Standard Operating Procedures for Tidal Monitoring

Integration of Citizen-based and Nontraditional Monitoring into the Chesapeake Bay Program Partnership

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In cooperation with
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Acknowledged Works

Much of the information in this manual has been adapted from the following methods manuals:

Alliance for the Chesapeake Bay. 2012. Citizen Monitoring Program Manual

EcoCheck. (2011). Sampling and data analysis protocols for Mid-Atlantic tidal tributary indicators. Wicks EC, Andreychek ML, Kelsey RH, Powell SL (eds). IAN Press, Cambridge, Maryland, USA.

Virginia Citizen Water Quality Monitoring Program. 2007. Virginia Citizen Water Quality Monitoring Program Methods Manual

Center for Marine Conservation & U. S. EPA. Volunteer Estuary Monitoring: A Methods Manual, Second Edition.

U.S. EPA. 1997. Volunteer Stream Monitoring: A Methods Manual. EPA 841-B-97-003.

U.S. EPA. 1996. Recommended Guidelines for Sampling and Analyses in the Chesapeake Bay Monitoring Program. EPA 903-R-96-006.

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1 Before You Begin

1.1 Safety, Equipment List, and Volunteer Responsibilities

1.1.1 Safety – General Precautions

a) Always perform water-monitoring activities under the guidance of an adult.

b) Read all instructions to familiarize yourself with the test procedure before you begin. Note any precautions in the instructions.

c) Keep all equipment and chemicals out of the reach of young children and pets.

d) Avoid contact between chemicals and skin, eyes, nose and mouth.

e) Read the label on each reagent container prior to use. Some containers include precautionary notices or antidote information on the back of the container.

f) In the event of an accident or suspected poisoning, immediately call the Poison Control Center phone number in the front of your local telephone directory or call your physician. Be prepared to give the name of the reagent in question and its code number. Most kit reagents are registered with POISINDEX, a computerized poison control information system available to all local poison control centers.

1.1.2 Protect Yourself & Your Equipment: Use Proper Technique

a) Wear safety goggles or glasses when handling reagent chemicals.

b) Use the test tube caps or stoppers, not your fingers, to cover test tubes during shaking or mixing.

c) When dispensing a reagent from a plastic squeeze bottle, hold the bottle vertically upside-down (not at an angle) and gently squeeze it (if a gentle squeeze does not suffice, the dispensing cap or plug may be clogged).

d) Wipe up any reagent spills, liquid or powder, as soon as they occur. Rinse area with a wet sponge, and then dry.

e) Thoroughly rinse test tubes before and after each test. Dry your hands and the outside of the tubes.

f) Tightly close all reagent containers immediately after use. Do not interchange caps from different containers.

g) Avoid prolonged exposure of equipment and reagents to direct sunlight. Protect them from extremely high temperatures. Protect them from freezing.

1.2 Monitor Responsibilities

Choose a regular sampling day: Choose a convenient day of the week for sampling. Samples should be taken at regular weekly or monthly intervals. If it is not possible to sample on the same day each week, try to sample within 2 days (either side) of your regular day spacing the sampling dates, 5 to 9 days apart. Sample at the same time of day each week; if you are sampling multiple locations, be sure to always sample your sites in the same order each monitoring run to achieve similar sample timing.

Record your test results: Record data on a data collection form provided. Always record the test results as you go along. Keep a copy of the data collected for your records and to provide a backup copy should the original be lost.

Provide comments as necessary: The "Comments" section can be used to record general observations about the site especially changes due to erosion, recent notable weather, and any problems you had with the sampling procedures.

Submit data to database: If you have access to the internet, submit your data to the project's online database.

Send datasheets once every three months. Mail the data sheets to the Alliance or your Watershed Coordinator every three months so that we can maintain a current database.

Stay certified: Attend a recertification session every year for the first two years of monitoring and then every other year after that to maintain your skills and learn new information and techniques. You can also attend any training session to refresh yourself of the concepts and procedures between re-certifications.

2 QA/QC Procedures

2.1 Certification and Re-certification

2.1.1 Certification

All monitors that wish to submit Tier II data must gain monitor certification. Monitors can become certified at their initial training session by demonstrating a mastery of the sampling procedures and complete understanding of the quality assurance protocols used during data collection to be assessed by a Project Team member or Certified Trainer. Monitors must also pass a test that assesses the monitor's understanding of QA/QC procedures outlined in this SOP and the project QAPP with a 90% score. The certification test must be taken by the monitor at least one day after their training event to ensure that they have retained the information.

