrument is as indicated by the range lights. When the fluorometer is in the X100 position, the sensitivity of the instrument is 100 times that indicated by the range lights.

The scale for the Turner Designs Model 10 Fluorometer is:

Scale	Range	Concentration (ppb)
X100	X10	0-1
X100	minimum sensitivity	1-10
X1	X10	10-100
X1	minimum sensitivity	100-1000

6.1. Fluorometer Usage

6.1.1. *Calibration-*

(Fluorometric Procedures for Dye Tracing, Book 3, Chapter A12, Revised 1986.)

- a. Use a range of dye concentrations (ex. 1 g/l, 10 g/l, 50 g/l, 100 g/l with g/l=ppb) to calibrate the fluorometer prior to a dye study.
- b. Calibrate all fluorometer at 1 ppb . DWR preference.
- c. Calibrate fluorometer prior to taking out in the field, before running samples in the field, and before running samples in the lab

6.1.2. *Sample Collection*

- a. Prepare solution standards- Dye standards of known concentrations should be prepared in accordance with the U. S. Geological Survey's dye tracing procedures contained in Turn the fluorometer on and allow it to stabilize for at least 10 minutes.
- b. Use a distilled water blank to zero the instrument.
- c. Rinse the cuvette with water from the sample bottle before running that sample. Wipe off moisture from the outside of the cuvette.
- d. Run samples.
- e. Record measurements on the field sheet.
- f. Keep samples for future analysis, especially if peak concentration is questionable.

VIII. FLOW MEASUREMENT

1. INTRODUCTION

Stream-flow or discharge is defined as the volume rate of flow of the water including any sediment or other solids that may be mixed with it (Buchanan and Somers 1968). Stream-flow is usually expressed in cubic feet per second (cfs) and discharge flow in million gallons per day (MGD).

Several methods of determining flow are used by DWR. Most consist of wading into the stream with a top-setting flow rod and a vertical axis type flow meter shown as a propeller in Figure 18.

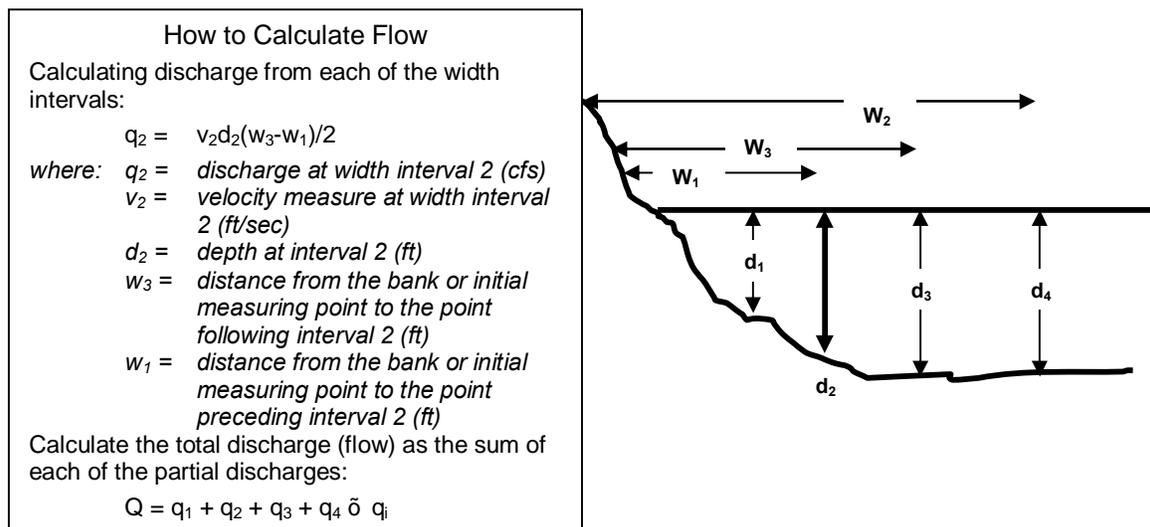
Other methods of determining flows (usually small, low-velocity flows) are:

- Volumetric method
- V-notched weir method
- Estimating flow mathematically method

The USGS maintains many gauging stations across the state and their stream-flow information is available in hardcopy and on-line. Discharge measurements using current meters are based on the equation:

$$Q=AV, \text{ where } Q=\text{Discharge, } A=\text{Area, } V=\text{Velocity}$$

It is as important to get good depth readings as it is to get good velocity readings.



DWR uses several different current meters and example is shown on Figure 18. The Price meter and the pygmy meter are vertical axis type meters, which use the number of revolutions over a period of time to calculate the water velocity. The Marsh McBirney meter works on the electromagnetics of the water passing by the meter.

Depth-measuring devices are used by DWR include two types of wading rods and a cable-winch bridge board. The top-setting wading rod easily sets the current meter at the proper height. With the other type of wading rod, the depth of the current meter must be calculated and set. The bridge board is used from a bridge handrail or the gunwale of a boat in streams and rivers where the water is too deep or the current is so strong that wading is dangerous or impossible. The winch has a depth-indicating gage.

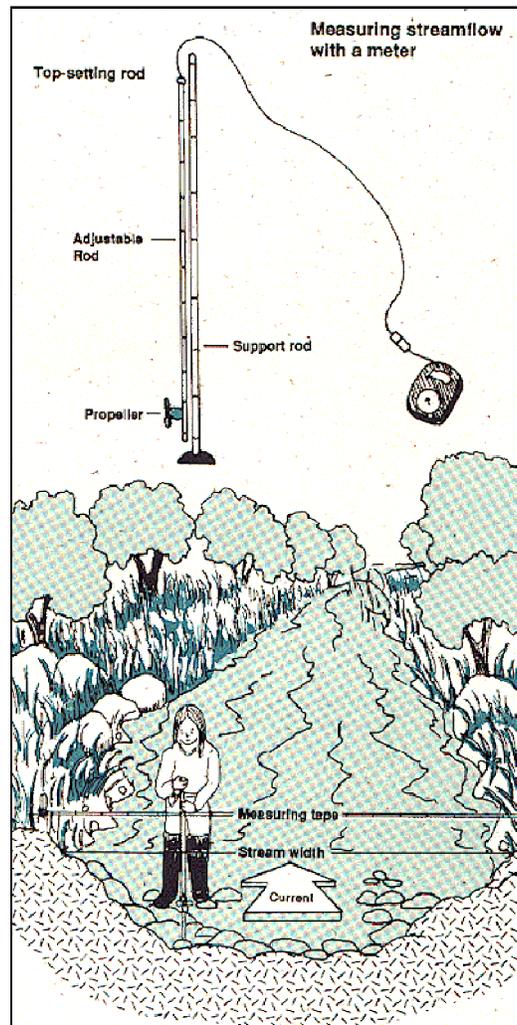


Figure 18. Instream Flow Measurement

2. ESTABLISHING AND USING A REFERENCE POINT

Before measuring any flow a stage reference point (RP) should be found or established. This point can be located on any stationary object over the water surface. Measure the distance from that point to the water surface. It is important to measure this distance before and after doing the flow measurement, as this will indicate any changes in the water level that occurred during the flow measurement.

Many bridges already have an established RP located somewhere on the bridge. These RPs have been established by the USGS and should be used if they can be located. If the RP on the bridge is not located, establish one. Make a mark on a structure over a deep pool near where the flow measurement is made. Clearly identify the mark so that others may use it also.

If many flows are to be done at a station, then a good reference point needs to be used. Each time a flow measurement is performed, record the tape down elevation. As multiple flows are compiled, a relationship between stage and flow can be graphed; eventually allowing flow estimates to be made by measuring the reference point. Occasional flows still need to be performed to make sure the tape down/flow relationship remains constant.

Use a metal tape and dimwap (weight) when measuring the tape down. Stand at the reference point. Let the tape feed out until it barely skims the water surface. Put the tape to the reference point and record the measurement. Be sure to add the length of the dimwap in the measurement.

The reference elevation should be measured to the top of the bolt, the top of the nail head, or to the top of the bridge rail (even if it is beveled). It is important to make your measurements from the exact same point each time.

3. FLOW EQUIPMENT

Price AA current meter	Small flathead screwdriver
Price pygmy meter	Hammer and nail
Top-setting wading rod	Spray paint (international orange)
Headset or beeper box	Torpedo weights (15 lb., 30 lb.)
Stopwatch	Bridge board
100 foot tape measure (in 1/10 ft)	Hand calculator
Chaining pins	
Stage tape measure with dingwap	
Clipboard	
Flow sheets	
Pencil	
Cleaning cloth	
Oil	
Flagging	

4. FLOW MEASUREMENT PROCEDURE

4.1. Flow Measurement Method

4.1.1. *Pre-Sampling:*

- a. Maintain all flow equipment in good working order. Refer to USGS publication, Discharge Measurements at Gauging Stations (Buchanan and Somers 1969).

Note: The spin test is a good indicator of flow equipment readiness.

- a. Check condition of flow equipment before leaving the office. An equipment checklist is helpful.
- b. Gather all equipment necessary to do the flow, see list in section 3 of this chapter.
- c. Select a reach of stream containing the following characteristics:
 - A straight reach with the threads of velocity parallel to each other
 - A stable stream bed free of large rocks, weeds, and protruding obstructions such as piers, which would create turbulence
 - A flat stream bed profile to eliminate vertical components of velocity.
 - Wait 15 minutes after moving rocks and vegetation prior to beginning flow measurements to allow stabilization of the stream flow.
- d. Establish a stage reference point (RP) (procedure described in following pages).
- e. Measure and record the starting stage.
- f. Install the current meter on the wading rod, attach the headphones - try a spin test (USGS publication, Smoot & Novak, 1968). Do the headphones click once for every revolution of the current meter? Make adjustments as necessary.
- g. Record pertinent information on the flow sheet (See Appendix 7). Include: stream name, date, time start, stage start, location of RP, person doing flow, person recording information, stream conditions.
- h. Determine the width of the stream. String a measuring tape across the stream perpendicular to the direction of the flow. Secure the tape on each bank with the chaining pins.
- i. Determine how many measurements are necessary to give an accurate total discharge. Measuring velocity at 20-30 equidistant points across the width of the stream is recommended. More measurement points should be chosen

in areas of significant depth or velocity change. Example: More measurements should be made in the area where the flow hugs one stream bank. A rule of thumb is that no area (point) being measured should contain more than 5% of the total flow of the stream.

- j. Looking upstream, record from which bank (right or left) the measurements are starting. Record the location on the tape of the starting bank. Example: The left bank starts at 1.5 feet on the tape.

4.1.2. Sampling Techniques

- a. Stand downstream and to the side so as not to obstruct the flow of the water to meter.
- b. Record the tape measure reading of the point.
- c. Measure the depth by placing the wading rod in the stream so that the base plate rests on the streambed. The depth is read from the graduated main rod and is estimated to the hundredth of a foot. Record depth readings on the flow sheet.
- d. Use the upper scale of the top setting rod to set the depth of the current meter. At depths of 2.5 feet and less, the average velocity is best measured at a point 0.6 of the depth from the water surface. Using the scale at the top of the wading rod automatically sets the current meter at the desired depth. To set the depth, press the rubber button on the flow rod. This releases the smaller rod. Move the smaller rod until the foot mark on it matches the appropriate tenth marker on the zero to ten scale at the top of the larger rod.
- e. At depths greater than 2.5 feet measure the velocity at 0.2 and 0.8 of the water depth (the average of these two velocities will later be recorded as a single value). The top-setting flow rod makes setting these two depths easy. Adjust the top scale readings to one half of the actual depth (this is the 0.8 reading) and to double the actual depth for the 0.2 reading.
- f. Start the stopwatch and count the number of revolutions (clicks on the headphones) for at least 40 seconds. Start the stopwatch on count number 0 and stop the watch exactly on a count, not a certain number of seconds.

- g. The pygmy meter is rated so that one revolution per second equals to one fps velocity. The Price meter rating is found in the top of the meter box. To use the Price meter table - count a certain number of revolutions: 1, 3, 5, 7, 10, 15, 20, 25, 30, 40 & 50. Compare to the time interval and read the velocity from the rating table. The Price meter also has a connection for measuring very high velocities where a signal is emitted from the meter for every 5 rotations instead of every single rotation.
- h. Record the number of revolutions and the time (to the nearest second) on the flow sheet.
- i. Move to the next point and repeat steps ~~g~~ until velocities in at least 20 cross sections have been measured.
- j. After the final measurement has been made, record the tape reading of the finishing bank.
- k. Measure and record the ending stage.
- l. Record the finishing clock time.
- m. Replace the current meter pivot with the traveling pin and return meter to box to prevent damage while traveling.

5. BRIDGE BOARD METHOD

5.1. Bridge Board Sampling Techniques and Supplies

5.1.1. *Equipment Needs*

- Clipboard with flow sheet and pencil
- Measuring tapes
- Duct tape
- Stage tape and dimwap
- Traffic safety cones and vests
- Bridge board assembly
- Price AA current meter (with tailpiece)
- Torpedo sounding weights (15 lb., 30 lb.)
- Headphones
- Stopwatch

5.1.2. *Bridge Pre-Sampling Setup*

(Refer to Buchanan & Somers, 1969, USGS publication)

- a. Assemble bridge board equipment. This involves attaching the assembled Price AA current meter and the appropriate torpedo sounding weight to the hanger end of the winch cable. The current meter's position on the hanger is dependent upon which torpedo weight is used.
- b. Attach the headphone jacks to the output terminals of the winch.

- c. Do a spin test to ensure that current meter is working properly.
- d. Determine the width of the stream and secure the measuring tape to the upstream handrail of the bridge with duct tape. More than one 100-foot tape may be necessary.
- e. Determine the distance interval of the 20 - 30 points necessary to make an accurate measurement. Example: Measure velocity every 2 feet on a 50-foot wide stream. Be ready to change the interval if velocity or depth changes significantly. Remember, no more than 5% of the flow in any one interval.
- f. Fill out flow sheet information (refer to step in section 4.1.1- ~~to~~ of the current meter procedure).

5.1.3 *Bridge Sampling Techniques*

- a. Measure the tape down from the reference point.
- b. Record the tape reading from the starting stream bank.
- c. Move to the first point to measure velocity. The bridge board rests on the handrail (guardrail) of the bridge.
- d. Zero the winch depth indicator by lowering the current meter until the cups of the meter are half in the water. Pull the zeroing armature out and rotate until the depth indicator reads zero. Because most bridge handrails are not level, make sure to zero the winch depth indicator at each point that a velocity measurement is made.
- e. Measure the depth of the water. To do this, lower the current meter until the cable goes slack. This indicates that the torpedo weight has hit something. Raise and lower the meter a couple of times to get a consistent depth reading for the bottom.
- f. Add 0.5 foot to the reading to get the actual depth of the water. This accounts for the torpedo weight that hangs 0.5 foot below the current meter (which was zeroed at the cups). Record the actual depth.
- g. At depths less than 2.5 feet, measure the velocity at 0.6 of the water depth (measured from the surface).
- h. At depths greater than 2.5 feet, measure the velocities at 0.2 and 0.8 of the water depth.
- i. Lower the current meter to the calculated depth.
- j. Measure the velocity by counting clicks (revolutions) for at least 40 seconds. Refer to velocity measurements using the Price AA current meter.
- k. Move to the next point and repeat steps ~~to~~.
- l. After the last velocity is measured, record the measurement of the finishing stream bank.

- m. Record the finish time.
- n. Measure and record the ending stage.
- o. Store all flow equipment properly.
- p. Compute the total discharge.

6. BOAT FLOW MEASUREMENT METHOD

Due to the danger because of boat traffic, extreme care should be taken when setting up for boat measurements. Locations where boat traffic is minimal should be chosen and any boats in the area should be warned off.

6.1. Boat Flow Sampling Techniques and Supplies

6.1.1. *Equipment Needs*

- Boat
- Rope (> width of stream)
- Bridge board assembly
- Headphones
- Stopwatch
- Clipboard with flow sheet and pencil
- Hand calculator
- Measuring tape - 100 foot
- Duct tape
- Stage tape and dingwap
- Cross piece assembly

6.1.2 *Boat Flow Pre-Sampling Setup*

(Refer to Buchanan & Somers, 1969, USGS publication Stations for more detailed instructions)

- a. Assemble bridge board equipment.
- b. Make a spin test.
- c. Prepare the boat for work. It takes a minimum of two people in the boat, one to operate the bridge board and one to calculate the meter depths and record the flow information. The cross piece attaches to the bow and holds the boat in position on the rope.
- d. Stretch and secure the rope across the stream channel, just over the water surface and perpendicular to the flow direction. Use duct tape to attach the measuring tape to the rope (an alternative is to use a rope marked at regular intervals).
- e. Fill out flow sheet information.
- f. Determine the interval of sampling points.

6.1.3 *Boat Flow Sampling Techniques*

- a. Record the tape measure reading at the starting stream bank.
- b. Measure the velocity at first point using the following steps. The bridge board rests on the gunwale of the boat.
 - Zero the winch depth indicator.
 - Lower the current meter until slack in the cable indicates the bottom.
 - Add 0.5 foot to the reading (to correct for the weight that hangs 0.5 foot below the meter).
 - Calculate the depths to set the current meter at 0.2, 0.8 (see step section in 4.1.1- ~~6.9~~) of current meter procedure).
 - Set the depth indicator at the proper depth.
 - Record the number of revolutions and the time interval at each depth.
 - Move boat to next point.
 - Repeat the steps under 6.9 until the entire stream has been measured.
- c. Record the tape measure reading of the finishing stream bank.
- d. Record the finish time.
- e. Compute the total discharge.

7. V-NOTCH WEIR METHOD

7.1. V-Notch Sampling Techniques and Supplies

7.1.1. *Equipment Needs*

- V-notch weir (> stream channel width)
- Vertical staff gage
- Carpenter's level
- Hammers (3 lb., 8 lb. sledge)
- Straight edge
- Graduated container
- Stopwatch

7.1.2. *V-Notch Flow Pre-Sampling Setup*

- a. Determine a good location for the weir plate. Avoid hard rock or loose sandy stream bottoms. Also avoid riffle areas where faster velocities erode the weir plate.
- b. Set up the vertical staff gage. The gage should be located in the upstream pool formed behind the weir. Use the carpenter's level to make sure that all faces of the gage are level. Because of the effect of drawdown, the gage should not be located too close to the weir plate.