Monitors that attend an initial training and are unable to pass the requirements to become certified at the end of the training will be encouraged to continue practicing their monitoring procedures. Un-certified monitors are encouraged assist a certified monitors in the field until they have become comfortable with the procedures and QA/QC protocols. Un-certified monitors are allowed to retake the certification test, and demonstrate proper sampling and analysis technique up to three times in order to become a certified monitor.

When a monitor achieves certification, they may be assigned a site and begin to collect Tier II data and submit it to the project database.

2.1.2 Re-certification

The Project Team and Certified Monitors will host recertification sessions annually and biennially for monitors that have passed the initial training and wish to maintain their certification. Recertification sessions are conducted in a fashion that is similar to a lab practical. Monitors are checked to assure that: they remain proficient in methodology and understanding of basic water quality parameters; their equipment is operational and properly calibrated / verified; and they have an adequate supply of viable chemicals, procedures, equipment verification/check, and updated information about monitoring.

The recertification session is set up with a "station" for each water quality parameter. Monitors perform the test and compare their results to a known or controlled result. Project staff observe the monitors' methods and ensure that monitors correctly perform the tests and accurately record the data. After completing and "passing" one parameter, the monitor moves through each of the other stations while completing a datasheet that serves as documentation of re-certification. Replacement equipment, datasheets, information, and chemicals are given if needed. Alliance for the Chesapeake Bay retains documentation of recertification sessions.

2.1.3 Field Audits

Project Team members, the QC manager, or Certified Trainers may accompany monitors in the field and observe field collection procedures as part of the recertification process for monitors. Monitors will demonstrate proper sample collection, analysis, labelling, and preservation in accordance with this SOP.

2.2 Pre-monitoring checks

2.2.1 Equipment Check

Prior to going out into the field, monitors should check their equipment for cleanliness, breakage, probe function and battery life, and chemical expiration dates. If a monitor finds that their equipment is damaged and will affect the quality of the data they collect they will not collect data that day and mark the reason on their data sheet. The monitor should contact their Project Team member to get the equipment repaired or replaced prior to the next scheduled sample.

Monitors measuring dissolved oxygen using the Winkler titration will check the viability of their sodium thiosulfate solution prior to each monitoring event and record the results on their field datasheet. Sodium thiosulfate is used for monitoring dissolved oxygen. By using a standard solution of iodate-iodide, with 10 mg/L dissolved oxygen value, the monitor must record a value of 9.4 – 10 mg/L with their sodium thiosulfate measurement.

If results of the first check are above or below these intended values, a second check is performed. If the second check yields unacceptable values or if the two checks are greater than 0.4 mg/L apart from each other, the monitor is instructed to abandon the dissolved oxygen test because the sodium thiosulfate is no longer viable. The monitor must replace all expired chemicals prior to sampling again.

2.2.2 Calibration

Monitors will calibrate any equipment that requires calibration prior to being used (within 24 hours of use), using standard solutions and following the manufacturer's instructions. Monitors will note on their data sheet that they calibrated their equipment.

After sampling, it is recommended that monitors check their probes against the standard solutions used for calibration to identify instrument drift. If pH is outside of +/- 0.20 units, DO is +/- 0.3 mg/L, or specific conductance is +/- 5% of verification standards, the data must be flagged and the probe must be assessed and fixed or replaced if needed.

Monitors record these calibration and verification values on their datasheet and values are entered into the online database.

Thermometers that are verified should be re-verified every year. Thermometers must be verified against the Alliance master precision thermometer that is annually verified against an NIST-traceable thermometer to 0.2° C. If the Alliance thermometer is found to be reading beyond 0.2° C of a NIST-traceable thermometer it will be discarded and replaced and the new thermometer will be verified. Temperature data collected from thermometers confirmed by the Alliance thermometer found to be out of compliance will be flagged as not verified and downgraded a tier back dated to the last calibration check.

2.3 Field QC

2.3.1 Duplicates

If monitors are using the Winkler titration method for measuring DO they will perform the dissolved oxygen test on the actual water sample in duplicate. Monitors are instructed to do a third titration if their two initial titrations differ by more than 0.6mg/L. The two closest values are recorded on the datasheet.

Monitors collecting samples for Tier II laboratory analysis will perform duplicate samples at least 10% of the time. Duplicates consist of either collecting a larger sample for mixing and splitting it between two containers or immersing sample containers side by side in the water at the same time.