- c. Use the sledge hammer to pound the weir plate into both banks so that the plate dams up all flow in the channel. Use the carpenter's level on all faces of the plate, it must be level. Make sure also that there is no water flowing around or under the weir plate.

7.1.3. *V-Notch Flow Sampling Procedures*

- a. Determine the zero point of the weir (very important reading):
 - Use the straight edge and level to measure a level line from the base of the angle on the weir plate back to the staff gage. Or if the distance from the top of the weir plate to the base of the angle is predetermined; then a level line is measured from the top of the plate to the gage and the distance subtracted.
 - Read the water level on the staff gage just at the point where the water starts to flow through the notch in the plate.
 - Let the water flowing through the V-notch stabilize.
- b. Read the staff gage.
- c. Determine the difference between the zero point on the staff gage and the water level flowing through the weir plate (read as the water level on the staff gage). This difference is known as head.
- d. Look at the flow table for the V-notch weir. Look up the corresponding flow for the particular head height. An alternative to using the flow table is the volumetric method (procedure described in following pages).
- e. Periodically clean the weir to prevent the buildup of sediments or solids around the notch. This buildup will affect the accuracy of the weir. Leaves are a problem to a V-notch weir. A leaf stuck at the base of the weir angle can cause a significant rise in water level.

8. VOLUMETRIC METHOD

8.1. Volumetric Sampling Techniques and Supplies

8.1.1. *Equipment Needs*

- Graduated container
- Stopwatch

8.1.2. *Volumetric Flow Procedure.*

- a. Mark the container to a known volume (examples: 1 gallon, 1 liter).
- b. Place the container under the discharge, collecting all flow.
- c. Time the interval needed to fill container to the volume mark.
- d. Empty the container.
- e. Repeat steps ~~b+c~~ several times.
- f. Average timed results.
- g. Calculate the flow rate as flow volume/time. Example: 1 gallon in 15 seconds.
- h. Convert flow to cfs or MGD.

9. MARSH MCBIRNEY MODEL 201 CURRENT METER

The principles of using the Marsh McBirney current meter are the same as using other current meters. The meter employs the velocity/area method of flow measurement. The sensor probe detects water velocity. The panel meter reads velocity in feet per second. The procedure for using the Marsh McBirney meter is the same as that described in Section 4 (Flow Measurement Procedure) but no calculations are needed as the flow is directly read on the instrument's screen.

This meter is used to measure small flows/low velocities. Because the probe has no moving parts, debris in the water has little effect on the reading. Another advantage is that the sensor probe attaches to the top-setting flow rod.

10. FLOW SHEET CALCULATIONS

10.1 Data Form

10.1.1. *Data to be Recorded on Data Sheet*

- Distance from the initial point
- Depth
- Time (in seconds)
- Revolution

10.1.2. *Calculations*

- a. Columns titled, velocity (mean in vertical), area, width, and discharge are calculated values.
- b. Calculate the width of each cross section. The width of the section is the sum of one-half the distance from the point of measurement to each adjacent point. Example: In the first

section the width is one-half the distance to the adjacent point plus one half the distance to the stream bank.

- c. Calculate the velocity in each cross section. In depths of greater than 2.5 feet, two velocity measurements are taken in each cross section (0.2, 0.8). Average the two readings and record in column. This depends on the current meter in use:
 - The Price pygmy meter - the number of revolutions divided by the seconds.
 - The Price AA meter - The velocity is taken directly from the meter rating table. Not all Price AA meters use the same rating table-be sure that you have the correct table for the meter you are using.
 - The Marsh McBirney meter - The velocity is read from the panel as feet per second.
- d. Calculate the cross-sectional area. Multiply the width times the depth.
- e. Calculate the discharge of each cross section. Multiply the cross-sectional area by its velocity.
- f. Calculate the total discharge of the stream. Add all the cross-sectional discharges together.
- g. Record the average velocity of the stream. Divide the total discharge by the total area.

11. OPEN CHANNEL FLOW MEASUREMENT METHOD

11.1 Introduction

The following section provides a brief overview of methods for determining flow in an open channel. For more detailed information regarding open channel flow measurements, refer to the references section.

Open channel flow can be defined as flow in any channel in which liquid flows with a free surface. Open channels are generally used in moving fluids at most municipal treatment facilities, industrial waste treatment operations and in most irrigation applications. An open channel can also be a stream or a ditch. Open channel flow is typically measured by the use of a calibrated restriction device placed in the channel that affects the surface level of the liquid as it moves past the restriction. This type of open channel measuring device is referred to as a "primary" device. The known dimensions and physical characteristics of the restriction device are used to correlate a relationship between water surface level and flow. After the water level/flow relationship has been established, the flow in the open channel can be easily measured by manually sighting the height of the liquid's surface level against a calibrated scale (staff) and then referring to the appropriate rating curve or table.

The following are the most commonly used types of "primary" open channel flow measuring devices, (restriction devices):

- 11.1.1 Weir: a dam constructed across an open channel, over which liquid flows through an opening or notch. The most commonly used types are rectangular, trapezoidal and triangular.
- 11.1.2. Flume: a specially shaped open channel, designed to change the channel area or slope, resulting in an increase velocity and surface level of the liquid flowing through it. The most commonly used types are: Parshall and Palmer-Bowlus

11.2 Flow Meter

- a. A flow meter is a mechanical device used to measure the liquid level in the channel and convert the level into a corresponding flow rate
- b. A stage recorder is a mechanical device used to record the surface level of the liquid over a period of time

Note: Measuring flow in an open channel by means of a weir or flume is a simple function of surface level and is the most basic and inexpensive method available. However, if continuous stage or flow recording is required, then the use of a stage recorder and/or a flow meter in conjunction with the primary device may be necessary. Some of the more commonly used methods employed by these devices to determine the surface level of a liquid are floats, dipping probes, ultrasonic sensors, and bubblers.

IX. BATHYMETRY

1. PROCEDURES

Recording fathometers are used to provide bathymetric traces of water depths. Since water depths are time dependent (especially in tidal areas) the date and time of all traces should be noted. Operating manuals provide operation and calibration procedures to be followed. In particular, tide and draft adjustments provide calibration in regard to the respective tidal amplitude and sensor probe depth. All traces should be noted with transect description, chart speed, direction of travel, and pertinent reference points and then indexed to a site map. When working in tidal areas, a water stage recorder should be positioned to provide a histogram of water levels to correlate with the bathymetric trace.

During the initial setup of each survey, the fathometer calibration should be checked against a field measurement of water depth made using a graduated sounding line.

2. EQUIPMENT AVAILABLE

The following equipment is available for bathymetric surveys:

- Water level recorder and/or referenced gauging stations(s)
- Depth gauge
- Calibrated sounding line(s)

3. SPECIFIC EQUIPMENT QUALITY CONTROL PROCEDURES

Number all equipment and keep a record of maintenance and calibration procedures. Use the following steps to maintain and calibrate bathymetric measurement equipment:

3.1. Recording fathometers:

- 3.1.1. Calibrate and maintain according to the manufacturer's instructions before use. The chart speed should be checked against a reliable time source before the instrument is sent to the field.
- 3.1.2. Check daily in the field against a field measurement of water depth using a calibrated sounding line.
- 3.1.3. Clean daily after use and before storing.

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

X. WATER QUALITY VESSEL OPERATION

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

1. BOAT SAFETY

1.1. Supplies Needed On-Board

1. **Fire Extinguishers** - before operating boat, familiarize yourself with where the fire extinguisher is located. Check to make sure that it is fully charged.
2. **Sound Producing Devices** - boats should be equipped with a can type air horn or a manually operated whistle.
3. **Paddles or Oars** - all boats should be equipped with oars or paddles.
4. **Visual Distress Signals** - when operating boats in coastal waters, the boat must be equipped with a flare kit. The kit should include hand held flares and a flare gun for aerial type flares.
5. **PFD's (Personal Floatation Devices)** - all DWR employees are required to wear life preservers at all times while on board DWR boats. Boats will be equipped with a type 1, 2, or 3 PFD of suitable size for each person on board and a throw-able floatation device (throw cushion, flotation ring).
6. **Lights** - when operating a boat at night, the boat must display the front green and red navigational light and the rear beacon light. If planning to operate at night, the lights should be checked before leaving the loading area.

1.2 Safety Check

1. **Weather** - check weather reports before leaving shore and remain watchful for signs of bad weather. Tune into the National Weather Service Report, on a Marine radio, periodically to check weather conditions, small craft advisories, gale warnings, etc. Do not go out on the water during lightning storms.
2. **Care and Maintenance** - all equipment and supplies should be properly secured. Keep decks and other spaces clean, free of clutter and trash. The vessel should be free of fire hazards with clean bilges and in good condition. Inspection and required maintenance on a regular schedule will ensure the hull and superstructure remain sound.

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

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

2. FIXED MOUNT/CONSOLE TYPE BOATS

2.1. Trailer

2.1.1. *Pre-Trip Check and Preparation*

- a. Install 1 or 2" trailer ball to trailer hitch depending on the trailer.
- b. Unscrew clamping mechanism on boat trailer tongue.
- c. Back vehicle up to boat trailer, with trailer tongue directly over the center of the trailer ball.
- d. Lower trailer jack so that the trailer tongue fits over the trailer ball.
- e. Screw down, or tighten, the clamping mechanism (all the way) onto the trailer ball. Lock with a 2640 Master Lock.
- f. Hook up both safety chains by crossing the chains and hooking to holes on the trailer hitch. Do not tow boat without safety chains.
- h. Hook up brake line cable to eye bolt attached to the vehicle.
- i. Plug in trailer lights and check the lights for proper operation.
- j. Secure gunwhale boat strap.
- k. Check the bow eye to make sure safety chain is hooked up and winch is locked down securely.
- l. Periodically check the clamping mechanism on the trailer tongue to assure that it is screwed down all the way.
- m. Conduct an inspection walk around the boat and trailer:
 - 1) Test to see if the boat motor starts before traveling.
 - 2) Check level of the engine oil on 4-cycle boat motors.
 - 3) Check the trailer tire pressure and adjust if necessary.
 - 4) Check the condition of the axel grease. Add grease as needed.
- n. When traveling, stop and check the trailer and boat; retighten boat straps as needed. Feel the trailer bearings to see if they are hot. If hot, they probably need to be greased or replaced.
- o. Trailer slowly over speed bumps and holes.

- p. When backing into parking areas, do not let back of trailer come in contact with curb to avoid damaging license plate bracket or trailer lights.

2.2. Boat Launching

2.2.1. *Unloading*

- a. Upon arrival at the boat ramp check the ramp to make sure it is suitable for launching including checking that the water level is high enough for proper launching.
- b. **Install all the boat plugs**; check inside the bilge to make sure that the plug is installed.
- c. Remove the boat strap.
- d. Load the boat with equipment.
- e. Unplug trailer lights
- f. Make sure the motor is in the "up" position before launching the boat.
- g. Keep winch "locked" until boat is in the water.
- h. When the boat is in the water lower the motor and then start the motor (see motor operating instructions).
- i. While one person is operating the boat another person should be manning the trailer winch.
- j. Unhook winch cable from bow eye, but do not remove safety chain until the boat is running and idling.
- k. If needed, back the vehicle up slightly and press the brake to bump the boat off the trailer.
- l. When boat is clear from the trailer, pull the vehicle out of the ramp **slowly** and park it.

2.2.2. *Loading*

- a. Slowly back the trailer into the water so that the center "guide roller" is visible above water.
- b. Line up the boat with the trailer and **very slowly** ease the bow of the boat onto the center roller. If boat is off center of the trailer, back up and try again.
- c. **Do not** approach trailer at a speed that will damage the boat hull or trailer if the center roller is missed!
- d. The person manning the trailer winch should signal the boat operator to go left or right, or to tell the boat driver to back off if they are going to miss the center roller.
- e. Once the bow is on the center roller, slowly advance the boat up onto the trailer as far as it will go. If it does not reach the stanchion then hold the boat in position until the person manning the winch can get out enough cable to hook to the bow eye.

- f. Once the cable is hooked and tension is maintained then power down the motor and cut it off.
- g. Winch the boat onto the trailer until the bow is snug against the stanchion.
- h. Lock down the winch gear.
- i. Raise the motor to the "up" position and flip down the tilt lock bar, then lower the motor until it presses against the tilt lock bar.
- j. **Slowly** drive out of the boat ramp to the parking lot.
- k. Remove the boat plugs.
- l. Unload the boat.
- m. Hook up boat strap.
- n. Plug in trailer lights.
- o. Make sure that all aerials and/or bimini tops are down.
- p. Walk around trailer and **double check everything!**

2.3. Boat Operation

2.3.1. *Fueling*

- a. Fill oil reservoir to required fill level with appropriate motor oil.
- b. When fueling boats with 2 cycle outboard motors without an oil reservoir add one pint of 2 cycle outboard motor oil to the gas tank for every six gallons of gasoline. Use marine fuel stabilizer in all fuel tanks.
- c. Fill tanks to their maximum fill level.

2.3.2. *Motor Operation*

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

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

3. SMALL BOATS WITH PORTABLE MOTORS

3.1. Trailing

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

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

3.2. Boat Operation

3.2.1. *Fueling*

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

3.2.2. *Motor Operation*

- a. Select the proper motor for the boat that is to be used.
 - 5 hp for Jon boat and 12' Alumacraft,
 - 15 or 25 hp for 14' Alumacrafts.
- b. Place outboard motor in the **center of the transom** and completely tighten the clamping screws on the outboard motor.
- c. Place tank in boat and connect to motor to assure proper fitting.
- d. Run a chain through the gas tank handle, then through the handle on the outboard motor, and then through the hole in the boat and lock with a pad lock.
- e. Secure the motor to one side with a bungee cord to keep motor from swaying back and forth while going down the road.
- f. To run: connect fuel line to motor, and pump primer ball.
- g. Pull choke knob if motor is "cold".
- h. Turn tiller throttle lever to "start" position.
- i. Pull starter cord. If motor does not start after one or two pulls then pump the primer ball and try again.
- j. Once motor starts push choke lever in and let run for about a minute then idle down with throttle lever.

- k. To put motor in gear, make sure motor is idling low and pull the gear lever forward to go in a forward direction. To go in reverse, push gear lever backward to reverse position with a brief stop in neutral.
- l. Once motor is in gear then throttle up the motor, once the boat is planned out back off of the throttle about 1/4 turn.
- m. **Do not run the motor at full throttle, run at 3/4 throttle.**
- n. To turn the motor off, press the red kill button located next to the choke lever. On some motors the kill button is located on the end of the tiller.

4. TROUBLESHOOTING: FOR ALL BOATS

- 4.1. Problem - No power to starter
 - a. Check to see if "PERKO" switch is on the "ALL" position.
 - b. Check to see if throttle lever is in neutral.
 - c. Check battery terminal connections.
 - d. Check main fuse in outboard motor.
 - e. Check fuses inside console.
- 4.2. Problem - Motor is Turning Over But Will Not "Fire" or Start
 - a. Check to see if gas line is connected to motor.
 - b. Check to see that primer ball has been pumped until tight.
 - c. Check to make sure "deadman's" or kill button is clipped.
 - d. Make sure air vent screw is open on gas can.
 - e. Check spark plug wires, replace spark plugs (they may be fouled)
- 4.3. Problem - No Water is Coming Out of Flow Hole
 - a. **Do not** continue running motor. Shut down and attempt to unclog flow hole with a coat hanger or similar object.
 - b. If problem persists, do not use boat. If out on the water when this occurs, get towed back to boat ramp.
- 4.4. Problem - Extreme Cavitations or "Porpoising"
 - a. Adjust trim with up and down button on throttle lever.
 - b. Make sure there is not an excessive amount of water in bilge.
 - c. Adjust the weight distribution of equipment and personnel in the boat (trim the boat up and shift weight).

XI. LAKES SAMPLING

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

1. FIELD PREPARATION

1.1. Pre-Sample Preparation

- a. Preparation of the lake sampling packet.
 1. Sample tags and lab sheets must be legibly hand written with permanent black ink. Adhesive labels for the sample tags may be prepared on a laser printer.
 2. Include maps showing the locations of the sampling stations. Electronic copies of lake maps are on the ISB shared drive (Lake Maps folder).
 3. Include a copy of the Field Observation form (Figure 19).
 4. Include special instructions and point-of-contact information as needed.
- b. Contact responsible parties at all publicly owned lakes several days in advance of sampling. Contact names are in the particular lakes file or in the Lakes Database. Changes in contact information will be noted and provided to the Lakes Database Administrator so that the database can be updated.
- c. Confirm availability and working condition of boats, motors, vehicles, and Hydrolab/YSI.
- d. Verify the lake stations on the map with the station numbers on the lab sheets and tags.
- e. Always include extra bottles in case of accidents, defective bottles, and/or discovery of algal blooms or other environmental conditions that justify additional samples.

1.2. Field Equipment Needed

Aquatic Plant & Algal Bloom Report Forms	Cooler(s) with ice
Field Observation & Stratified Data Forms	Lab Sheets and Tags in sealed bag
Preservatives- Lugols solution, H ₂ SO ₄ , HNO ₃	Hydrolab/YSI Meters
Labline with Rope	Camera
Sample Bottles	Calibrated backup meters
Pens and Pencils	Maps
Life Jackets	Boat Oars
Gas Tank for boat	Boat Plug & Anchor
Winter- cold weather suit (as needed)	First aid/safety box
Electric motor & 2 fully charged batteries (if needed)	
Secchi Disc with line marked in 1 centimeter increments	

Calibration Materials-(e.g. *Calibration and D.O. sheets, pH and conductivity standards, meter manuals*).

1.3. Field Sheets

- a. *Stratified Field Sheets*: Make sure that stratified field sheets (Figure 5, page 25) are carried to the lake stations along with pens or any non-erasable ink for writing and a clipboard. Clearly write the station number, date, time, depth, dissolved oxygen, temperature, pH, conductivity, and secchi are recorded on the field sheet. At the top of the field sheet record, the name of the water body, which meter is used, and the names of the field samplers is also recorded.
- b. *Field Observation Form*: In addition, a separate form is to be filled out for all of the ambient lakes (Figure 19). This form requests information about the use support status, restoration activities, weather conditions, the watershed, and lake water quality.
- c. After sampling, both of these forms are filed in the current lake files in the Intensive Survey Branch. Data are entered into the lakes database within 72 hours of the lake trip.

FIELD OBSERVATION FORM Lake Name _____
 Page 1 of 2 Samplers Name: _____ Date: _____

WEATHER CONDITIONS

Air Temperature	% Cloud Cover	Wind Direction (from)	Wind Velocity	Rainfall (last 48 hrs)
<input type="checkbox"/> <60°	<input type="checkbox"/> 0-25%	_____	<input type="checkbox"/> <10 mph	<input type="checkbox"/> None
<input type="checkbox"/> 60-70°	<input type="checkbox"/> 25-50%		<input type="checkbox"/> 10-20 mph	<input type="checkbox"/> < ¼ inch
<input type="checkbox"/> 75-90°	<input type="checkbox"/> 50-75%		<input type="checkbox"/> >20 mph	<input type="checkbox"/> ¼ - 1 inch
<input type="checkbox"/> 90°	<input type="checkbox"/> 75-100%			<input type="checkbox"/> >1 inch

SHORELINE AND WATERSHED OBSERVATIONS
 Please describe *development* around the lake shore:

Type of Development	Density/Intensity	% of Shoreline Developed
<input type="checkbox"/> Residential/Urban	<input type="checkbox"/> Slight	<input type="checkbox"/> 0-25%
<input type="checkbox"/> Commercial/Industrial	<input type="checkbox"/> Moderate	<input type="checkbox"/> 25-50%
	<input type="checkbox"/> heavy	<input type="checkbox"/> 50-75%
		<input type="checkbox"/> 75-100%

Please check *land uses* observed in the watershed:

- Agriculture (specify if possible)
 - Crop production
 - Pasture land
 - Feedlots/Animal production
- Forest
- Wetlands
- Urban/Residential
- Commercial/Industrial

LAKE QUALITY
 Please check the one statement that best describes the *physical condition* of the lake water today:

- Crystal clear water.
- Not quite crystal clear, a little algae/suspended sediment visible.
- Definite algal greenness, yellowness, or brownness apparent.
- High algal/sediment levels with one or more of the following: floating scums on lake or washed up on shore; strong foul odor; or fish kill.

Please check the one statement that best describes the *aquatic macrophyte* community:

- None observed
- Small amount of vegetation evident along shoreline and/or headwaters of the lake; <25% of the lake's total surface area covered.
- Macrophytes extend out from shoreline well into the lake; 25-50% of the surface area covered.
- Dense growths of several species cover more than 50% of the surface area.
- Nuisance levels of a single species cover more than 50% of the surface area.

Please check the one statement that best describes your *opinion* of how suitable the lake water is for recreation and aesthetic enjoyment today:

- Beautiful, could not be nicer.
- Very minor aesthetic problems; excellent for swimming, boating, enjoyment.
- Swimming and aesthetic enjoyment slightly impaired because of levels of algae/sediment/or weeds (please indicate which).
- Desire to swim and level of enjoyment of the lake substantially reduced because of algae/sediment/or weeds (please indicate which).
- Swimming and aesthetic enjoyment of the lake nearly impossible because of algae/sediment/or weeds (please indicate which).

Figure 19. Field Observations Form

LAKE NAME: _____
Page 2 of 2

Designated Use Classification: _____

Supplemental Classification: _____

USE SUPPORT STATUS
Designated uses appear to be:

- Fully supported.
- Fully supported, but threatened (impairment could result if pollution controls are not implemented).
- Partially supported
- Not supported

If uses are not fully supported, what pollutants or conditions are *causing* impairment (check all that apply):

- Nutrients
- Situation
- Flow alteration
- Suspended solids
- Noxious aquatic plants
- Organic enrichment/low DO
- Thermal modification
- Filling and draining
- Other (please specify)

If uses are not fully supported, what *sources* of pollutants contribute to use impairment:

POINT SOURCES

- Industrial
- Municipal
- Municipal pretreatment
- Other point sources (specify)

NONPOINT SOURCES

- Agriculture (specify if possible)
 - Crop production
 - Pasture land
 - Feedlots
 - Aquaculture
 - Other (specify)
- Silviculture
- Construction/Land development
- Urban runoff
- Mining/resource extraction
- Land disposal of waste (e.g. landfills, wastewater and sludge application, on-site septic tanks, etc.)
- Hydrologic/habitat modification (e.g. canalization, dredging, flow regulation, etc.)
- In-place contaminants
- Recreational activities (e.g. motor boating)
- Other nonpoint sources (please specify):
- Source of impairment unknown

RESTORATION ACTIVITIES
Please describe any lake restoration or water quality management activities that have taken place:

Figure 19. Field Observations Form (continued)

2. LAKE DATA COLLECTION

2.1 Lake Physical Data Collection Methods

- a. Secchi depth measurement is taken as described in Chapter III, Section 6.
- b. Dissolved oxygen, water temperature, conductivity and pH are measured with a multiprobe (Hydrolab) meter beginning at the surface of the lake (0.15 meters from the surface).
- c. From the surface to either the bottom of the lake or to a depth of 10 meters, physical measurements are recorded at 1- meter increments
- d. Below ten meters, physical measurements are recorded at 5-meter increments until the bottom of the lake is reached

2.2. Lake Water Sample Collection

2.2.1 *Description*

- a. Samples will be collected at the surface, photic zone, or at the bottom, which are described below and in detail in Chapter I.
- b. If "SUR" is part of the station number, the sample is to be collected at the surface. If "BOT" is part of the station number, the sample is collected one foot above the bottom. If "SUR" or "BOT" doesn't accompany the station number, the sample is collected in the photic zone.
- c. As with all samples for laboratory analysis, a lab sheet must be completed as described in Chapter II, Section 1 of this SOP.
- d. Detailed definitions of these parameters and methods of collection found in Chapter I, Section 3.

2.2.2. *Types of Typical Lake Samples*

- a. **Surface Grab** samples (chloride, hardness, fecal coliform bacteria, and metals) are collected 0.15 meters below the water's surface and this can be done by hand dipping the bottle. The bottle top and bottle opening should be protected from contamination. Grasp the bottle near the base and plunge it mouth down into the water, avoiding surface scum. Position the bottle away from the hand of the collector, the shore, the side of the sampling platform, or boat.
- b. **Photic Zone** . the photic zone is defined as the column of water in the lake from the surface down to a depth equal to twice the secchi depth measurement. Photic zone samples (residue, turbidity, chlorophyll *a*, nutrients, and phytoplankton) are collected by raising and lowering the Labline at a steady speed within the photic zone until it is full. A description of this procedure is given in this SOP in Chapter I, Section 3.2.3.

- c. **Bottom sampling** (nutrients) is accomplished by inserting the two plugs in the top of the Labline and lowering it just above the bottom of the lake. This needs to be done gently as not to stir up sediments on the bottom. The plugs can be released by firmly jerking the rope on the Labline. Wait until the Labline is full before bringing it back to the surface. This can be determined by observing air bubbles from the sampler rising to the lake surface, the stopping or feeling the weight of the Labline at the end of the rope.

2.2.3 *Field Records and Information*

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

2.3. Typical Lake Sampling Parameters

Below is a list of typical lake water sample types. Descriptions on how samples are preserved and collected are found in Chapter I, section 3, section water samples as well as more detailed descriptions can be found in this SOP in the Sample Collection Section (Chapter IV).

2.3.1. *Physical Parameters include*

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

2.3.2. *Chemical Parameters include*

Nutrients	Residue
Turbidity	Chloride
Magnesium	Calcium
Metals	Chlorophyll a

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

2.3.3. *Biological Parameters*

- a. Fecal coliform bacteria: Water samples are collected at the surface of the lake.
- b. Phytoplankton: Water samples are generally collected as a photic zone sample. Bloom samples may be collected at the surface of the lake, as needed.
- c. Aquatic Plants: Use the Aquatic Plant Report Form supplied by the Ecosystems Branch of the Environmental Sciences Branch and submit it along with a specimen if there appears to be problematic aquatic plants or for identification. Refer to the Aquatic Plant Report Form for collection and preservation of aquatic weeds. Include a map of the location showing where the plant specimen(s) were collected.
- d. AGPT (Algal Growth Potential Test): These samples are collected after consultation with EPA since they perform the tests. The bottles (1 liter) are furnished by EPA as are the tags and coolers. The samples are collected in the photic zone and no preservative is used. The samples are shipped back to the EPA Athens, GA laboratory for analysis. The address and telephone number is: Bob Quinn, U.S. EPA, Region IV, Environmental Services Division, Athens, Georgia 30613, (706) 546-2420.

2.3.4 Lab and Field Sheets: All lab and field sheets should be **legibly** filled out with applicable dates, times, depths, etc.

- a. The same time is recorded on both field and lab sheets for the same station. A field observation sheet should also be filled out and any other notable features recorded.
- b. Any notes of unusual observations of lake water quality or shoreline activities that could impact water quality should also be submitted.
- c. Field sheets and filed observations sheets along with camera are to be submitted to the Lakes Database Manager upon return from the field.

3. LAKE DATA MANAGEMENT

3.1 Data specific to the Intensive Survey Branch Lake Monitoring Program are warehoused in the Lakes Database. This database is maintained by the Lake Database Administrator. The responsibility of the Lake Database Administrator includes entry of data, verification data entry accuracy and reporting issues related to the functioning of the database to the ESS IT staff.

- a. Physical field data are entered into the ISB's Lakes Database within 24 hours of receipt from the field sampling team.

- b. Chemistry results from the DWR laboratory are entered into the Lakes Database within 72 hours of receipt from the laboratory.
- c. Lake data which have been entered into the Lakes Database but not checked for input accuracy and/or completeness are designated ~~P~~q for ~~Provisional~~q
- d. Lake data which has been reviewed and verified for input accuracy and completeness are indicated with the designation ~~A~~q for ~~Accepted~~q

XII. SEDIMENT OXYGEN DEMAND

1. GENERAL DESCRIPTION OF SOD TEST

Sediment Oxygen Demand (SOD) is one of the more significant variables in water quality modeling evaluations for determining stream assimilative capacity. SOD data are primarily used for waste-load allocation purposes in the evaluation of receiving waters.

The SOD test involves placing an SOD chamber on the bottom sediment, securing it to prevent water infiltration and monitoring oxygen change within the chamber. A dissolved oxygen sensor inside the chamber measures the rate of decrease in oxygen that is used by organic materials in the bottom sediments over a given period of time. A standard SOD test includes seven SOD chambers of which two are water column control (blank) chambers and five are replicate SOD chambers (Figure 20). The blank chambers, used to determine water column respiration rate, have bottom plates that prevent bottom sediment from contacting the water in the chamber. The SOD replicate chambers have open bottoms allowing the internal water to circulate over the bottom sediment. The rate of oxygen change in the replicate SOD chambers minus the water column respiration of the blank chambers equals the SOD rate.

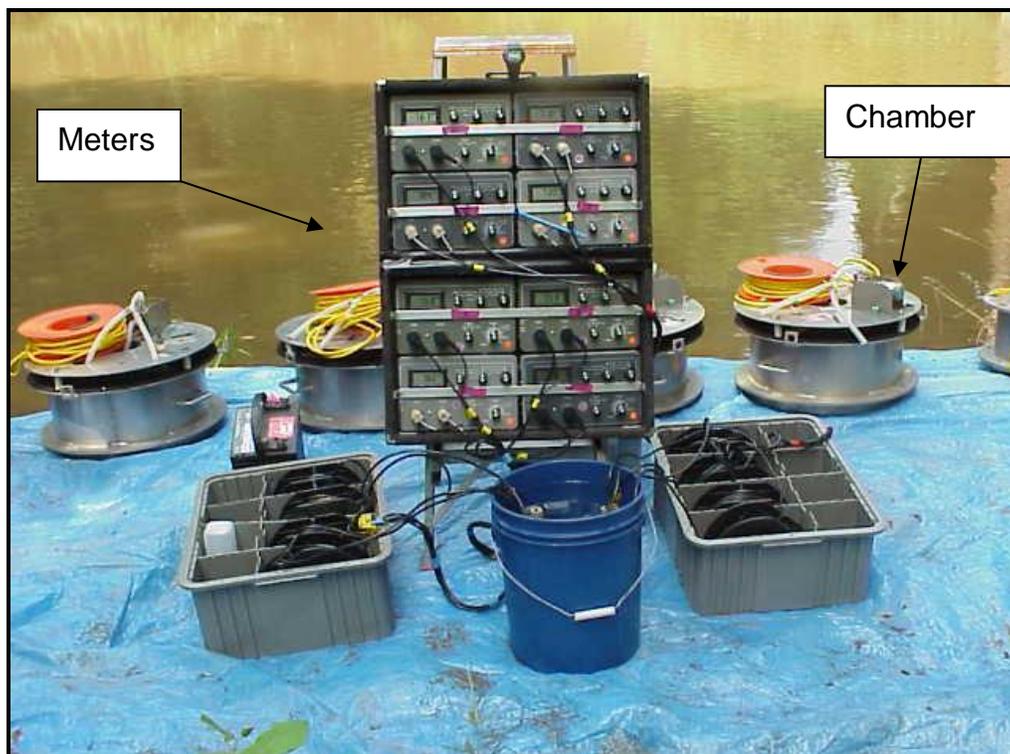


Figure 20. SOD Equipment

1.1. SOD Rate Formula:

The SOD rate for any study location is then calculated by using the SOD rate formula:

$$\beta \times (K \times V) \div A = \text{gr } O_2/m^2/hr.$$

where: β = rate of change in D.O. as mg O_2 /L/min.

V = chamber volume in liters

A = chamber area in meters square

K = 0.06 (constant) converts liters to square meters

SOD rates are dependent on benthic metabolic processes, sediment particle size, stream velocity and other factors. SOD rates from 77 in-situ tests performed at locations with various substrate compositions are presented in Sediment Oxygen Demand, Processes, Modeling and Measurement (Murphy and Hicks, 1986, p. 318). An example SOD Excel Worksheet is provided at the end of this section for reference.

- 1.2. SOD Equipment List . Due to the amount of gear and equipment necessary to successfully complete SOD tests, a checklist is recommended when preparing for testing. See Figure 21.
- 1.3. Site Evaluation . Each site should be visited and checked out to determine if sediment is suitable in the area under investigation. Figure 22 is the SOD Site Evaluation Form that should be completed for each location.

2. FIELD CALIBRATION DISSOLVED OXYGEN METERS

An initial calibration is performed on the YSI 58 meters prior to the SOD test and a terminal calibration is performed on the meters after the test is completed. All calibration data is recorded on the SOD Calibration Forms (Figure 23). The need for accuracy is paramount for SOD evaluations due to the extremely small increments of change in D.O. measured during the test (+/- 0.01 mg/L). Because of the number of meters being calibrated on site and the extreme accuracy required for SOD testing, initial and terminal calibration procedures in this section vary from other D.O. meter calibration methods in this document. SOD meters are calibrated using the Modified Winkler Azide method as opposed to saturated air calibration methods.

Figure 21. SOD Equipment List:

CHAMBERS:

- FIVE REP CHAMBERS ALUM
- TWO BLANK CHAMBERS ALUM
- ONE CLEAR BLANK CHAMBER
- TUBES ON CHAMBERS
- FLOW RESTRICTORS IN TUBES
- SPACERS ON CHAMBER
- BATTERY CLIPS ON DC LEADS
- RUBBER SEALS OK
- SILICON SEALS OK
- TEST PUMPS
- CHAMBER COLLARS
- 3 BATTERIES MINIMUM (CHARGED)
- CHAMBER HARNESS AND FLOATS
- STOPPERS -#1 & #1½

METERS:

- DO METERS
- NEW MEMBRANES ON PROBES
- CONDO METER
- MEMBRANES & ELECTROLITE KIT
- 100qCABLES WITH PROBES
- EXTRA 50qCABLE AND PROBE
- CALIBRATION & SOD FIELD SHEETS
- COPPER BATTERY BARS
- STAND FOR METERS (BOAT OR BANK)
- %G+CLAMPS (LARGE) - BOAT METER STAND
- BUNGIES FOR METER STAND
- BOARD FOR METER STAND MOUNT

WINKLER

- WINKLER KIT (CHECKED OUT)
- EXTRA CHEMICALS
- BURET AND GLASSWARE
- EXTRA BURET AND GLASSWARE
- BUCKET FOR CALIBRATION
- WATER CALIBRATION

- BURET STAND
- BURET WIRE
- STARCH

PERSONAL EQUIPMENT

- RAIN GEAR
- BOOTS
- WATCH
- COOLER AND ICE
- WASH WATER/SOAP
- INSECT REPELLANT
- SUN SCREEN
- HAT
- SUN GLASSES
- FOOD/DRINKS/WATER

MISC. EQUIPMENT

- CAMERA AND ACCESSORIES
- SEDIMENT JARS AND TAGS
- SOD TOOL BOX
- MAPS
- CALCULATOR/PENCILS
- TARPS
- FIRST AID KIT
- MACH, SHOVEL
- CHAIRS, BOX
- ROPES FOR BANK OR BOAT
- CELL PHONE
- COLORED TAPE
- BATTERY TESTER
- %G+CLAMPS (SMALL) FOR REP LIDS
- FIELD LOG FOR SOD TEST
- PLASTIC CRATES

SOD SITE EVALUATION	
Site Location - _____ _____	
Date: _____	Time: _____
Site Description - _____ _____ _____ _____	
Topo Map # _____	% From Right Bank (facing US) _____
Weather - _____ _____	
Bank Description - _____ _____	
Depth - _____	Velocity (fps) - _____
Sediment Description - _____ _____	
Bottom Topography - _____	
Water Description (turbid, clear, etc.) _____ _____	
Site Schematic:	

Figure 22. SOD SITE EVALUATION FORM

All meters are to be air calibrated prior to field operations (and on-site calibration) to assure all meters are functioning properly and stabilized.