2.3.2 Replicates

Duplicates of field parameters generally do not occur as monitors have one set of equipment and field parameters change too quickly for a true duplicate to occur. However, a field replicate can be performed for many field parameters where the monitor takes the instrument to the site and obtains a reading. The instrument is removed or a new sample is collected and a second reading is obtained from the exact location and depth of the first sample. Monitors will perform replicate samples of all other parameters (DO using Winkler titration method must be done in duplicate each sample) 10% of the time. The quality control samples are prepared and analyzed for all parameters of interest. The field replicate data are used to determine the overall precision of the field and laboratory procedures.

2.3.3 Field Blanks

A field blank is sample of analyte-free deionized water supplied by a laboratory and processed in the field as a regular sample and then returned to the lab for analysis. Monitors will perform blank samples 10% of the time for samples to be sent to a lab for analysis. Monitors will perform all field procedures including preserving the samples as required and taking to the lab for analysis using deionized water provided by the laboratory. Results from field blanks will be recorded and appropriately marked during database entry.

3 Field Monitoring Procedures

3.1 Field Sampling Procedures

3.1.1 Best Practices

a) Use of protective gloves. Gloves serve a dual purpose: 1) protecting the sample collector from potential exposure to sample constituents and 2) minimizing accidental contamination of samples by the collector. Wearing protective gloves at all times while sampling is recommended. Latex or nitrile gloves may be used for common sampling conditions.

b) Safety always comes first. All sampling should be conducted with the proper equipment and least amount of danger to field personnel.

c) Permission must be obtained from landowners before entering private property.

d) Care should be taken not to disturb the bottom when sampling. When entering a stream, always walk in an upstream direction.

e) Surface water should always be collected facing upstream and in the center of main area of flow. Therefore, unless safety is an issue, samples should be obtained from a bridge or instream.

f) Samples should be collected in the main flow representative of the stream you are monitoring (for small streams, this is usually mid-channel).

   i. If you are sampling from a boat in Virginia, and have appropriate equipment, surface samples should be taken at a depth of 1 m.
   ii. If you are sampling from a boat in Maryland, and have appropriate equipment, surface samples should be taken at a depth of 0.5 m.
   iii. If you are unable to collect at these depths, due to equipment limitations, surface samples can be taken at 0.3 meters depth. All sample depths must be recorded on your data sheet.

g) Whenever possible, collect field measurements directly from the sample site, not from bucket. If the field parameters need to be measured in the bucket, collect water quality samples (nutrients, etc.) first before placing the multi probe instrument in the bucket.

h) When there are obvious standing pools of water during low or no flow conditions, do not collect samples or field measurements. Make a note of this on the data sheet.

i) When collecting bacterial samples:

   i. DO NOT rinse the bacteria sample bottle before collecting the sample.
   ii. If sample bottles contain a dechlorinating tablet (usually small white tablet) and

you are collecting an unchlorinated sample, dump out the tablet before collecting the sample.

iii. Be careful not to insert fingers into the mouth of the container or on the interior of the cap.

3.1.2 Sampling from a Boat

Using a probe

If you are measuring with a multi probe and have a long enough cord, you are encouraged to do depth profiles. If the depth is ≤3 m deep take a surface measurement 1 m below the surface in Virginia and 0.5 m below the surface in Maryland and a bottom measurement at 1 m above the bottom. If the depth is >3 m measure 1.0 m above the bottom, then 1 meter intervals up to 1 m below the surface in Virginia and take an additional measurement at 0.5 m below the surface in Maryland (Example: At a 3.4 m deep site, measure at 2.4, 2.0, 1.0 m and take an additional measurement at 0.5 m if in Maryland). At each iteration allow the probe to stabilize before recording your reading at the corresponding depth.

If you only are taking surface measurements, place your probe 0.5 m beneath the surface if sampling in Maryland and 1 m beneath the surface if you are sampling in Virginia, wait for the probe to stabilize, and then record your reading. If you are unable to measure at these depths due to equipment limitations you may sample at 0.3 m beneath the surface. All sampling depths must be recorded on your field sheet.

If the meter is not equipped with a pressure gauge for depth estimation and the current is strong enough to pull the meter so that the cable is at an angle noticeably different than vertical, estimation of depth will have to be corrected. Weighted probe guards may help prevent displacement by current.

Using sample bottles

For chlorophyll and nutrient grab samples, rinse the sample bottles three times with sample water. If sampling for chlorophyll, rinse your syringes three times with sample water as well. Drain the bottle until it is empty, put the cap on, lower it one forearm's length under water (about 0.3 meters) then remove the cap. Wait for the bottle to fill, then cap it and return it to the surface.

3.1.3 Streambank and Instream Sampling

If possible, wade into the stream to collect the sample. If wading to the sample site, always proceed upstream to allow the flow of the water to push any disturbed sediment downstream of where you will be collecting the sample.