The procedures are as follows:

2.1. Calibration Procedures

2.1.1 *INITIAL CALIBRATION Method*

- a. Turn off all electronics (cellular phones, depth finders, etc.) prior to reading and calibrating meters.
- b. Connect the D.O. probe to the probe receptacle of the YSI 58 meter and screw the retaining ring finger tight.
- c. Connect the D.O. stirrer to the stirrer receptacle of the YSI 58 meter and screw the retaining ring finger tight. Check the stirrer battery condition by turning the stirrer switch to its spring-loaded battery check position. The warning LOBAT will indicate when approximately 5 hours of battery life remain.
- d. Zero the instrument. Set the function switch to ZERO and adjust the display to read 0.00 with the O₂ ZERO control.
- e. Switch to the 0.01 mg/l position and wait at least 60 minutes for the probe to polarize. Allowing additional time to re-polarize the probe is necessary whenever the meter has been turned off or the probe has been disconnected.
- f. After the 60 minute wait, turn the function switch to ZERO and readjust the O₂ ZERO control to 0.00 if necessary. The meter is now ready to calibrate.
- g. Calibration - Meters are calibrated using the Winkler azide method as described in this SOP in the Field Measurements Chapter III - section 3.2.
- h. The D.O. probes are placed in a container of tap water with a relatively stable temperature. A minimum of four Winkler tests are then performed on the tap water. Three of the four resulting Winkler values must be within a 0.1 mg/l range. If the values are not in the 0.1 mg/l range, the Winkler tests should be repeated until the values are within the + or - 0.1 range. The three Winkler values are then averaged to provide an initial calibration value.
- i. After the probes have stabilized in the container of tap water, the function switch is set to 0.01 mg/l, the meters are then adjusted to the initial calibration value by turning the O₂ CALIB control. The meters are now calibrated.
- j. Leave the instrument on throughout the test to avoid re-polarizing the probe. Reactivate the stirrer approximately 2 minutes before each reading and turned off after the reading.

- k. Obtain a bottom salinity reading using a YSI Model 33 S-C-T Meter. If salinity is present, the SALINITY knob on the YSI Model 58 D.O. Meter is adjusted accordingly.
- l. Upon completion of the SOD test, perform a terminal calibration on all YSI 58 D.O. meters used. All terminal calibration data is recorded on the SOD terminal calibration form (Figure 23).

Note: If SOD tests are performed in coastal areas where tidal influence may cause salinity values to fluctuate during the test, salinity readings should be taken frequently and salinity adjustments made to the YSI 58 D.O. meters.

2.1.2 *TERMINAL CALIBRATION Method*

- a. The D.O. probes are placed in a container of tap water with a relatively stable temperature and allowed to stabilize. A minimum of 4 Winkler tests are then performed on the tap water. The resulting Winkler values must be within a 0.1 mg/l range. If three of the four values are not in the 0.1 mg/l range, the Winkler tests should be repeated until the values are within the range. The Winkler values are then averaged to provide a terminal calibration value.
- b. After the Winkler bottles have been filled with the tap water, turn on the stirrers, wait one minute and then record the D.O. and temperature readings.
- c. Each D.O. reading should be within a 0.1 mg/l range from the average Winkler calibration value.

3. QUALITY ASSURANCE

3.1. Procedure

- a. Complete the Pre-Sampling Calibration, Post-Sampling Calibration Check, and SOD Worksheets (Figure 23) on-site during each SOD test.
- b. Perform Winkler tests per this SOP . Chapter III- section 3.1 . azide modification.
- c. Perform a minimum of three Winkler titrations for Initial Calibration and Terminal Calibration.
- d. Winkler values must be within a 0.1 mg/l range. If any value is outside the 0.1 mg/l range, then additional Winkler tests are performed until the values are within the range.
- e. The terminal YSI 58 D.O. Meter reading should be within a 0.1 mg/l range from the average terminal Winkler calibration value.
- f. A minimum ambient bottom D.O. of 2.0 mg/l is required to perform an SOD test (Murphy and Hicks, 1986).

- g. Chamber velocities must be in a 0.08 to 0.12 ft/sec. range (Howard, 1988).
- h. Take detailed field notes during the SOD test including a site description.
- i. Conduct a pre-check to each SOD study to provide information on the study feasibility and station characteristics. During the -check sediment samples are generally collected to determine bottom characteristics.

Figure 23. Sediment Oxygen Demand Calibration Worksheet

SEDIMENT OXYGEN DEMAND CALIBRATION WORKSHEET

STUDY AREA	STATION
DATE	STAFF ON SITE

ALL METERS ZERO PRIOR TO CALIBRATION (YES NO)	CALIBRATION NOTES:
MEMBRANES VISUALLY CHECKED PRIOR TO CALIBRATION (YES NO)	
MEMBRANES LAST REPLACED	
BATTERIES LAST REPLACED	
CALIBRATOIN METHOD (SATURATED AIR WINKLER)	
CALIBRATION PERFORMED BY	
SALINITY	

INITIAL CALIBRATION

TIME OF INITIAL CALIBRATION												
WINKLER READINGS: (A) (B) (C) (AVERAGE)												
METER READINGS BEFORE CAL	AMB	BLANK O	BLANK OO	CLEAR	1	2	3	4	5	A	B	C
WINKLER DIFFERENCE ADJUSTED												

TERMINAL CALIBRATION

TIME OF INITIAL CALIBRATION												
WINKLER READINGS: (A) (B) (C) (AVERAGE)												
METER READINGS BEFORE CAL	AMB	BLANK O	BLANK OO	CLEAR	1	2	3	4	5	A	B	C
WINKLER DIFFERENCE												

4. CHAMBER DEPLOYMENT

After the D.O. meter calibration procedure is complete, the SOD chambers are prepared for the test. All chambers are prepared as follows prior to being placed into the water (Lawhorn, 1988).

4.1. Setting up Chambers

4.1.1. Chamber Preparation

- a. Place the lids on replicate chambers in the up position with spacers located between the lid and lid companion ring. Wing nuts should be tight enough to hold the spacers in place but not so tight as to hamper removal after the chamber has been set in place on the bottom.
- b. Insert the water sampling port stoppers on each chamber lid (size #1, two on each lid).
- c. Open the monitoring probe port on all chamber lids (no stoppers).
- d. Inspect the replicate chamber lid gaskets for damage or debris that could prevent a watertight seal.
- e. Inspect the seals on the blank chambers for damage.
- f. Clip the harness ropes to each chamber.
- g. Open the water intake ports located on the bottom of the blank chambers (no stoppers).
- h. Disconnect the return pump tubing from the chamber lid male connectors.

4.2. Boat operation only:

- a. Hang the chambers in sequential order along the gunwale with the chamber lids several inches below the surface of the water.
- b. Tie the chamber harness to a gunwale cleat.
- c. Situate the boat over the bottom where the chambers will be placed.
- d. Do not allow the chambers to disturb the bottom sediment.

4.3. Land operation:

Chambers are placed on the stream bank in the order that they will be deployed. This will prevent harness ropes, pump cables and probe cables from becoming tangled during the chamber deployment and the SOD test.

4.4. Chamber deployment:

- a. Blank chambers are deployed first because sufficient time is required to replace surface water trapped inside the chamber with ambient bottom water prior to initiating the SOD test.

- b. When deploying blank chambers in soft sediment, place the chambers in an area away from the area that the replicate chambers will be deployed in order to avoid stirred up sediments from being drawn into the chamber through the open probe port.
- c. One clear polycarbonate and acrylic blank chamber is used in addition to the conventional aluminum blank chambers to provide an indication of whether or not photosynthesis is occurring in the water column. The mechanical functions and the deployment procedure for the clear blank chamber are identical to that of the aluminum blank chambers. In cases of high flow a weighted band should be placed around the clear chamber to prevent it from being washed away.
- d. Each blank chamber must be filled with surface water that enters through the two filling ports located on the bottom plate of the chamber.
- e. After the blank chamber is filled at the surface and prior to lowering the chamber, two #11½ stoppers must be inserted into the filling ports. Surface water is used to fill the blank chamber to create negative buoyancy so the chamber can be lowered to the bottom.
- f. After the filling port stoppers are in place, the chamber is agitated to dislodge any air that is trapped under the lid. The trapped air will exit through the probe port.
- g. The chamber is then lowered to the bottom.
- h. When the blank chamber is on the bottom, the pump is turned on. Unlike the replicate chambers, the lid and bottom of the blank chambers are permanently sealed thus no water exchange occurs when the chamber is lowered to the bottom. Surface water must be purged from the chambers by operating the pump with the tubing disconnected from the male adapters on the lid while the chamber is on the bottom. Bottom water is drawn into the chamber through the open probe port while the surface water is purged through the disconnected return tubing. With the two return pump tubes disconnected, the chamber will purge surface water and draw in bottom water.
- i. A light tapping on the pump housing and tubing will dislodge air bubbles trapped in the pump system.
- j. The pump is then allowed to run while the other chambers are being deployed.
- k. This procedure is repeated for each blank chamber.

4.5. *Replicate Chamber Deployment*

- a. After the blank chambers are deployed, each replicate chamber is slowly lowered to the bottom substrate prior to setting the chamber.

- b. Set the replicate chambers out in downstream to upstream order to prevent sediment disturbance and any silt that may have been disturbed from settling on areas where other chambers will be placed.
 - c. If the chamber location is unsatisfactory because of debris, or other bottom characteristics that would prevent the chamber from sealing then the chamber is carefully relocated. In addition, if the chamber location is atypical of the general stream area, the chamber or possibly the station should be relocated.
 - d. After the replicate chamber is placed in a satisfactory location on the bottom, carefully examine the sediment/flange seal and the sediment/inner core seal to assure that ambient water infiltration will not occur during the test.
 - e. The replicate chamber lid is then lowered by loosening the four wing nuts and removing the PVC spacers. The lid must be lowered very slowly as not to create a pressure wave and stir up silt inside the chamber. If silting occurs in the chamber, initial D.O. readings will be erratic and a longer period will be required for SOD rate stabilization (see: Section 5. Procedure for Recording SOD Data).
 - f. Replace the spacers between the companion ring and stainless steel washers. The wing nuts are then tightened and the gasket forms a watertight seal.
 - g. Activate the pump and lightly tap the pump housing and tubing to dislodge air trapped in the pump system.
 - h. Turn off the pump and allow any silt that may have been suspended to resettle before starting the test.
 - i. Reconnect the return tubing.
 - j. Repeat this process until all replicate chambers are deployed.
- 4.6. Once all replicate chambers are in place, insert DO probes into the probe ports beginning with the first blank chamber deployed and ending with the final replicate chamber.
- 4.7. During the D.O. probe installation, replicate chamber pumps can be turned on and a final check of the chamber and pump tubing can be performed.
- 4.8. In addition to the D.O. probes located inside the SOD chambers, one D.O. probe is placed on the outside of a chamber to record ambient D.O. values.
- 4.9. When an SOD test has been completed, chambers can usually be lifted from the bottom using the harness ropes.

5. RECORDING SOD FIELD DATA.

5.1. After SOD Chambers and Probes are installed

5.1.1 Readings

- a. Stirrers are activated approximately 2 minutes prior to reading meters and turned off after the data is recorded.
- b. All meters (including the ambient meter) are read at 15 minute intervals. For each chamber, D.O., temperature, and the change in D.O. per 15 minute time period is recorded on the SOD field sheet form (Figure 24).
- c. D.O. readings from the replicate chambers will usually decrease at a relatively similar rate. Typically, if relatively uniform decreases in D.O. are observed in the replicate chambers after stabilization, a sufficient SOD rate can be calculated from approximately 2 hours of testing (Murphy and Hicks, 1986).
- d. A minimum oxygen reduction of 0.4 mg/l is required before an SOD test should be terminated. This situation is not typically encountered and would provide an extremely low SOD rate indicating little organic content in the sediment.
- e. SOD tests with very slow oxygen uptake rates may be less reliable due to an extremely small amount of oxygen depletion over a greater period of time. Since longer tests are necessary when slow oxygen uptake is occurring, the potential for meter calibration drift increases.
- f. See Figure 25 for an example of completed SOD worksheet.

5.2 Recording Errors

5.2.1 *Erratic D.O. Readings Troubleshooting*

If observed in replicate chambers the following are possible problems:

- a. Initial D.O. readings may be erratic if sediment was disturbed during chamber placement on the bottom. This problem occurs often at stations where sediment consists of soft mud or a silt-like composition and is usually observed in all of the replicate chambers. For this reason, several of the initial D.O. readings may be omitted from the SOD rate calculations. The readings will usually stabilize as the suspended particles in the chamber settle out, (generally about 15 to 30 minutes, 1 to 2 readings).
- b. If D.O. readings from all chambers do not stabilize after 30 minutes, it may indicate that the chambers are sinking into the soft sediment causing the circulation diffusers to become close to the sediment and continually disturbing the silt. If this occurs, the chambers must be reset on the bottom and a chamber collar must be placed around the bottom chamber

flange to prevent the chambers from sinking. Chamber collars are flat, thin pieces of material, that increases the surface area of the chamber flange and prevent the chamber from sinking into soft sediment.

- c. If D.O. readings from a replicate chamber do not stabilize and begin to decrease after the other chambers have stabilized, it may indicate that the chamber was not initially sealed and ambient bottom water is leaking into the chamber via the ports, gasket seal, pump tubes or the sediment flange seal. The chamber must be reset and the seal integrity reconfirmed.
- d. On occasion, ambient water will begin leaking into a chamber. Chamber leaks (blowouts) are the most frequent problem encountered in SOD tests. This problem is easily recognized when D.O. values in a chamber that have been steadily decreasing suddenly begin to rise rapidly. However, if the chamber leak is small, the rate of decrease in D.O. may only be slowed, resulting in an unrealistically low rate for the chamber. For these reasons, the rate of D.O. change in each chamber must be carefully evaluated and recorded for each 15 minute time period during the SOD test.

If a chamber leak is detected the following options may be considered:

- Stop the leak and restart the test for that chamber; or
 - Delete the data from that chamber from the SOD test; or
 - Terminate entire test, if sufficient data has been recorded to establish a reliable linear regression.
- e. If the D.O. in a chamber falls much more rapidly than in the other chambers, it may indicate that the chamber has been inadvertently placed on organic debris such as decaying leaves or other organically rich deposits that may be uncharacteristic of the area. The chambers must be placed on sediment that is typical for the station area. If this problem is encountered, the chamber should be relocated or the data deleted from the SOD test.

The validity of SOD test data is dependent on locating the test site at an area that is typical of the water body being studied. If the chamber location is atypical of the general stream area then the chamber or possibly the station should be relocated.

- f. When other obvious D.O. or temperature problems occur during the SOD test, it is usually the result of meter or probe malfunction and can be detected by the terminal calibration results.

6. METER AND PROBE PREPARATION

6.1. Procedure

- a. Check all D.O. meters, cables and probes to assure proper functioning **before** the survey.
- b. Evaluate the YSI 58 instrument batteries and replaced if necessary. Stirrer batteries should be checked to assure that batteries are adequate to complete SOD test.
- c. Replace all D.O. probe membranes prior to each SOD survey. After the membrane has been changed, a minimum of 24 hours should be allowed for the probe to equilibrate before it is used for an SOD test. YSI Standard Membranes should be used.

7. SOD CHAMBER VELOCITY TEST

SOD rates are directly related to the sediment/water interface velocity, therefore specific and consistent velocities must be maintained in all chambers for accurate SOD testing. EPA recommends a constant chamber velocity of 0.1 ft/sec and an acceptable range of 0.08 to 0.12 ft/sec (Howard 1988). To maintain this velocity range, DWR uses a flow restrictor placed in the chamber pump tubing to reduce pumping velocity. The restrictor is 1" long, made from brass stock, and has a 7/64" opening in the center to allow a desired velocity of water.

All SOD chambers are periodically tested in the lab to ensure that velocities remain constant after repeated field use and pump wear. Velocity tests are performed using a Marsh McBirney Magnetic Flow Meter Model 201. The Marsh McBirney meter is factory calibrated. Chamber velocity tests procedures are as follows:

7.1. Velocity test procedures for replicate chambers:

- a. Insert a # 11½ stopper in the monitoring probe port and two # 1 stoppers in water sampling ports. All pump tubing should be connected and the chamber lid must be tight against the chamber companion ring.
- b. Place the chamber upside-down on a support in a manner that will allow access to the monitoring probe port. The support should not touch the pump tubing or alter the pump flow in any manner. (The chamber and support should be located over a sink or other acceptable area where the test water can be easily drained).
- c. Fill the chamber with water to the cutting ring flange (normal water/sediment interface).
- d. Turn the pump on. It may be necessary to add more water to fill pump and pump tubing after the pump is turned on and to tap the pump and tubing to dislodge trapped air. Place the Marsh McBirney probe 2 inches below the surface of the water halfway between the outer and inner chamber wall. Allow the

water circulation in the chamber to reach the maximum velocity (approximately 15 minutes).

- e. Read the Marsh McBirney Meter. The velocity in the chamber should be within a range of 0.08 to 0.12 ft/sec.
- f. If the velocity is not constant or out of the acceptable range, check the following:
 - Probe orientation or placement in the chamber.
 - Restrictions in pump tubing (7/64" brass restrictor may be blocked).
 - Air bubbles could be locking the pump or altering flow.
 - Pump may be damaged and not pumping maximum flow.
 - Check pump battery voltage output (12 volt)
 - Check velocity meter calibration.

7.2. Velocity Tests for Blank Chambers:

- a. Insert two # 11½ stoppers into the filling ports on the bottom of the blank chamber. All pump tubing should be connected.
- b. Place chamber right side up on a support in a manner that will allow access to the filling ports.
- c. Fill the chamber completely with water.
- d. Turn the pump on . It will be necessary to add more water to fill pump and pump tubing after the pump is turned on and to tap the pump and tubing to dislodge trapped air. Place the Marsh McBirney probe through the D.O. probe monitoring port at a depth of 2 inches. Allow the water circulation in the chamber to reach the maximum velocity (approximately 15 minutes).
- e. Read the Marsh McBirney Meter.
- f. Use the same trouble shooting procedures as with the replicate chambers if problems are encountered.

8. LEAK TEST FOR SOD CHAMBERS

SOD chambers must remain watertight during the SOD test to prevent ambient bottom water from entering the chamber and invalidating the test. The exchange of ambient bottom water and chamber water can occur by two means, by leaking between the sediment and chamber cutting edge or by leaking through any of the normally sealed chamber gaskets, stoppers, fittings and tube connections. Chamber leaks at the sediment/chamber interface generally occur as a result of sediment or sand washing out from around the chamber due to scouring and are usually detected during the test. Leaks through chamber seals, other than the sediment/chamber interface can be detected during the Chamber Velocity Test (Section 7). Note: leak test is under worst case conditions because chamber water (inside/outside) is equalized during the test. Procedures for leak testing SOD chambers are as follows:

1. Insert a # 11½ stopper in the monitoring probe port and # 1 stoppers in water sampling ports. All pump tubing should be connected and the chamber lid must be tight against the chamber companion ring.
2. Place the chamber upside-down on a support in a manner that will allow access to the monitoring probe port. The support should not touch the pump tubing or alter the pump flow in any manner. The chamber and support should be located over a sink or other acceptable area where the test water can be easily drained.
3. Fill the chamber with water to the cutting ring flange (normal water/sediment interface for replicate chambers and to bottom plate on blank chambers).
4. Turn the pump on.
5. If water leaks out, repair or replace the seal and repeat the leak test.

9. THREE POINT ANCHOR TECHNIQUE

If a boat operation is necessary to perform a SOD test, care must be taken to provide maximum stability and minimize wave action and horizontal swing over the bottom. Movement of the boat by wave action or swing on a single anchor line will result in chambers being lifted and the SOD test terminated. This problem can be avoided by using the following three-point anchor technique:

1. After the boat is on station, align the bow into the current.
2. Set bow anchor on SOD boat allowing a minimum scope of 3 times depth. More scope may be necessary if strong current or winds are present.
3. Use support boat to set aft port and aft starboard anchors (minimum scope 3 times depth).
4. Anchors should be oriented in a 3-point (tripod like) pattern with the SOD boat in the center.
5. After all anchors are set, the lines should be tightened as much as possible and cleated to provide maximum stability and minimize horizontal movement of the SOD boat.
6. While anchoring, care should be taken not to disturb the sediment where the SOD test is to be performed.
7. It is potentially dangerous to anchor with the stern of the boat facing upstream if current, waves or bad weather exists. The 3-point anchor method should not be used in areas affected by strong tidal current unless the test can be completed prior to the turning of the tide.

Figure 24. SOD Field Sheet

STUDY AREA																						
STATION LOCATION																						
DATE		SEDIMENT TYPE				DEPTH		CHAMBERS DIVER DEPLOYED? YES/NO				VELOCITY FT/SEC (at bottom)										
PERSON(S) READING METERS								STAFF ON SITE				BOAT SOD/BANK SOD										
TIME	MIN.	AMB		BLANK O		BLANK OO		BLANK CLEAR		REP 1		REP 2		REP 3		REP 4		REP 5		BACK-UP		
		DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	DO	TEMP	
	START																					
	15																					
	30																					
	45																					
	60																					
	75																					
	90																					
	105																					
	120																					
	135																					
	150																					
	165																					
	180																					
	195																					
	210																					
	225																					
	240																					
	255																					
	270																					

TIME	MIN.	Change in DO																				
	START																					
	15																					
	30																					
	45																					
	60																					
	75																					
	90																					
	105																					
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	180																					
	195																					
	210																					
	225																					
	240																					
	255																					

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APPENDICES

Appendix 1: DWR’s Hydrolab Multiparameter Guidance Sheet

DISSOLVED OXYGEN	HYDROLAB with CLARK CELL D.O. SENSOR (DOES NOT APPLY TO LDO SENSORS)
CALIBRATION	<p><u>Clark Cell Dissolved Oxygen (D.O.) Calibration for Hydrolab Meters:</u> (% AIR CALIBRATION IN WATER-SATURATED AIR)</p> <ol style="list-style-type: none"> 1) Secure probe to work surface and inspect membrane for tears and debris. 2) Rinse calibration cup with deionized water and attach to probe. 3) Fill calibration cup with tap water until water is just level with the O-ring used to secure the D.O. membrane. Do not cover the membrane. 4) Remove any water droplets from D.O. membrane with the corner of a chem-wipe or a lint-free cloth. 5) Place inverted lid (concave upward) on top of calibration cup. The lid should not completely seal or cover the cup (lid should be slightly tilted inwards on top of the cup, leaving a small gap or opening). 6) Wait 5 to 10 minutes for readings to stabilize. 7) Once readings are stable, record the following values on the calibration sheet: “Temperature”, “Initial % Saturation”, and “Initial Meter Reading (mg/L)”. 8) Record “Barometric Pressure” and “Altitude” on the calibration sheet. These values are available on the <i>Dissolved Oxygen Table</i> for your corresponding regional office. All calibrations should be performed in a controlled environment (field calibrations are not recommended). 9) Use the <i>Dissolved Oxygen Table</i> for your location to find the “D.O. Table Value” (based on the temperature displayed on the meter), and record value on calibration sheet. 10) Follow menu prompts to calibrate for Dissolved Oxygen, Percent Saturation, which is displayed as “DO%:SAT”. * NOTE: Dissolved oxygen should always be calibrated using % saturation. <i>Calibrations based on “mg/l” require a water sample with a known D.O. concentration (Winkler titration must be performed).</i> 11) When prompted, enter the barometric pressure for your location in millimeters of Mercury (mmHg). The unit should display “CALIBRATION SUCCESSFUL!” 12) On the calibration sheet, record the displayed mg/L value as “Calibrated Meter Reading” and the % SAT value as the “Calibrated % Saturation” value. * NOTE: “D.O. Table Value” and “Calibrated Meter Reading” value should be within ±0.5 mg/L of each other. <p><u>Terminal Calibration Check (Post-Sampling Meter Check)</u></p> <ol style="list-style-type: none"> a. Repeat calibration steps 1 thru 9. b. The post-sampling “D.O. Table Value” and the post-sampling “Initial Meter Reading” should be within ±0.5 mg/L of each other.
MAINTENANCE	<p><u>Sonde Storage - Calibration/Storage Cup:</u> Store sonde and sensors in the Calibration/Storage Cup filled with pH 4 buffer solution.</p> <p><u>Clark Cell D.O. Membrane Replacement:</u> D.O. membrane should be replaced when calibration is impossible, if calibration drift occurs quickly or frequently, or if membrane is dry or damaged.</p> <ol style="list-style-type: none"> 1) Remove O-ring and shake out old electrolyte. 2) Rinse sensor cavity with deionized water. 3) If gold cathode appears tarnished, dry and lightly buff with a pencil eraser or Kimwipe until gold is bright. 4) Refill with fresh D.O. electrolyte (2M KCl) until a meniscus forms. Remove any bubbles trapped in electrolyte. 5) Replace membrane and secure with O-ring. Inspect O-ring for any tears or breaks; replace as needed. 6) Trim excess membrane. 7) Allow membrane to soak overnight in tap water before calibrating. <p><u>D.O. Circulator Maintenance (Remove dirt and debris build-up from inside circulator impeller shaft):</u></p> <ol style="list-style-type: none"> 1) Use a flat-head screwdriver to remove impeller screw. 2) Clean dirt and debris from the screw, impeller, and inside the impeller shaft. 3) Replace screw and re-attach impeller to circulator with flat-head screwdriver. <p>Frequency of cleaning will depend on use and environment.</p>

DISSOLVED OXYGEN

HYDROLAB with LUMINESCENCE D.O. (LDO) SENSOR
(DOES NOT APPLY TO CLARK CELL SENSORS)

CALIBRATION

Hach Luminescence Dissolved Oxygen (LDO) Calibration for Hydrolab Meters:

(% AIR CALIBRATION IN AIR-SATURATED WATER)

- 1) Fill a 1-liter bottle half-full of tap water. Water bottle should remain open (no cap or seal) for at least 12 hours (overnight) to allow water to equilibrate to ambient temperature and atmospheric pressure.
- 2) After 12 hours have passed, use a thermometer to confirm that the water in the bottle is close to room temperature.
- 3) Seal/cap bottle and shake it very vigorously for 40 seconds to saturate the water with air.
- 4) With sonde positioned with sensors facing upward, pour the water into the calibration cup such that the LDO sensor cap and the temperature sensor are completely submersed (water should come close to the top of the calibration cup).
- 5) Completely cover the top of calibration cup with the inverted lid (do not tightly seal the cup).
- 6) Wait 10 minutes for readings to stabilize. If temperature changes more than ± 0.5 °C during calibration, recalibration of the sensor is recommended.
- 7) Once readings are stable, record the following values on the calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)".
- 8) Record "Barometric Pressure" and "Altitude" on the calibration sheet.
These values are available on the *Dissolved Oxygen Table* for your corresponding regional office. All calibrations should be performed in a controlled environment (field calibrations are not recommended).
- 9) Use the *Dissolved Oxygen Table* for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter), and record value on calibration sheet.
- 10) Follow menu prompts to calibrate for Dissolved Oxygen, Percent Saturation, which is displayed as "LDO%:SAT".
* **NOTE: Dissolved oxygen should always be calibrated using % saturation.**
Calibrations based on "mg/l" require a water sample with a known D.O. concentration (Winkler titration must be performed).
- 11) When prompted, enter the barometric pressure for your location in millimeters of Mercury (mmHg). The unit should display "CALIBRATION SUCCESSFUL"
- 12) On the calibration sheet, record the displayed mg/L value as "Calibrated Meter Reading" and the % SAT value as the "Calibrated % Saturation" value.
* **NOTE: "D.O. Table" value and "Calibrated Meter Reading" value should be within ± 0.5 mg/L of each other.**

Terminal Calibration Check (Post-Sampling Meter Check)

- a. Repeat calibration steps 1 thru 10.
- b. The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ± 0.5 mg/L of each other.

MAINTENANCE

Sonde Storage - Calibration/Storage Cup:

Store sonde and sensors in the Calibration/Storage Cup filled with pH 4 buffer solution.

Hach LDO Sensor Cap Replacement:

Replace once a year or when cap is damaged.

- 1) Unscrew old sensor cap from end of probe.
- 2) Carefully dry clear plastic window at the end of probe with cotton swab.
- 3) Place cap seal and o-ring on the probe tip.
- 4) Screw new sensor cap onto probe so that the o-ring seal is compressed. Do not over-tighten cap.
No water or moisture should be present between sensor cap and clear plastic window at top of probe.
- 5) Do NOT use alcohol or any organic solvent solutions to clean the Hach LDO sensor. These solvents will damage the plastic sensor cap.

SPECIFIC CONDUCTANCE	HYDROLAB
CALIBRATION	<p><u>THREE-STEP SPECIFIC CONDUCTIVITY PROCEDURE:</u></p> <p>I. “Dry Air” (ALWAYS ZERO): The “Dry Air” step is a check for the Quanta meters only, and a calibration for the Hydrolab 4a and MS5 meters.</p> <ol style="list-style-type: none"> 1) Attach calibration cup to probe. Fill calibration cup half-full with deionized water and seal with lid. Shake probe to rinse. Repeat. 2) Secure probe to work surface, and remove calibration cup. 3) Dry the inside of conductivity sensor slot thoroughly. 4) Record displayed value as “Initial Meter Reading” in the “Dry Air” section of the calibration sheet. If the reading is not within ± 2, follow cleaning procedure, and repeat calibration procedure. <p>If using a Quanta, proceed to step 8.</p> <p>Steps 5-7 are for Hydrolab 4a and MS5 meters only:</p> <ol style="list-style-type: none"> 5) Follow menu prompts to calibrate for specific conductance. 6) When prompted, enter “0” (zero) as specific conductance standard. Display should read “CALIBRATION SUCCESSFUL!” 7) Record displayed value as “Calibrated Meter Reading” in the “Dry Air” section of the calibration sheet. <p>II. Conductivity Standard: Calibrations should be performed using fresh, certified conductivity standards that bracket the range of measurements to be taken that day. Record the standard’s “true value” (found on the certificate of analysis or bottle label) and lot number (also called analysis number) on the calibration sheet. Traceable® Certificate of Analysis for Conductivity Solution sheets (one certificate for each lot number) should be retained and stored in a notebook.</p> <ol style="list-style-type: none"> 8) Re-attach calibration cup to probe. Fill cup half-full with deionized water. Seal cup with lid and shake probe to rinse. Repeat. 9) Rinse sensors with small amount of fresh conductivity standard. Discard rinse. 10) Fill calibration cup with conductivity standard to within a centimeter of the top of the Calibration Cup. (Pour standard down the interior side of the cup to avoid trapping bubbles.) 11) Wait approximately 1 to 3 minutes for readings to stabilize. 12) Record displayed value as “Initial Meter Reading” in the “Conductivity Standard” section of the calibration sheet. 13) Follow menu prompts to calibrate for Specific Conductance. When prompted, enter the value of the standard. Unit will display “CALIBRATION SUCCESSFUL!” 14) Record displayed value as “Calibrated Meter Reading” in the “Conductivity Standard” section on the calibration sheet. <p>III. Calibration Check:</p> <ol style="list-style-type: none"> 15) Rinse with deionized water and wipe dry with a chem-wipe or a lint-free cloth. Confirm that the meter display is reading 0 (zero) μS before going to the next step. 16) Repeat steps 9-11 with a fresh conductivity standard with a value different from the one used in the previous steps. Choose a standard that will give the best range of values for the anticipated conductivity of the samples to be collected. 17) Record displayed value as “Initial Meter Reading” in the “Calibration Check” section of the calibration sheet. <p><u>Terminal Calibration Check (Post-Sampling Meter Check)</u></p> <ol style="list-style-type: none"> a. Repeat calibration steps 1 thru 4, and record value in the “Dry Air” section on the calibration sheet. For the “Dry Air” check, displayed value should be between -2 and $2 \mu\text{S}$. b. Repeat calibration steps 8 thru 12, and record value in the “Conductivity Standard” section on the calibration sheet. “Conductivity Standard” value should be within $\pm 10\%$ of the standard. c. Repeat step 15 thru 17, and record value in the “Calibration Check” section on the calibration sheet. “Calibration Check”, value should be within $\pm 10\%$ of the standard.
MAINTENANCE	<p><u>Cleaning Conductivity Sensor:</u> Conductivity cell should be cleaned frequently (in addition to rinsing with DI water after field use). A clean cell is imperative for accurate readings.</p> <ol style="list-style-type: none"> 1) Use a cotton swab and mild soap to remove any films or deposits on the sensor. 2) Rinse sensor with deionized water.

pH

HYDROLAB

CALIBRATION

TWO-POINT PH CALIBRATION REQUIRED:**1st Calibration Point (always start with 7 buffer):**

- 1) Attach calibration cup to probe. Fill cup half-full with deionized water. Seal cup with lid and shake probe to rinse. Repeat.
- 2) Rinse sensors with small amount of 7 pH buffer. Discard buffer rinse.
- 3) Secure probe to work surface.
- 4) Fill calibration cup with **fresh** 7.0 buffer to within a centimeter of the top of the calibration cup. Wait at least 2 minutes for stabilization.
- 5) Record displayed value as the "Initial Meter Reading" under Buffer # 1 on the calibration sheet.
- 6) Follow menu prompts to calibrate for pH. When prompted, enter "7.0" as the value of your standard. Unit will display "CALIBRATION SUCCESSFUL!"
- 7) Record the displayed value as "Calibrated Meter Reading" for Buffer #1 on the calibration sheet.

2nd Calibration Point:

- 8) Rinse sensors with deionized water.
- 9) Rinse sensors with small amount of a pH buffer that is similar to the anticipated pH of the samples to be collected. Discard buffer rinse.
- 10) Fill calibration cup with **fresh** buffer to within a centimeter of the top of the calibration cup. Wait 1 to 3 minutes for solution to stabilize.
- 11) Record displayed value as the "Initial Meter Reading" for Buffer # 2 on the calibration sheet.
- 12) Follow menu prompts to calibrate for pH. When prompted, enter the value of Buffer #2 (also called the slope buffer value).
Unit will display "CALIBRATION SUCCESSFUL!"
- 13) Record the displayed value as "Calibrated Meter Reading" for Buffer #2 on the calibration sheet.

Confirmation Buffer:

- 14) Rinse sensors and calibration cup with deionized water.
- 15) Rinse sensors with small amount of 7 buffer. Discard buffer rinse.
- 16) Fill calibration cup with 7 buffer to within a centimeter of the top of the calibration cup. Wait 1 to 3 minutes for solution to stabilize.
- 17) Record the displayed value as the "Meter Reading" under "Confirmation Buffer 7.0" on the calibration sheet
- 18) Confirm that the "Meter Reading" value is within ± 0.1 of the buffer value (between 6.9 and 7.1).

Terminal Check (Post-Sampling Meter Check)

- a. Repeat steps 1 thru 5 (for 7 buffer); record displayed value on the calibration sheet.
This value should be within ± 0.2 of 7 (for 7 buffer).
- b. Repeat steps 8 thru 11 for Buffer #2. Record value on calibration sheet.
Value should be within ± 0.2 of Buffer #2.