Volunteers can sample from a streambank if they are unable to collect a mid-channel sample by wading, from a boat, or from a bridge or if conditions are unsafe. When sampling from the streambank, care should be taken to sample from an area that will most closely represent the entire stream. Typically, this will be the area of the greatest flow in the stream and away from stagnant pools or eddies.

Step	Bacteria Samples	Nutrient and Chlorophyll Samples
1.	Walk upstream to the sample location. Be sure any sediment or debris disturbed from your movement in the streambed is not present where you will collect the sample.	Walk upstream to the sample location. Be sure any sediment or debris disturbed from your movement in the streambed is not present where you will collect the sample.
2.	Submerge the container; neck first into the water. The mouth of the bottle should be completely below the water surface approximately 0.3 m or 1 foot.	Lower the capped sample bottle 0.3 m or 1 foot beneath the surface and up-cap the bottle to fill.
3.	Invert the bottle so the neck is upright and pointing into the water flow.	Allow the bottle to fill to the neck of the bottle.
4.	Move the bottle forward away from the body for at least six inches.	Lift the filled container. Do not pour out any excess water.
5.	Return the filled container quickly to the surface. Pour any excess water and cap.	

3.1.4 Dock or Bridge Sampling

1. Sample in the center of main flow from or as close as you can get on the dock. If sampling from a bridge sample from the safest side of the bridge and where contamination is least likely to occur. Typically, sampling on the upstream side of the bridge or dock is less likely to be contaminated.

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

3. Obtain field parameters (DO, pH, temperature) first before lowering a sample bucket.

4. When lowering the sample bucket, allow it to fill ¼ the way full and retrieve. Swirl the contents and dump the rinse away from the sample location to avoid kicking up sediment.

5. Repeat step 4 two more times and on the final time fill ½ to ¾ the way full.

6. Retrieve the bucket and collect the samples in the following order. Be sure to gently stir the

water with a clean tool before taking samples or measurements (do not introduce air bubbles).

1. Bacteria
   - Open the bottle without touching the inner wall of the bottle or lid.
   - Invert the bottle by holding to the main body of the bottle and lower into the bucket 3-6 inches.
   - Fill the bottle in a 'U' from the side of the bucket closest to you to the opposite end.
   - At the end, bottle opening should be facing up and remove from the bucket.
   - Pour off any excess water and cap with the lid.
   - If collecting a replicate sample, hold two bottles in one hand to fill both bottles at the same time.

2. Nutrients and Chlorophyll
   - Open the bottle and tilt so that one side of the bottle will be below the waterline of the bucket.
   - Allow the bottle to fill to the neck of the bottle.
   - Remove the bottle and cap. Do not pour off any excess sample.
   - If collecting a replicate sample, collect using two bottles simultaneously and fill both bottles at the same time.

7. In situations where field parameters must be obtained from the bucket, all water samples must be collected prior to inserting the probe in the bucket.

8. When performing a replicate sample using a probe, take your first measurement, remove the probe, take a second measurement, and record both measurements on your data sheet.

9. If you sampled using a bucket, mark your data sheet to indicate your sampling technique.

3.2 Air Temperature Measurement

Equipment: armored, digital thermistor, or probe

Temperature is reported in degrees Celsius (ºC). Always measure air temperature before water temperature.

Method:

1. Locate a place near your site and hang the thermometer out of the direct sun.

2. Wait 3-5 minutes to allow the thermometer to equilibrate.

3. Record air temperature to the nearest 0.5 ºC for the armored thermometer or to the nearest tenth of a degree for the digital thermistor or probe on Page 2 of the datasheet.

3.3 Recording General Observations

Record weather and general observations on the datasheet.

3.4 Water Clarity & Turbidity Measurement

3.4.1 Secchi Disk

Equipment: 8" Secchi disk with attached line

Method:

1. Remove sunglasses if you are wearing them and stand with the sun to your back. Try to lower the disk into a shaded area.

2. Lower the disk into the water until the disk barely disappears from sight. Note the depth reading, in meters, based on the length of line submerged. Each mark is one-tenth (or 0.1) meter.

3. Slowly raise the disk and record the depth at which it reappears (i.e. is barely perceptible).

4. Average the two depth readings obtained above. The average of the two readings is considered to be the limit of visibility, or index of transparency. Record this average to the nearest tenth of a meter on your data form.

3.4.2 Transparency Tube

Transparency tubes are a type of equipment used for measuring transparency of water in streams and rivers. They are helpful for measuring transparency in situations where the stream is too shallow for the Secchi disk to be practical and for running waters where flow is too fast that the Secchi disk cannot remain vertical. Sample water collected either directly from the stream or from the sampling bucket is analyzed.