The "Confirmation Buffer" step is not required for post-sampling meter checks.

pH	HYDROLAB
MAINTENANCE	<p><u>Indicators that maintenance is needed include:</u></p> <ul style="list-style-type: none"> • Unable to calibrate • Slow response • Erratic readings • Clogged reference junction • Black reference junction • Coated glass bulb <p>Maintain as directed below. The pH electrolyte should be changed at least 3 to 4 times a year, or as needed.</p> <p><u>pH Reference Electrode Maintenance: (pg 45 - Hydrolab MS 5 User Manual, Feb 2006 ed. 3)</u></p> <p>Check the reference electrode regularly to confirm flow through the Teflon junction. To test for flow through the junction, press lightly on the top of the reference electrode. A bead of electrolyte should wet the Teflon junction. Maintain as directed below.</p> <ol style="list-style-type: none"> 1) Remove the pH reference sleeve, and discard old electrolyte. 2) Drop two KCL salt pellets into reference sleeve. Refill the sleeve (to the top) with electrolyte, which is provided in the maintenance kit (3M KCl, saturated with AgCl). 3) With the sensors pointed down, gently push the reference sleeve back onto its mount, until the sleeve just covers the O-ring located on the mount. 4) Turn probe so that the sensors point up and push the sleeve the rest of the way onto its mount. Air and electrolyte should flow through the Teflon junction. If it does not, repeat steps 1-4. If the second attempt fails, replace the old junction. 5) Rinse with tap water. <p><u>Cleaning pH Glass Electrode:</u></p> <p>Check the glass bulb regularly for a dirty film or scratches. Clean as directed below.</p> <ol style="list-style-type: none"> 1) Wet a cotton swab with a mild soap solution. 2) Gently swab the pH glass electrode. 3) Rinse electrode with tap water.
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Appendix 2: DWR's YSI 85 and Accumet Guidance Sheet

DISSOLVED OXYGEN	YSI 85
CALIBRATION	<p>D.O. Calibration for YSI-85 Meters: (% AIR CALIBRATION IN WATER-SATURATED AIR)</p> <ol style="list-style-type: none"> 1) Inspect membrane. Membrane should be taut, flat, and free of tears and debris. 2) Confirm that sponge in the calibration/storage chamber is moist (not soaking wet). Dry the probe and sides of calibration chamber with a lens cloth. 3) Insert probe into the calibration/storage chamber. Make sure there are no water droplets on the membrane. 4) Turn meter "ON" and press "MODE" until dissolved oxygen is displayed in "%". 5) Wait approximately 15 to 30 minutes for readings to stabilize. 6) Once readings are stable, record the following displayed values on the calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)" (press mode to switch from % Saturation to mg/L). Then press mode until % Saturation is again displayed on the screen. 7) Record "Altitude" and "Barometric Pressure" on the calibration sheet. These values are available on the <i>Dissolved Oxygen Table</i> for your corresponding regional office. All calibrations should be performed in a controlled environment (field calibrations are not recommended). 8) Use the <i>Dissolved Oxygen Table</i> for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter); record value on calibration sheet. 9) Press and release the "Up" and "Down" arrow buttons at the same time. 10) Use the arrow buttons to find the local altitude (to the nearest 100 ft) and press "ENTER". 11) "CAL" will be visible in the lower left of the display. The current % reading should be visible on the main display. Press "ENTER". 12) "SAVE" will be displayed, and the unit will automatically return to the Normal Operation Mode. % Saturation will be displayed in the main screen. Record value as "Calibrated % Saturation". 13) Press mode until mg/L is displayed. Record the displayed value as the "Calibrated Meter Reading (mg/L)" on the calibration sheet. <p>NOTE: "D.O. Table Value" and "Calibrated Meter Reading" value should be within ±0.5 mg/L of each other.</p> <p>Once calibrated, the YSI-85 should remain "On" until terminal calibration checks are completed at the end of the sampling day; otherwise, meter calibrations may be compromised.</p> <p>Terminal D.O. Calibration Check (Post-Sampling Meter Check)</p> <ol style="list-style-type: none"> a. Repeat calibration steps 1 thru 8. b. The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ±0.5 mg/L.
	MAINTENANCE
<p>YSI 85 - Accumet 8-15-07.doc Page 1 of 4 YSI-8/15/2007</p>	

SPECIFIC CONDUCTANCE	YSI 85
CALIBRATION CHECK	<p>“Dry Air” Check (Zero):</p> <ol style="list-style-type: none"> 1) Turn meter “ON”. 2) Press “MODE” to advance to Specific Conductance. “°C” should be flashing on the display. 3) The displayed value should be within $\pm 2 \mu\text{S}$ of zero. Record displayed value as “Initial Meter Reading” in the “Dry Air” section of the calibration sheet. If the displayed value is not within the range of -2 to 2 μS, clean, rinse and thoroughly dry the conductivity cell. <p>Note (YSI 85 Meters): You are only <u>checking</u> the meter’s calibration (as opposed to actually calibrating the meter); therefore, no value should be recorded as the “Calibrated Meter Reading” on the calibration sheet.</p> <p>Check using Conductivity Standard:</p> <p>Conductivity calibrations should be checked using a standard that is similar to the anticipated measurements to be collected in the field that day. Record the standard’s “true value” (found on the certificate of analysis or bottle label) and lot number (also called analysis number) on the calibration sheet. Traceable® Certificate of Analysis for Conductivity Solution sheets (1 certificate for each lot number) should be retained and stored in a notebook.</p> <ol style="list-style-type: none"> 4) Rinse probe with distilled or deionized water and wipe dry with a chem-wipe or a lint-free cloth. 5) Rinse probe with a small amount of conductivity standard (make sure some of the standard rinse goes into the oval-shaped hole on the side of the probe). 6) Insert probe into a vessel containing the standard such that the conductivity cell is completely submerged. Do not rest the probe on the bottom of the container (probe should be approximately ¼ inch from the bottom). Move the probe from side to side to dislodge any bubbles; wait for readings to stabilize. 7) Record the displayed value as the “Initial Meter Reading” in the “Conductivity Standard” section of the calibration sheet. This value must be $\pm 10\%$ of the standard value. 8) If the displayed value is not within $\pm 10\%$ of the standard value, clean probe and repeat calibration check with a FRESH standard. <p>Calibration Confirmation:</p> <ol style="list-style-type: none"> 9) Rinse probe with distilled or deionized water and wipe dry with a chem-wipe or a lint-free cloth. Confirm that the meter display is reading 0 (zero) μS before moving to the next step. 10) Repeat steps 5 thru 8 with a second standard that has a different specific conductance value. Record value in the “Calibration Check” section of the calibration sheet. <p><u>Terminal Conductivity Calibration Check (Post-Sampling Meter Check)</u></p> <ol style="list-style-type: none"> a. Repeat steps 1 thru 10, and record values on the calibration sheet. b. For the “Dry Air” check, displayed value should be between -2 and 2 μS. c. For “Conductivity Standard” and “Calibration Check”, values should be within $\pm 10\%$ of the standard.
MAINTENANCE	<p>Cleaning Conductivity Cell:</p> <ol style="list-style-type: none"> 1) Dip the conductivity cell (oval-shaped hole on the side of the probe) in alcohol or a mild detergent and agitate for 2 to 3 minutes. Remove from cleaning solution. 2) Use a soft nylon brush to remove any contaminants from the inside of the electrode chamber. 3) Repeat steps 1 and 2 until the cell is clean. 4) Rinse cell with deionized water. 5) Dry cell thoroughly, and verify that the unit reads between -2 to 2 μS in dry air. <p>If the above cleaning procedure does not restore the meter, repeat steps 1-5 using 1:1 isopropyl alcohol and 1 N HCl. This more extensive cleaning is rarely required.</p> <p>Conductivity Cell Check:</p> <p>If having difficulty calibrating or readings are erratic, check the conductivity cell constant:</p> <ol style="list-style-type: none"> 1) Turn meter “ON”. The unit will go through a self-test procedure. 2) A value will be displayed, along with “CEL”. The displayed value should be between 4.8 and 5.2. <p>If the displayed value is not within the specified range, clean the cell, and recalibrate the meter (see Meter Manual for calibration instructions; re-calibration is RARELY required).</p>

pH	ACCUMET AP61
CALIBRATION	<p>Two-point pH Calibration Required:</p>
	<p><u>Clear Previous Slope Efficiency</u></p>
	<ol style="list-style-type: none"> 1) Turn meter on. 2) Press "SETUP" to view the electrode efficiency (as percent slope) stored in the meter. In most cases, you do NOT want to accept this existing efficiency and should clear it. 3) Press "SETUP" again to access the clear buffers option. 4) "Clr" will be displayed on the unit. Press "ENTER" to clear the existing buffers and return to the Measure screen.
	<p><u>1st Calibration Point (start with 7 Buffer):</u></p>
	<ol style="list-style-type: none"> 5) Open fill hole on probe. 6) Rinse sensors with distilled or deionized water and blot dry. 7) Rinse sensors with small amount of 7 buffer. Discard buffer rinse. 8) If the meter is not in the pH Mode, press "MODE" until the display indicates the pH mode. 9) Immerse the end of the probe into 7 buffer. Wait for reading to stabilize. 10) Record buffer temperature. Record displayed value as the "Initial Meter Reading" for Buffer # 1 on the calibration sheet. 11) Press "std" to access the Standardize Screen. The buffer group used by the meter will be displayed briefly, and the prompt "PRESS std TO STANDARDIZE" will flash. 12) Press "std" again to initiate standardization. The meter will automatically recognize the buffer and display the value on the screen. 13) Record the displayed value as "Calibrated Meter Reading" for Buffer #1 on the calibration sheet.
	<p><u>2nd Calibration Point (4 or 10 Buffer):</u></p>
	<ol style="list-style-type: none"> 14) Repeat steps 5 thru 12 using a pH buffer similar to the anticipated pH of the samples to be measured. 15) Record values as instructed above for Buffer #2 on the calibration sheet.
	<p><u>Slope Efficiency Check:</u></p>
	<ol style="list-style-type: none"> 16) When the meter accepts the second buffer, the unit will briefly display the efficiency (as the percent slope) of the electrode's performance. 17) Record displayed value as "Slope Efficiency" on the calibration sheet.
	<p>The "Slope Efficiency" should be $\geq 95\%$.</p> <p>If the menu changes before the displayed value can be recorded, the slope efficiency can be accessed by pressing "SETUP".</p>
<p><u>Confirmation Buffer (7.0)</u></p>	
<ol style="list-style-type: none"> 18) Rinse sensors with distilled or deionized water and blot dry. 19) Rinse sensors with small amount of 7 buffer. Discard buffer rinse. 20) Immerse the probe into 7 buffer again to confirm the calibration. 21) Record the displayed value as the "Meter Reading" under "Confirmation Buffer" on the calibration sheet. Confirm that the "Meter Reading" value is within ± 0.1 of the buffer value (between 6.9 and 7.1). 	
<p><u>Terminal pH Check (Post-Sampling Meter Check)</u></p>	
<ol style="list-style-type: none"> a. Repeat calibration steps 5 thru 10 (for 7 buffer) and record displayed value on the calibration sheet. This value should be within ± 0.2 of 7 (for 7 buffer). b. Repeat procedure for the other buffer (Buffer #2) that was used to calibrate meter. Record value on calibration sheet. Value should be within ± 0.2 of Buffer #2. 	

pH	ACCUMET AP61
MAINTENANCE	Electrolyte Level Check the electrolyte level frequently. The electrolyte level should be within ¼ inch of the cap. Fill as needed.
	Refilling Electrolyte: <ol style="list-style-type: none">1) Open the fill hole on the cap ring.2) Hold probe such that sensors are facing downwards.3) Insert the tip of the electrolyte-dispensing bottle into the fill hole and press firmly to make an airtight seal. Squeeze the dispensing bottle for approximately 30 seconds or until adequately filled.4) Remove dispensing bottle from fill hole.
	Cleaning pH Glass Electrode: <ol style="list-style-type: none">1) Wet a cotton swab with alcohol.2) Gently swab the pH glass electrode.3) Rinse electrode with deionized water
	Cleaning the pH reference electrode: If crystal residue forms on electrode junction or inside the electrolyte reservoir: <ol style="list-style-type: none">1) Empty filling solution from reservoir by shaking it out through the fill holes.2) Rinse electrolyte reservoir repeatedly with distilled or deionized water until all crystals are dissolved. Warm tap water can be used as a preliminary step to quickly dissolve crystals.3) Refill reservoir with the electrolyte (4M KCl saturated with AgCl).
	Installing and Filling pH Reference Electrode: <ol style="list-style-type: none">1) Carefully remove new probe from packaging. Be careful when handling the probe; even a small scratch on the glass bulb can cause irreparable damage.2) Rinse electrode and sensors with distilled or deionized water to remove crystal residue that may have formed on the surface during storage.3) Open the fill hole on the cap ring.4) Check the electrolyte level. If level is low, add electrolyte (4M KCl saturated with AgCl) as described above. Electrolyte solution is included with each new electrode.5) Connect new probe to the display unit. Soak new probe in pH 4 buffer for 10 minutes prior to standardization.
	Probe Storage: When not in use, store the probe in pH 4 buffer and confirm that the fill hole is closed. Never store probe in distilled or deionized water!

Appendix 3: DWR's YSI 6920 Multiparameter Guidance Sheet

DISSOLVED OXYGEN	YSI 6920 with ROX (OPTICAL D.O.) SENSOR
CALIBRATION	<p><u>D.O. Calibration for YSI Meters with ROX (Optical D.O.) Sensor:</u> (% AIR CALIBRATION IN WATER-SATURATED AIR)</p> <ol style="list-style-type: none"> 1) Remove calibration storage cup from sonde, and confirm that optical D.O. probe has been stored in moist environment. Place calibration cup on work surface with the uncapped end facing upward. 2) Use lens tissue to carefully dry all sensors. The temperature and optical D.O. sensors must be completely dry. 3) Pour a small amount of tap water into the calibration cup (just enough to completely cover the bottom of the calibration chamber and create a 100% humid environment). Temperature and optical D.O. sensors CANNOT be in contact with water during calibration. 4) Place probes (pointing downward) into calibration cup carefully so that no water droplets get on the temperature sensor or optical D.O. sensor. 5) Twist the calibration cup onto the sonde no more than one or two threads, so that the cup is vented to the atmosphere. 6) Wait approximately 15 minutes to guarantee thermal equilibration between the temperature and optical D.O. sensors. To observe readings during this time, place the sonde in Run Mode: 650 Main Menu ⇒ Sonde run 7) Access the D.O. Calibration Menu: 650 Main Menu ⇒ Sonde Menu ⇒ calibrate ⇒ optic-T Dissolved oxy ⇒ ODosat % ⇒ 1 point NOTE: Dissolved oxygen should always be calibrated using % saturation. <i>Calibrations based on "mg/l" require a water sample with a known D.O. concentration (requires Winkler titration).</i> 8) Enter Barometric Pressure in mmHg. Record "Barometric Pressure" and "Altitude" on calibration sheet. These values are available on the <i>Dissolved Oxygen Table</i> for your corresponding regional office. All calibrations should be performed in a controlled environment (field calibrations are not recommended). 9) Real-time values will be displayed for all active parameters. When readings are stable for 30 seconds, record the following values on the calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)". 10) Use the <i>Dissolved Oxygen Table</i> for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter), and record value on calibration sheet. 11) Press ↵ (Enter) to calibrate Dissolved Oxygen. "Calibrated" should be displayed at the top of the screen. 12) On the calibration sheet, record the displayed mg/L value as "Calibrated Meter Reading" and the % SAT value as the "Calibrated % Saturation" value. * NOTE: The "D.O. Table Value" and the "Calibrated Meter Reading" value should be within ±0.5 mg/L of each other. 13) Press ↵ (Enter) to return to the D.O. Calibration Menu. Press "Esc" (3 times) to return to the main menu.
	<p><u>Terminal Calibration Check (Post-Sampling Meter Check)</u></p> <ol style="list-style-type: none"> 14) Repeat calibration steps 1 thru 6. Record "Temperature", "% Saturation", "Initial Meter Reading" from the Run Mode. 15) Record the barometric pressure and altitude where the terminal calibration check is being performed. 16) Repeat calibration step 10. 17) The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ±0.5 mg/L of each other.

**DISSOLVED
OXYGEN****YSI 6920 with ROX (OPTICAL D. O.) SENSOR****MAINTENANCE****Probe Storage:**

The probe must be stored in a moist environment.

Store probe in Calibration/Storage Cup approximately half full of tap water. Do not use distilled water (this will negatively affect the pH probe).

During long-term storage, inspect at least once a month to ensure the probe is still in a moist environment.

Optical D.O. Membrane Re-hydration:

If left in ambient air for more than 2 hours, the optical D.O. membrane must be re-hydrated.

- 1.) Pour approximately 400 mL of water into a 600 mL glass beaker (plastic containers should NOT be used). Use a thermostatted hotplate or an oven to heat the water to a consistent temperature of 50° C, ± 5° C.
- 2.) Place the probe tip containing the optical D.O. membrane in warm water and leave it at the elevated temperature for approximately 24 hours. Cover vessel to minimize evaporation.
- 3.) After re-hydration, store the probe in either water or water-saturated air before calibration and deployment.

Optical D.O. Sensor Cleaning:

Clean only with a lens tissue that has been moistened with water.

NEVER use alcohol or other organic solvents; organic solvents will ruin the membrane.

Wiper Operation:

The wiper can be used as-needed to wipe the sensor face during sampling.

- 1.) Use the display menus to activate the wiper:
 - 650 main menu ⇒ sonde run ⇒ clean optics (upper right corner of screen) ⇒ Press ← (Enter) to clean optics.
- 2.) After the wiper has finished rotating, wait 30 seconds before recording a measurement.

Changing the Wiper:

- 1.) Loosen setscrew until the wiper can be removed from the shaft.
- 2.) Place new wiper on the wiper shaft.
- 3.) Gently press the wiper against the face of the probe until the foam pad is compressed to roughly one half of the original thickness and then tighten the setscrew.

It is recommended that a business card be slid in between the wiper arm body and the probe face when installing the wiper. After installation, a gap about the thickness of a business card should be between the wiper arm body and the face of the probe
- 4.) Rotate the wiper to confirm that it "parks" correctly (180° from the ROX membrane):
 - 650 main menu ⇒ sonde run ⇒ clean optics (upper right corner of screen) ⇒ Press ← (Enter) to clean optics.

NEVER rotate the wiper manually. This will void the warranty.

Optical D.O. Membrane (sensor cap) Replacement:

Optical D.O. membrane should be replaced once a year or if damaged.

Detailed instructions are sent with the new membrane kit (YSI 6155).

When installing a new membrane, new calibration codes (included with each new membrane) must be entered.