Equipment: Transparency tube

Method:

1. Close the drain tube by squeezing the crimp.

2. Fill the transparency tube with your sample water. Water may be collected directly from the stream in the vicinity of the sampling location if the stream is too small to fill the bucket, or sample water collected in the sampling bucket may be used (See 5.4, "Collecting the Water Sample"). To collect water directly from the stream, point the top of the tube in the upstream direction and collect surface water, being careful not to disturb the stream bed. To analyze water collected in the bucket, pour sample water from the bucket water directly into the transparency tube.

3. While looking down through the opening of the tube, partially open drain crimp, slowly draw off sample (Control flow by squeezing the crimp).

4. When the black and white pattern begins to appear, immediately tighten the crimp.

5. Record the level of water remaining via the centimeter ruler on the side of tube.

3.4.3 Turbidity Kit

This test is performed by comparing the turbidity of a measured amount of the sample with an identical amount of turbidity-free water containing a measured amount of standardized turbidity reagent. The readings are made by looking down through the column of liquid at a black dot. If turbidity is present, it will interfere with the passage of light through the column of liquid. Small amounts of turbidity will cause a "blurring" of the black dot in the bottom of the tube. Large amounts of turbidity may provide sufficient "cloudiness" so that it is not possible to see the black dot when looking down through the column. Any color that may be present in the sample should be disregarded. This determination is concerned only with the haziness or cloudy nature of the sample.

Equipment: Turbidity kit – LaMotte 7519-01

Method:

1. Fill one Turbidity Column to the 50 mL line with the sample water. If the black dot on the bottom of the tube is not visible when looking down through the column of liquid, pour out a sufficient amount of the test sample so that the tube is filled to the 25 mL line.

2. Fill the second Turbidity Column with an amount of turbidity-free water that is equal to the amount of sample being measured. Distilled water is preferred; however, clear tap water may be used. This is the "clear water" tube.

3. Place the two tubes side by side and note the difference in clarity. If the black dot is equally clear in both tubes, the turbidity is zero. If the black dot in the sample tube is less clear, proceed to Step 4.

4. Shake the Standard Turbidity Reagent vigorously. Add 0.5 mL to the "clear water" tube. Use the stirring rod to stir contents of both tubes to equally distribute turbid particles. Check for amount of turbidity by looking down through the solution at the black dot. If the turbidity of the sample water is greater than that of the "clear water", continue to add Standard Turbidity Reagent in 0.5 mL increments to the "clear water" tube, mixing after each addition until the turbidity equals that of the sample. Record total amount of Standard Turbidity Reagent added.

5. Each 0.5 mL addition to the 50 mL size sample is equal to 5 Jackson Turbidity Units (JTUs). If a 25 mL sample size is used, each 0.5 mL addition of the Standard Turbidity Reagent is equal to 10 Jackson Turbidity Units (JTUs). See Table 3.4-1 below. Rinse both tubes carefully after each determination.

Table 3.4-1-1. Turbidity Test Results – from LaMotte 7519-01 instructions

TURBITITY TEST RESULTS

Number of Measured Additions	Amount in mL	50 mL Graduation	25 mL Graduation
1	0.5	5 JTU	10 JTU
2	1.0	10 JTU	20 JTU
3	1.5	15 JTU	30 JTU
4	2.0	20 JTU	40 JTU
5	2.5	25 JTU	50 JTU
6	3.0	30 JTU	60 JTU
7	3.5	35 JTU	70 JTU
8	4.0	40 JTU	80 JTU
9	4.5	45 JTU	90 JTU
10	5.0	50 JTU	100 JTU
15	7.5	75 JTU	150 JTU
20	10.0	100 JTU	200 JTU

3.5 Water Temperature Measurement

Equipment: armored, digital thermistor, or probe

Method:

Depth Profile Sampling (>3m):
If you are measuring with a multi probe and have a long enough cord, you are encouraged to do depth profiles.

1. Measure 1.0 m above the bottom, then move your sensor up to the next whole integer depth, then proceed at 1 meter intervals up through the water column until you reach 1 m below the surface if sampling in Virginia, take an additional measurement at 0.5 m below the surface if sampling in Maryland. (Example: At a 3.4 m deep site, measure at 2.4, 2.0, 1.0 m and take an additional measurement at 0.5 m if in Maryland)

2. At each iteration allow the probe to stabilize before recording your temperature reading at the corresponding depth

3. Measure salinity and DO at each depth as well

4. Record depth, DO, temperature, and salinity on your data sheet for each depth

Depth Profile Sampling (≤3 m):

1. Measure 1.0 m above the bottom, allow the probe to stabilize and record your result

2. Measure 0.5 m below the surface if sampling in Maryland and 1 m below the surface if sampling in Virginia, allow the probe to stabilize and record your result

Surface Sampling with Probe:

1. Place your probe 0.5 m beneath the surface of the water if sampling in Maryland and 1 m beneath the surface if sampling in Virginia.

2. Wait for the probe or thermometer to stabilize

3. Record your reading

Surface Sampling with Individual Thermometer:

1. Place your thermometer 0.5 m beneath the surface of the water if sampling in Maryland and 1 m beneath the surface if sampling in Virginia.

2. Wait for the thermometer to stabilize

3. Record your reading

Sample with bucket:

1. Hang thermometer in the bucket
2. Wait for the probe or thermometer to stabilize
3. Record your reading. Mark on your data sheet that the measurement was taken from a bucket.

3.6 Water Depth Measurement

Equipment: Secchi disk (for <3 m deep), measuring tape with weighted end, or DO probe with marked lengths (if doing depth profile sampling)

Method:

1. At your sampling site, lower the measuring device into the water until it is resting on the bottom and the line is slack.

2. Record the depth reading, to the nearest tenth, based on the length of line submerged.

3.7 Dissolved Oxygen

3.7.1 Winkler Titration Method

Equipment: LaMotte Dissolved Oxygen Test Kit

Sodium Thiosulfate Check:
Prior to each sampling event (either the night before or the day of), you must run a test to make sure your Sodium Thiosulfate is still fresh and functional. Sodium Thiosulfate is fairly unstable and can degrade very suddenly, making it necessary to check it before each DO sampling. Perform this check at home before you go out. It is important to perform this check in a room temperature environment at 20°C. Here is how you do the check…

1. Rinse the titrating tube (small glass vial with plastic lid with hole in it) with a small amount of Iodate-Iodide Standard Solution (in large amber bottle).

2. Pour into waste container.

3. Repeat step 1 and 2 two more times

4. Pour 20 ml of the Iodate-Iodide Standard Solution into the rinsed titrating tube.

5. Add 8 drops of Sulfuric Acid (hold the bottle vertical to ensure equal drop size) to the 20 ml of solution and mix by swirling. Then place plastic cap (with hole in it) onto titrating tube.

6. Fill titrating syringe to the "0" mark with Sodium Thiosulfate.

7. Titrate using the Sodium Thiosulfate.

8. When solution turns a pale yellow color, but not clear:
   a) Remove cap, leaving syringe in cap.
   b) Add 8 drops Starch Solution (white bottle). Swirl titration sample gently to mix to a uniform blue color. Recap glass tube and continue titration process.

9. Continue adding Sodium Thiosulfate until solution turns from blue to clear.

10. Read results on syringe - Record your results under the Dissolved Oxygen portion on your field datasheet.

11. If results are less than 9.4 mg/L or greater than 10.0 mg/L, perform a 2nd test and record in the space on datasheet marked "2nd check".

12. Dispose of solution in titrating tube and syringe by pouring down sink and flushing with additional tap water.

13. Keep the amber bottle solution at home- you don't need to take into the field.

DO Analysis Method:

NOTE: Duplicate tests are run simultaneously on each sample to guard against error. If the amount of DO in the second test is more than 0.6 ppm different than the first test, you should do a third test. Record the average of the two closest results.

Since you will be doing two tests at the same time, thoroughly rinse both water sampling bottles with the sample water, filling and dumping the waste water downstream three times before collecting your sample.

1. Using the first sample bottle, submerge about 1/2 of the bottle opening allowing the water to gently flow into the bottle. Try to fill the bottle without causing a lot of bubbles. Submerge the filled bottle.

2. Turn the submerged bottle upright and tap the sides of the bottle to dislodge any air bubbles clinging to the inside of the bottle. Cap the bottle while it is still submerged.

3. Retrieve the bottle and turn it upside down to make sure that no air bubbles are trapped inside. If any air bubbles are present, empty the sample bottle downstream and refill. Fill the second sample bottle. Once two satisfactory samples have been collected, proceed immediately with Steps 4 & 5.

4. Place both sample bottles on a flat surface and uncap. While holding the bottle vertical, add 8 drops of Manganese Sulfate Solution followed by 8 drops of Alkaline Potassium Iodide Solution to each sample bottle. Always add the Manganese Sulfate first. Cap each sample bottle and mix by inverting gently several times. A precipitate will form. Allow the precipitate to settle to the shoulder of the bottle. Mix both bottles again and allow the precipitate to settle to the shoulder again.