SPECIFIC CONDUCTANCE	YSI 6920
CALIBRATION	<p><u>THREE-STEP SPECIFIC CONDUCTIVITY PROCEDURE:</u></p> <p>I. "DRY AIR" (ALWAYS ZERO):</p> <p>The "Dry Air" step is a check for YSI meters.</p> <ol style="list-style-type: none"> 1) Attach calibration cup to probe. Fill calibration cup half-full with deionized water and seal with lid. Shake probe to rinse. Repeat. 2) Remove calibration cup. Place cup on work surface with the uncapped end facing upward. 3) Use a cotton swab to dry the inside of the conductivity cells. 4) Record displayed value as "Initial Meter Reading" in the "Dry Air" section of the calibration sheet. The probe should read close to zero (± 2). If the reading is not within ± 2, follow cleaning procedure, and repeat calibration procedure. <p>II. CONDUCTIVITY STANDARD:</p> <p>Calibrations should be performed using a fresh, certified conductivity standard that is similar to the conductivity of the samples to be collected that day. Record the standard's "true value" (found on the certificate of analysis or bottle label) and lot number (also called analysis number) on the calibration sheet. Traceable® Certificate of Analysis for Conductivity Solution sheets (one certificate for each lot number) should be retained and stored in a notebook.</p> <ol style="list-style-type: none"> 5) Re-attach calibration cup. Fill cup half-full with deionized water. Seal cup with lid and shake probe to rinse. Repeat. 6) Rinse sensors with small amount of fresh conductivity standard. Discard rinse. 7) Remove calibration cup from sonde. Place cup on work surface with the uncapped end facing upward. 8) Pour conductivity standard into the calibration cup. Make sure there is enough standard to cover the entire conductivity cell when the probe is placed in the cup. 9) Place sonde into the calibration cup. Agitate sonde to remove air bubbles trapped in the conductivity cells. Air bubbles will give erroneously low readings. 10) Enter Run mode to view readings: 650 Main Menu \Rightarrow Sonde run 11) Press Esc to go back to the 650 Main Menu. 12) Access the Calibrate menu for Specific Conductance: 650 Main Menu \Rightarrow Sonde menu \Rightarrow calibrate \Rightarrow Conductivity \Rightarrow SpCond 13) Enter the True Value of the conductivity standard in milliSiemens/cm. Press ↵ (Enter). 14) Wait for readings to stabilize. 15) Record displayed value as "Initial Meter Reading" in the "Conductivity Standard" section of the calibration sheet. 16) Press ↵ (Enter) to calibrate meter. The message in the top center of the screen will switch to "calibrated". Record displayed value as "Calibrated Meter Reading" on calibration sheet. Never accept an out-of-range calibration. 17) Press ↵ (Enter) to return to the Calibrate menu. <p>III. CALIBRATION CHECK:</p> <ol style="list-style-type: none"> 18) Rinse with deionized water and wipe dry with a lens tissue or a lint-free cloth. 19) Confirm that the meter display is reading 0 (zero) μS before going to the next step. 20) Repeat steps 5-10 with a fresh conductivity standard of a value different from the one used in the previous calibration steps. Choose a standard that will give the best range of values for the anticipated samples to be collected. 21) Record SpCond value as "Initial Meter Reading" in the Calibration Check section on the calibration sheet. The value must be within 10% of the standard. <p><u>Terminal Calibration Check (Post-Sampling Meter Check)</u></p> <ol style="list-style-type: none"> a. Repeat calibration steps 1 thru 4, and record value in the "Dry Air" section on the calibration sheet. For the "Dry Air" check, displayed value should be between -2 and $2 \mu\text{S}$. b. Repeat calibration steps 5 thru 10. Record value in the "Conductivity Standard" section on the calibration sheet. "Conductivity Standard" value should be within $\pm 10\%$ of the standard. c. Repeat step 18-21, and record value in the "Calibration Check" section on the calibration sheet. "Calibration Check" value should be within $\pm 10\%$ of the standard.
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SPECIFIC CONDUCTANCE	YSI 6920
MAINTENANCE	<p>* Never accept an out-of-range calibration!</p> <p><u>Checking the Conductivity Cell Constant:</u> When troubleshooting the conductivity probe, first check the cell constant.</p> <ol style="list-style-type: none"> 1) 650 Main Menu ⇒ Sonde menu ⇒ Advanced ⇒ Cal constants 2) The value displayed next to "Cond" should be 5.0, ± 0.45. Numbers outside of this range indicate a problem in the calibration process or that a contaminated standard was used to calibrate the meter. 3) If conductivity cell constant is not within the acceptance range (between 4.55 and 5.45), clean the cell, and reset the calibration cell constant (see instructions below). <p><u>Cleaning Conductivity Sensor:</u> Conductivity cell should be rinsed with deionized water after field use. Clean conductivity cell frequently. A clean cell is imperative for accurate readings.</p> <ol style="list-style-type: none"> 1) Dip small cleaning brush (provided with new meters) into distilled or deionized water and insert brush into each hole 15-20 times. For a more thorough cleaning, use a mild dishwashing detergent with the brush. 2) Rinse sensor thoroughly with deionized water. 3) Perform the Dry Air Check described in Calibration Steps 1-4 to ensure probe reads close to zero in air. <p><u>Reset Calibration Cell Constant:</u></p> <ol style="list-style-type: none"> 1) Reset the calibration cell constant by accessing the Calibrate menu: 650 Main Menu ⇒ Sonde menu ⇒ Calibrate ⇒ Conductivity ⇒ SpCond 2) When prompted to "Enter the spCond (mS/cm)", press and hold the Enter key (↵) and press the Esc key. 3) The menu will ask "Unca1?" Select Yes. Press the Enter key (↵) 4) Recalibrate the meter using fresh, certified conductivity standards.
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pH	YSI 6920
CALIBRATION	Two-point pH Calibration Required (Three-point pH Calibration is Optional):
	1ST CALIBRATION POINT (ALWAYS START WITH 7 BUFFER):
	1) Rinse probes and calibration cup with distilled water.
	2) Rinse probes and calibration cup with small amount of 7 pH buffer. Discard buffer rinse. Repeat.
	3) Remove calibration cup from sonde. Place cup on work surface with uncapped end facing upward.
	4) Fill calibration cup with enough fresh buffer to cover the pH glass bulb and temperature sensor.
	5) Check temperature of pH buffer. Record value on calibration sheet. To view buffer temperature: 650 Main Menu ⇒ Sonde run
	6) Note: If the temperature at which you are calibrating is significantly different from 25° C, check the buffer bottle for the corrected pH value at the corresponding temperature (the corrected pH value will be used in step 8).
	6) Access calibrate menu for pH: 650 Main Menu ⇒ Sonde menu ⇒ Calibrate ⇒ ISE1 pH
	7) Choose either the 2-point or 3-point calibration.
	8) The prompt " Enter 1st pH " will appear. Enter 7.0 (or, if applicable, the corrected pH value from step 5).
	9) Real-time readings will be displayed. When readings have stabilized, record displayed pH value as "Initial Meter Reading" for Buffer # 1 on calibration sheet.
	10) Press ↵ (Enter) to calibrate.
	11) " Calibrated " will be displayed at the top of the screen. Record displayed pH value as "Calibrated Meter Reading" for Buffer #1.
	2ND CALIBRATION POINT:
12) Remove calibration cup from sonde. Note: "Calibrated" should still be displayed at the top of the screen. Remain on the same display screen as in Step 11 in order to see the real-time temperature reading for the 2 nd buffer. Do not return to the calibration menu.	
13) Rinse probes with distilled water.	
14) Rinse probes and calibration cup with small amount of 2 nd buffer (either 4 or 10 pH buffer).	
15) Fill calibration cup with enough fresh buffer to cover the pH glass bulb and temperature sensor.	
16) Real-time readings will be displayed. When readings have stabilized, record the temperature reading. Note: If the temperature at which you are calibrating is significantly different from 25° C, check the buffer bottle for the corrected pH value at the corresponding temperature (the corrected pH value will be used in step 18).	
17) Press ↵ (Enter) to return to the calibrate menu. The prompt "Enter 2 nd pH" will be displayed.	
18) At the " Enter 2nd pH " prompt, enter value of 2 nd buffer (or, if applicable, the corrected pH value from Step 16).	
19) Real-time readings will be displayed. When readings have stabilized, record displayed pH value as "Initial Meter Reading" for Buffer # 2 on the calibration sheet.	
20) Press ↵ (Enter) to calibrate.	
21) " Calibrated " will be displayed at the top of the screen. Record displayed pH value as "Calibrated Meter Reading" for Buffer #2.	
If you chose to do a "3-point calibration", repeat steps 12 through 21 using the 3 rd buffer.	
If only performing a 2-point calibration, press ↵ (Enter) and then Esc to return to the main menu.	
CONFIRMATION BUFFER:	
22) Rinse probes and calibration cup with distilled water.	
23) Rinse probes and calibration cup with small amount of 7.0 pH buffer. Discard buffer rinse. Repeat.	
24) Remove calibration cup from sonde. Place cup on work surface with the uncapped end facing upward.	
25) Fill the calibration cup with enough fresh buffer to cover the pH glass bulb and temperature sensor.	
26) Enter Run mode to view readings: 650 Main Menu ⇒ Sonde run	
27) Wait 1 to 3 minutes for pH readings to stabilize.	
28) Record the displayed pH value as the "Meter Reading" under "Confirmation Buffer 7.0" on the calibration sheet	
29) Confirm that the "Meter Reading" value is within ± 0.1 of the buffer value (between 6.9 and 7.1).	
Terminal Check (Post-Sampling Meter Check)	
a. Repeat steps 22 thru 29 (for 7 buffer); record displayed value on calibration sheet. Value should be within ±0.2 of 7.0.	
b. Repeat steps 22 thru 29 for Buffer #2. Record value on calibration sheet. Value should be within ±0.2 of Buffer #2.	

pH	YSI 6920
MAINTENANCE	<p><u>Indicators that maintenance is needed:</u> Difficulty calibrating pH sensor, slow response, erratic readings, clogged or black reference junction, coated glass bulb.</p>
	<p><u>Probe Storage:</u> Store probe in calibration/storage cup filled half-full with tap water (never use distilled water to store probe). If probe will not be used for several months, remove probe and store in pH 4 buffer or electrode storage solution.</p>
	<p><u>Probe Lifespan:</u> The pH probe has a lifetime of approximately 18-24 months (in some cases, probes may last 3+ years). When troubleshooting pH sensor problems, start by checking age of probe and replace as-needed: Near the silver stainless steel connector of each probe is the imprint "YSI 6561" followed by 2 numbers and a letter. The 2 numbers and the letter indicate the year and month in which the probe was made. For instance, 07D means the probe was made in April, 2007. (i.e. A=Jan, B=Feb, etc.).</p>
	<p><u>Troubleshooting with mV readings:</u></p> <ol style="list-style-type: none"> 1) Activate pH mV readings in the Report menu: 650 Main Menu ⇒ Sonde menu ⇒ Report ⇒ pH mV Note: pH mV is active when a black dot appears in the circle next to it. Press "Enter" to toggle between active and inactive. 2) Follow steps for pH calibration. During calibration, record pH mV values from the "Calibrated " screen for each buffer. 3) Evaluate the pH mV values: The span or "slope" between the pH 4 and pH 7 and between pH 7 and pH 10 should be approximately 165 to 180 mV. pH 7 should be 0 mV ± 50 mV. pH 4 should be 180 mV ± 50 mV. pH 10 should be -180 mV ± 50 mV. Example: If a probe reads +10 mV in pH 7 buffer, then the probe should also read between 175 and 190 mV in pH 4 buffer, and between -155 mV and -170mV in pH 10 buffer. 4) If the mV values fall outside the range of 160-180 mV, the probe should be replaced soon. Note: The probe will no longer calibrate when the span is outside of the range of 150-210 mV.
	<p><u>General pH Probe Cleaning:</u> Use deionized water and a soft lens cloth or a cotton swab to remove foreign material from the glass bulb. If good response is not restored, perform the following procedure:</p> <ol style="list-style-type: none"> 1) GENTLY clean the glass bulb and white probe face by carefully rubbing a cotton swab soaked in mild dishwashing detergent. Apply little to no pressure, as the glass bulb is very thin and fragile! 2) Rinse probe thoroughly with deionized water. 3) Wipe probe with cotton swab that has been saturated with water. Rinse probe again.
	<p><u>Advanced pH Probe Cleaning and Restoration:</u> To remove more resistant deposits and biological growth, use HCl acid and bleach. The need and frequency depend on the type of surface water being monitoring. The probe must be removed from the sonde before advanced cleaning. To perform an advanced cleaning, refer to Section 2.10.2 of the YSI 6-Series User Manual.</p> <p><u>Reference Junction:</u> The reference junction is a small tab located between the edge of the white surface of the pH probe face and the gray raised area around the pH probe face. When new, the junction will be an off-white color. As it ages, the junction will become darker. A black reference junction coupled with slow response and/or erratic readings indicates a more advanced cleaning may be needed.</p>

Appendix 4: DWR's YSI Pro Plus Multiparameter Guidance Sheet

DISSOLVED OXYGEN	YSI PRO PLUS with POLARGRAPHIC SENSOR		
CALIBRATION	<p><u>D.O. Calibration for YSI Meters with Polarographic Sensor:</u> <i>All calibrations should be performed in a controlled environment. Field calibrations are not recommended.</i></p> <p>I. BAROMETER CALIBRATION <i>Access the Barometer Calibration Menu:</i></p> <ol style="list-style-type: none"> 1) Press Cal key, highlight Barometer, and press Enter. 2) Highlight mmHg, and press Enter. 3) Highlight Calibration Value, and press Enter. Input the "true" barometric pressure (mmHg). Highlight <<<Enter>>>, and press Enter. <i>True barometric pressure is listed on the Dissolved Oxygen Table for your corresponding regional office.</i> 4) Wait for readings to stabilize. Record displayed value as "Initial Reading" in the "Barometer Calibration" section of the calibration sheet. 5) Highlight Accept Calibration and press Enter to calibrate Barometric Pressure. 6) "calibrating channel..." and then "saving configuration..." will be displayed at bottom of calibration screen before returning to the main screen. 7) Record displayed value as "Calibrated Value" in the "Barometer Calibration" section of the calibration sheet. <p>II. (% AIR CALIBRATION IN WATER-SATURATED AIR)</p> <ol style="list-style-type: none"> 8) Remove calibration storage cup from sonde. Confirm D.O. probe has been stored in moist environment. Place calibration cup on work surface with uncapped end facing upward. 9) Use lens tissue to carefully dry all sensors. Temperature and D.O. sensors must be completely dry. 10) Pour small amount of tap water into calibration cup (approximately 1/8" - just enough to completely cover the bottom of the calibration cup and create a 100% humid environment). Temperature and D.O. sensors CANNOT be in contact with water during calibration. 11) Carefully place probes (pointing downward) into calibration cup so that no water gets on the temperature or D.O. sensor. 12) Twist calibration cup onto sonde no more than 1 or 2 threads, so the cup is able to vent to the atmosphere. 13) Wait at least 15 minutes for D.O. sensor to stabilize. 14) Record the following values on calibration sheet: "Temperature", "Initial % Saturation", and "Initial Meter Reading (mg/L)". 15) Use the <i>Dissolved Oxygen Table</i> for your location to find the "D.O. Table Value" (based on the temperature displayed on the meter), and record value on calibration sheet. <p><i>Access the D.O. Calibration Menu:</i></p> <ol style="list-style-type: none"> 16) Press Cal key, highlight DO, and press Enter. 17) Highlight DO%, and press Enter. <p>NOTE: Dissolved oxygen should always be calibrated using % saturation. <i>Calibrations based on "mg/l" require a water sample with a known D.O. concentration (requires Winkler titration).</i></p> <ol style="list-style-type: none"> 18) In the "Dissolved Oxygen" section of the calibration sheet, record the "Barometric Pressure" displayed on the meter and the altitude for your location (provided on regional office <i>Dissolved Oxygen Tables</i>). 19) Highlight Accept Calibration and press Enter to calibrate Dissolved Oxygen. "calibrating channel..." and then "saving configuration..." will be displayed at bottom of calibration screen before returning to the main screen. 20) On the calibration sheet, record the displayed mg/L value as "Calibrated Meter Reading" and the DO% value as the "Calibrated % Saturation" value. <p>* NOTE: The "D.O. Table Value" and the "Calibrated Meter Reading" value should be within ±0.5 mg/L of each other.</p> <p><u>Terminal Calibration Check (Post-Sampling Meter Check)</u> <i>NOTE: Barometric pressure is not checked or recalibrated post-sampling.</i></p> <ol style="list-style-type: none"> a. Repeat calibration steps 8 thru 13. Record "Temperature", "% Saturation", "Initial Meter Reading". b. Record the barometric pressure and altitude for the location post-sampling checks are being performed. c. Repeat calibration step 15. d. The post-sampling "D.O. Table Value" and the post-sampling "Initial Meter Reading" should be within ±0.5 mg/L of each other. 		
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**DISSOLVED
OXYGEN****YSI PRO PLUS with POLARGRAPHIC SENSOR****MAINTENANCE****Cracked Probe:**

If meter readings are unusual and calibrating the meter does not correct the issue, check the condition of the D.O. probe – sometimes a crack can develop in the plastic along the side of the probe. Cracked probes should be replaced immediately.

Probe Storage:

Store probe in Calibration/Storage Cup with a small of tap water to create a 100% saturated air environment.

During storage, probes should not be submerged in water.

Do not use distilled water (this will damage the pH probe).

During long-term storage, inspect at least once a month to ensure the probe is still in a moist environment.

D.O. Membrane Replacement (YSI 5908- Yellow Teflon):

Replace electrode solution and membrane at least every 30 days during regular use or if: bubbles are visible under membrane; significant deposits of dried electrolyte are visible on membrane; calibration is impossible; readings are erratic or unstable; or membrane is damaged.

- 1) Remove and discard old membrane cap.
- 2) Rinse sensor tip with distilled or deionized water.
- 3) Prepare electrolyte solution (Na₂SO₄, KCl) according to the directions on the bottle (included in Membrane Cap Kit). Newly prepared solution must sit for 1 hour before using to prevent air bubbles under the membrane. When a new electrolyte solution is prepared, record preparation date (in permanent ink) on the side of the solution bottle. Discard electrolyte solutions 12 months after the recorded preparation date.
- 4) Fill new membrane cap half-full with electrolyte solution. Do not touch membrane surface. Tap side of cap lightly to release bubbles.
- 5) Screw membrane cap onto probe (small amount of electrolyte should overflow).
- 6) Re-attach probe sensor guard.

Cleaning Dirty, Tarnished Silver Anode and Gold Cathode:

SANDING AND CLEANING THE ELECTRODE ARE NOT PART OF THE ROUTINE MAINTENANCE AND SHOULD ONLY BE PERFORMED WHEN ABSOLUTELY NECESSARY! If performed too frequently, the electrode will be destroyed!