5. Add 8 drops of the Sulfuric Acid both sample bottles. Cap the bottles and gently shake to mix, until both the reagent and the precipitate have dissolved. A clear-yellow to brown-orange color will develop. If brown flecks are present, keep mixing the samples until the flecks will not dissolve any further.

NOTE: Following the completion of Step 5, the samples have been "fixed," which means that dissolved oxygen cannot be added to the sample bottles. The titration procedure described in Steps 6-13 may be performed at a later time (but must be performed within 8 hours of sample collection). This means that several samples can be collected and "fixed" in the field and then carried back to a testing station for the remaining steps.

6. Pour 20 ml of the solution from one of the sample bottles into one of the glass tubes with a hole in its cap. Fill to white line so that the bottom of the meniscus (the curved surface of the liquid in the tube) rests on the top of the white line. The amount is critical so be

sure to use the glass dropper to add or remove the sample solution from the tube. Place cap on the tube.

7. Fill syringe (titrator) to the 0 mark with Sodium Thiosulfate solution. Be sure that there are no air bubbles in the syringe. Refer to kit manual for instructions on how to properly fill syringe.

8. To titrate the solution in the tube, insert the syringe into the cap of tube. Add 1 drop of Sodium Thiosulfate to test tube and gently swirl the glass tube to mix. Add another drop of the Sodium Thiosulfate and swirl the tube. Continue this process one drop at a time until the yellow-brown solution in the glass tube turns a pale yellow (lighter than the original yellow-brown solution but not clear). Once you reach this point, take the cap off while leaving the syringe in the cap.

9. Add 8 drops of Starch Solution to the glass tube. Swirl the tube gently to mix. The solution should turn from light yellow to dark blue.

10. Recap the glass tube and continue the titration process with the Sodium Thiosulfate remaining in the syringe (adding one drop at a time and swirling as described in Step 9), until the test tube solution turns from blue to clear. This is the endpoint. If the solution turns blue again, ignore it. Do not add any more Sodium Thiosulfate than is necessary to produce this first color change. Be sure to gently swirl the test tube after each drop.

NOTE: When the dissolved oxygen level is above 10 ppm, the solution in the tube will still be blue when the plunger tip of the titrator reaches 10 units. If it reaches this 10 unit line, do not go beyond that line. Usually, this will only happen when the water temperature is cold. In this case, refill the syringe to the 0 line from the Sodium Thiosulfate bottle and continue adding a drop at a time and swirling until reaching the endpoint.

11. Using the scale on the side of the syringe, read the total number of units of Sodium Thiosulfate used. Each line is 0.2 units. This number equals the number of parts per million (ppm) or milligrams per liter (mg/L) of dissolved oxygen in the water sample.

12. Carry out Steps 7-12 on second sample bottle and second glass tube.

13. Record the results of the two tests on the data sheet. If the difference between Test 1 and Test 2 is more than 0.6 ppm, you should do a third test and record the two results which are within 0.6 ppm.

NOTE: If using transparency tube to measure turbidity, perform this measurement now.

3.7.2 Electronic Probe Method

Equipment: Various models of dissolved oxygen probes and meters

Calibrating Dissolved Oxygen Probes and Meters

With practice and proper care for the DO probe, users can complete the entire DO probe calibration process within 5-10 minutes.

NOTE: Some probes may differ in displaying values. For DO probes, parts per million (ppm), and milligrams per liter (mg/L) are the same value. In addition, barometric pressure may be displayed in millibars (mBar) or in millimeters of mercury (mmHg).

Method:

1. Record the date of calibration. Calibration must be done each day you collect DO samples.

2. Record the temperature of the probe just before you calibrate the probe.

3. Set the barometric pressure (BP) mmHg or mBar- Most probes allow the user to adjust the barometric pressure readout of the probe for calibrating DO. The standard unit for barometric pressure is millimeters of mercury (mmHg) or millibars (mBar). You can get local barometric pressure readings from www.weatherunderground.com or www.noaa.gov. If using weather station data, it is important to adjust the reading by the altitude of the weather station. Appendix II explains how to calculate the correct reading.

4. Calculate the Theoretical DO Value mg/L- Prior to calibrating your probe, you should determine the theoretical DO value to confirm your probes readout. To determine the theoretical value, please follow the instructions found in Appendix II.