- 1) Remove membrane and soak probe overnight in 3% ammonium hydroxide (NH₄OH).
- 2) Rinse sensor tip with deionized water.
- 3) Use 400 or 600 grit wet/dry sandpaper to clean and polish the anode and cathode – no more than 3 to 4 twists of the sandpaper should be sufficient to remove any deposits or tarnish.
- 4) Rinse heavily with deionized water.
- 5) Install new membrane.
- 6) Turn meter "ON" and allow unit to stabilize for at least 30 minutes to 3 hours before calibrating.

May take several hours for the meter to stabilize.

SPECIFIC CONDUCTANCE	YSI PRO PLUS
CALIBRATION	<p><u>THREE-STEP SPECIFIC CONDUCTANCE PROCEDURE:</u></p> <p>I. "DRY AIR" (ALWAYS ZERO): The "Dry Air" step is a check for YSI meters.</p> <ol style="list-style-type: none"> 1) Attach calibration cup to probe. Fill cup half-full with deionized water and seal with lid. Shake probe to rinse. 2) Remove calibration cup. Place cup on work surface with the uncapped end facing upward. 3) Use a cotton swab to dry the inside of the conductivity cells. 4) Record displayed value as "Initial Meter Reading" in the "Dry Air" section of the calibration sheet. The probe should read close to zero (± 2). <p style="padding-left: 20px;">If the reading is not within ± 2, follow cleaning procedure, and repeat calibration procedure.</p> <p>II. CONDUCTIVITY STANDARD: Calibrations should be performed using a fresh, certified conductivity standard that is similar to the conductivity of the samples to be collected that day. Record the standard's "true value" (found on the certificate of analysis or bottle label) and lot number (also called analysis number) on the calibration sheet. Traceable® Certificate of Analysis for Conductivity Solution sheets (one certificate for each lot number) should be retained and stored in a notebook.</p> <ol style="list-style-type: none"> 5) Re-attach calibration cup. Fill cup half-full with deionized water. Seal cup with lid and shake probe to rinse. Discard rinse water. 6) Rinse sensors with small amount of conductivity standard. Discard rinse. 7) Pour fresh conductivity standard ($\geq 1000 \mu\text{S/cm}$) into the calibration cup. Make sure there is enough standard to cover the entire conductivity cell and temperature sensor when the probe is placed in the cup. 8) Tap or agitate sonde to remove air bubbles trapped in the conductivity cells. Air bubbles will result in erroneously low readings. 9) Press Cal key, highlight Conductivity, and press Enter. 10) Highlight Sp. Conductance and press Enter. 11) Highlight SPC- uS/cm and press Enter. 12) Highlight Calibration Value and press Enter. Input the True Value of the conductivity standard in microSiemens/cm ($\mu\text{S/cm}$). Highlight <<<Enter>>>, and press Enter. 13) Wait for readings to stabilize. Record displayed value as "Initial Meter Reading" in the "Conductivity Standard" section of the calibration sheet. 14) Highlight Accept Calibration and press Enter to calibrate meter. "Calibrating Channel..." and then "Saving Configuration..." will be displayed at bottom of calibration screen before returning to the main screen. 15) Record displayed value as "Calibrated Meter Reading" on calibration sheet. Never accept an out-of-range calibration (flagged by an error message on the meter). <p>III. CALIBRATION CHECK:</p> <ol style="list-style-type: none"> 16) Rinse with deionized water and wipe dry with a lens tissue or a lint-free cloth. 17) Confirm that the meter display is reading 0 (zero) μS before going to the next step. 18) Repeat steps 5-8 with a conductivity standard of a value different from the one used in the previous calibration steps. Choose a standard that will give the best range of values for the anticipated samples to be collected. 19) Record SpCond value as "Initial Meter Reading" in the Calibration Check section on the calibration sheet. The value must be within 10% of the standard. <p><u>Terminal Calibration Check (Post-Sampling Meter Check)</u></p> <ol style="list-style-type: none"> a. Repeat calibration steps 1 thru 4, and record value in the "Dry Air" section on the calibration sheet. For the "Dry Air" check, displayed value should be between -2 and 2 μS. b. Repeat calibration steps 5 thru 8. Record value in the "Conductivity Standard" section on the calibration sheet. "Conductivity Standard" value should be within $\pm 10\%$ of the standard. c. Repeat steps 16-19, and record value in the "Calibration Check" section on the calibration sheet. "Calibration Check" value should be within $\pm 10\%$ of the standard.

SPECIFIC CONDUCTANCE	YSI PRO PLUS	
MAINTENANCE	<p>* Never accept an out-of-range calibration! (flagged by an error message on the meter display)</p> <p><u>Checking the Conductivity Cell Constant:</u> When troubleshooting the conductivity probe, first check the cell constant.</p> <ol style="list-style-type: none"> 1) Press Folder Key. Highlight View GLP, and press Enter. Scroll to most recent conductivity calibration to view the Cal Cell Constant. 2) The value displayed next to "Cal Cell Constant" should be 5.0, \pm 0.45. Numbers outside of this range indicate a problem in the calibration process or that a contaminated standard was used to calibrate the meter. 3) If conductivity cell constant is not within the acceptance range (between 4.55 and 5.45), clean the cell, and reset the calibration cell constant (see instructions below). <p><u>Cleaning Conductivity Sensor:</u> Conductivity cell should be rinsed with deionized water after field use. Clean conductivity cell frequently. A clean cell is imperative for accurate readings.</p> <ol style="list-style-type: none"> 1) Dip small cleaning brush (provided with new meters) into distilled or deionized water and insert brush into each hole 15-20 times. For a more thorough cleaning, use a mild liquid or foam dishwashing detergent with the brush. 2) Rinse sensor thoroughly with deionized water. 3) Perform the Dry Air Check described in Calibration Steps 1-4 to ensure probe reads close to zero in air. <p><u>Reset Calibration Cell Constant:</u> <i>Reset the calibration cell constant by accessing the Calibrate menu:</i></p> <ol style="list-style-type: none"> 1) Press Cal Key, highlight Restore Default Cal, and press Enter. Highlight Conductivity, and press Enter. 2) The menu will ask "Are you sure you want to remove the current user calibration parameters for this channel?" Highlight Yes. Press the Enter key. 3) Recalibrate the meter using fresh, certified conductivity standards. 	
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pH	YSI PRO PLUS
CALIBRATION	<p>Two-point pH Calibration Required (Three-point pH Calibration is Optional):</p> <p>1ST CALIBRATION POINT (ALWAYS START WITH 7 BUFFER):</p> <ol style="list-style-type: none"> 1) Rinse probes and calibration cup with distilled water. 2) Rinse probes and calibration cup with small amount of 7 pH buffer. Discard buffer rinse. 3) Fill calibration cup with enough fresh 7 pH buffer to cover the pH glass bulb and temperature sensor. 4) Check temperature of pH buffer. Record value on calibration sheet. Note: If the temperature at which you are calibrating is significantly different from 25° C, check the buffer bottle for the corrected pH value at the corresponding temperature (the corrected pH value will be used in step 7). 5) Press Cal key. Highlight ISE1 (pH) and press Enter. 6) The prompt "Ready for point 1" will appear briefly at the bottom of the screen. Check Calibration Value. If value is correct, go to Step 7. If value is incorrect, highlight Calibration Value and press Enter. Input 7.0 (or, if applicable, the corrected pH value from step 4). Highlight <<<Enter>>>, and press Enter. 7) Actual-time readings will be displayed. When readings have stabilized, record displayed actual pH value as "Initial Meter Reading" for Buffer # 1 on calibration sheet. 8) Highlight Accept Calibration, and press Enter to calibrate. 9) "Ready for Point 2" will be displayed at the bottom of the screen very briefly. Record displayed pH calibration value as "Calibrated Meter Reading" for Buffer #1. <p>NOTE: IF YOU ACCIDENTALLY LEAVE THE pH CALIBRATION MENU BEFORE CALIBRATING YOUR 2ND POINT, YOU MUST START OVER BECAUSE THE 1ST CALIBRATION POINT WAS NOT COMPLETED. "Calibrate ISE1 (pH)" should still be displayed at the top of the screen. Remain on the same display screen as in Step 9 in order to see the actual-time temperature reading for the 2nd buffer.</p> <p>2ND CALIBRATION POINT:</p> <ol style="list-style-type: none"> 10) Rinse probes and calibration cup with distilled water. 11) Rinse probes and calibration cup with small amount of 2nd buffer (either 4 or 10 pH buffer). Discard buffer rinse. 12) Fill calibration cup with enough fresh buffer to cover the pH glass bulb and temperature sensor. 13) Actual-time readings will be displayed. When readings have stabilized, record the temperature reading. Note: If the temperature at which you are calibrating is significantly different from 25° C, check the buffer bottle for the corrected pH value at the corresponding temperature (the corrected pH value will be used in step 14). 14) Check Calibration Value. If value is correct, go to Step 15. If value is incorrect, highlight Calibration Value and press Enter. Input correct buffer value (or, if applicable, the corrected pH value from step 13). Highlight <<<Enter>>>, and press Enter. 15) Actual-time readings will be displayed. When readings have stabilized, record displayed actual pH value as "Initial Meter Reading" for Buffer # 2 on the calibration sheet. 16) Highlight Accept Calibration and press Enter to calibrate. 17) "Ready for Point 3" will be displayed briefly at the bottom of the screen. If only performing a 2-point calibration, press Cal Key to complete calibration process. 18) Record displayed pH value as "Calibrated Meter Reading" for Buffer #2. <p>If you chose to do a "3-point calibration", do NOT press Cal key in step 17 and repeat steps 10 through 17 using the 3rd buffer.</p> <p>CONFIRMATION BUFFER: CONFIRMATION BUFFER STEP IS VERY CRITICAL FOR THE YSI PRO PLUS – DO NOT SKIP IT!</p> <ol style="list-style-type: none"> 20) Rinse probes and calibration cup with distilled water. 21) Rinse probes and calibration cup with small amount of 7.0 pH buffer. Discard buffer rinse. 22) Fill the calibration cup with enough fresh buffer to cover the pH glass bulb and temperature sensor. 23) Wait 1 to 3 minutes for pH readings to stabilize. 24) Record the displayed pH value as the "Meter Reading" under "Confirmation Buffer 7.0" on the calibration sheet 25) Confirm that the "Meter Reading" value is within ± 0.1 of the buffer value (between 6.9 and 7.1). <p>Terminal Check (Post-Sampling Meter Check)</p> <ol style="list-style-type: none"> a. Repeat steps 20 thru 23 (for 7 buffer); record displayed value on calibration sheet. Value should be within ± 0.2 of 7.0. b. Repeat steps 20 thru 23 for Buffer #2. Record value on calibration sheet. Value should be within ± 0.2 of Buffer #2.
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pH	YSI PRO PLUS
MAINTENANCE	<p>★ Never accept an out-of-range calibration! (flagged by an error message on the meter display)</p> <p><u>Indicators that maintenance is needed:</u> Difficulty calibrating pH sensor, slow response, erratic readings, clogged or black reference junction, coated glass bulb.</p> <p><u>Probe Storage:</u> Do NOT allow the pH sensor to dry out! Sensors that have dried out may be permanently damaged! Store probe in calibration/storage cup filled with 1/8" of tap water (never use distilled water to store probe). If probe will not be used for several months, remove probe and store in pH 4 buffer. Seal the vacant port with a port plug.</p> <p><u>Probe Lifespan:</u> The pH probe has a lifetime of approximately 12-24 months (in some cases, probes may last 3+ years). When troubleshooting pH sensor problems, start by checking age of probe and replace as-needed: On the side of each probe is the imprint "YSI 1001" followed by 2 numbers and a letter. The 2 numbers and the letter indicate the year and month in which the probe was made. For instance, 07D means the probe was made in April, 2007. (i.e. A=Jan, B=Feb, etc.).</p> <p><u>Troubleshooting with mV readings:</u></p> <ol style="list-style-type: none"> 1) Follow steps for pH calibration. During calibration, record pH mV values from the "Calibrated" screen for each buffer. 2) Evaluate the pH mV values: The span or "slope" between the pH 4 and pH 7 and between pH 7 and pH 10 should be approximately 165 to 180 mV. pH 7 should be 0 mV ± 50 mV. pH 4 should be 180 mV ± 50 mV. pH 10 should be -180 mV ± 50 mV. Example: If a probe reads +10 mV in pH 7 buffer, then the probe should also read between 175 and 190 mV in pH 4 buffer, and between -155 mV and -170mV in pH 10 buffer. 3) If the mV values fall outside the range of 160-180 mV, the probe should be replaced soon. Note: The probe will no longer calibrate when the span is outside of the range of 150-210 mV. <p><u>General pH Probe Cleaning:</u> Use deionized water and a soft lens cloth or a cotton swab to remove foreign material from the glass bulb. If good response is not restored, perform the following procedure:</p> <ol style="list-style-type: none"> 1) GENTLY clean the glass bulb and white probe face by carefully rubbing a cotton swab soaked in mild dishwashing detergent. Apply little to no pressure, as the glass bulb is very thin and fragile! 2) Rinse probe thoroughly with deionized water. 3) Wipe probe with cotton swab that has been saturated with water. Rinse probe again. <p><u>Advanced pH Probe Cleaning and Restoration:</u> The need and frequency depend on the type of surface water being monitoring. The probe must be removed from the sonde before advanced cleaning. To remove more resistant deposits and biological growth, use HCl acid and bleach. To perform an advanced cleaning, refer to the Care, Maintenance, and Storage section of the YSI Professional Plus User Manual.</p> <p><u>Reference Junction:</u> The reference junction is a small tab located between the edge of the white surface of the pH probe face and the gray raised area around the pH probe face. When new, the junction will be an off-white color. As it ages, the junction will become darker. A black reference junction coupled with slow response and/or erratic readings indicates a more advanced cleaning may be needed.</p>
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APPENDIX 5: Uncorrected Dissolved Oxygen Table

**Corrected D.O. Tables
for a specific location
or DWR office are
available upon
request from the ESS**

**Sea Level (Uncorrected D.O. Values)
Dissolved Oxygen (D.O.) TABLE**

Altitude at Sea Level = 0 feet

Barometric Pressure (BP) at Sea Level = 760 mm Hg

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

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

Uncorrected Table

08/24/2007

D.O. Correction Chart

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

How to Correct D.O. Table Values:

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

- 1) Use the temperature displayed on your meter and the "Sea Level Table" (on the back of this page) to find the *Uncorrected D.O. Value*.
- 2) Use your location's altitude and the "D.O. Correction Chart" (on this page) to find the corresponding *Correction Factor*.
- 3) Multiply the *Uncorrected D.O. Value* (from step 1) by the *Correction Factor* (from step 2) to get the *Corrected D.O. Value*.
- 4) The value calculated in step 3 (*Corrected D.O. Value*) and the value displayed on the meter should be within ± 0.5 mg/L of each other.

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

D.O. Correction Factors

08/24/2007

APPENDIX 6: SOP for Filtering in the Field

STANDARD OPERATING PROCEDURES FOR FIELD FILTERING USING THE VACUUM PUMP PROCEDURE.**Field Procedure:**

1. Obtain filtering equipment including sterile 0.45 μ m 47 mm diameter Millipore filters, glass fiber filters, nitrile gloves, forceps, and a supply of deionized (DI) water. An example of an appropriate filtering kit is Nalgene - filter holder with receiver 500 mL (Nalgene #300-4050).
2. After donning gloves, thoroughly rinse the field filtering equipment with deionized water on the day of sampling at the first sampling station.
3. Remove 0.45 μ m filter from package with clean forceps and place on the filter platform, gridded side up.
4. Inspect filter for proper placement-centered; no wrinkles, bends, cracks, holes, or gaps.
5. Reassemble apparatus.
6. Attach hand pump to outlet of bottom chamber with tubing.
7. If first sample of the day, do field blank for quality control first using DI water and following steps 8-16.
8. Pour required volume of sample water in the top chamber (example-volume for orthophosphorus and dissolved phosphorus is at least 200 mL for each).
9. Use hand pump to create vacuum.
10. Continue adding sample and pumping until required filtered volume (based on parameter) is obtained and top chamber is empty. **Note:** It may be necessary to change filters several times or use a glass fiber pre-filter (see turbid samples options below) to obtain enough filtrate.
11. Samples for all dissolved parameters can be filtered at once.
12. Before disassembling, make sure that no sample remains in the top chamber and no pressure in the bottom chamber: remove tubing, or press release on pump.
13. Disassemble apparatus.
14. Decant filtrate into sample bottles, preserve and handle as per laboratory guidance.
15. Remove filter with forceps and dispose of filter.
16. Rinse filtering apparatus with DI water. This rinse must be repeated before field filtering at any additional locations (i.e. between stations).
17. After last sample of the day is completed, do terminal field blank sample.

Turbid Samples Options:

When the filter becomes clogged:

Option 1: Change filters

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

*Field Filtering SOP (Cont.)*Option 2: Pre-filter

1. The sample can be taken through a preliminary step using a filter with a larger pore size, such as a glass fiber filter.
2. This can be accomplished by placing the glass fiber filter on top of the 0.45 μm filter on the filter platform. A small amount of DI water can be squirted on top of the combined filters to prevent vapor lock. It may be necessary to change these filters as they get clogged to obtain enough filtrate but this procedure should minimize the number of times the filters must be changed.

Quality Control Procedures

1. The filtering apparatus and DI wash bottle should be regularly cleaned with phosphate-free detergent and completely rinsed with DI water, as is done with all other sampling equipment.
2. Initial and terminal quality control samples (blanks) of filtered deionized water must be taken for each day of sampling for each parameter and submitted to the laboratory.
3. Blanks must be filtered in the field: one at the beginning of the day before the first water sample is processed, and one at the end of the day after the last water sample is processed.
4. Sources of contamination include:
 - air/environment;
 - field staff;
 - sampling equipment and bottle;
 - filtration equipment (filter holder, filter, tubing);
 - DI water, and
 - chemical preservatives.
5. Station location on the lab sheet should indicate QC sample type.
6. Blanks should come back as non-detects.
7. If blanks show detectable levels of analytes:
 - results from associated samples must be flagged, and flags reported to data users.
 - perform rigorous data review to see if contamination concerns are severe enough to warrant discarding the data.
 - patterns of dirty blanks should be reviewed and a plan for contamination source identification, corrective actions, and re-evaluations should be developed.