5. Record the mg/L reading of the calibrated DO level. If everything is working properly, the probe should display the correct DO level based on the altitude and temperature that you are calibrating at. The theoretical DO value and the probes calibrated readout should be within 0.2 mg/L. If not, try to recalibrate the probe or perform maintenance on the probe based on manufacturer instructions.

6. Turn off the probe if the manufacturer says so. If not, keep the probe on at all times while you are taking it out to the field and performing your field samples.

Measure DO

Depth Profile Sampling (>3m)
If you are measuring with a multi probe and have a long enough cord, you are encouraged to do depth profiles.

1. Measure 1.0 m above the bottom, then move your sensor up to the next whole integer depth, then proceed at 1 meter intervals up through the water column until you reach 1 m below the surface if sampling in Virginia, take an additional measurement at 0.5 m below the surface if sampling in Maryland. (Example: At a 3.4 m deep site, measure at 2.4, 2.0, 1.0 m and take an additional measurement at 0.5 m if in Maryland)

2. At each iteration allow the probe to stabilize before recording your DO reading at the corresponding depth

3. Measure salinity and temperature at each depth as well

4. Record depth, DO, temperature, and salinity on your data sheet for each depth

Depth Profile Sampling (≤3 m)

1. Measure 1.0 m above the bottom, allow the probe to stabilize and record your result

2. Measure 0.5 m below the surface if sampling in Maryland and 1 m below the surface if measuring in Virginia, allow the probe to stabilize and record your result

Surface Sampling

1. Place your probe 0.5 m beneath the surface of the water if sampling in Maryland and 1 m beneath the surface if sampling in Virginia.

2. Wait for the probe to stabilize, and then record your reading

Post Sampling Calibration Check

After the sample run is complete, return the probe to the calibration station to perform a quick post check. The post check consists of placing the probe in the DO calibration chamber and letting it equalize. This may take between 2 to 10 minutes depending on the condition of the probe.

1. Measure and record the temperature. If you did the morning calibration indoors, the probe temperature should be roughly close to the same as the morning calibration. If you are calibrating the probe outside, the temperature may be different from the earlier reading. This should not affect the post check.

2. Record the barometric pressure reading of the probe. This may have changed from the morning reading due to weather changes. You can get current local barometric pressure readings from the Internet. Remember to adjust any weather station data based on the instructions found in Appendix II.

3. As in the morning calibration, use Appendix II to determine your theoretical DO level.

4. Record the DO reading of the probe (ppm or mg/L). DO NOT recalibrate the probe. The purpose of this check is to see if the probe has drifted out of acceptable limits during the day.

5. Calculate the difference between the probe reported value and the theoretical DO value. If the probe is functioning properly there should be a difference of less than 0.50 mg/L from the afternoon theoretical DO level and the probe readout. If the calibration

difference is greater than 0.50 mg/L the probe needs service and you must flag the data because the probe did not hold onto the calibration. If the calibration difference is 0.16 to 0.50 mg/L. The calibration of the probe is approaching the limits of accuracy and preventative maintenance may be required. It may be wise to clean the probe or replace the probe membrane when this occurs.

3.8 pH

3.8.1 Electronic probe method

Equipment: Various models of pH probes and meters

Calibration

The pH probe calibration procedure a similar protocol used in calibrating the DO probe. Most meters allow calibrating the pH probe using two different buffers. In most cases the use the 7.00 and 4.00 pH buffer solutions is suitable. If you are experiencing pH values above 7.00, calibrate using 7.00 and 10.00 buffer.

Use fresh buffer solution when you calibrate the probe and check the readings at the end of the day. If the probe is capable in doing so, please record the probe readings to the nearest hundredth unit place (Ex. 7.01) when performing the calibration.

1. Record the date of calibration. Calibration must be done each day you perform samples.

2. Record the temperature of the probe during calibration.

3. Record the probe reading as you place the probe in the 7.00 buffer solution. Gently swirl the buffer or the probe to obtain an accurate reading.

4. Calibrate the probe, the probe should now read a value close to 7.00 pH units. Most manufacturers of buffers provide a table showing the pH result that probes should display based on temperature. Check against this value displayed on the probe is close to this value.

5. Clean the probe with distilled or deionized water and blot dry

6. Immerse the probe in the 4.00 (or 10.00) buffer solution, record the stabilized value.

7. Calibrate the probe and it should now read a value close to 4 (or 10) pH units. Again, consult the buffer solution table to ensure accuracy.

After calibration, you may turn off the probe if the manufacturer says so. If not, the probe should be kept on at all times while going out into the field and prior to the post check. Follow manufacturer instructions regarding transporting of the probe into the field to prevent damage and drying out of the pH probe.

Field Sampling with Multiprobe

IMPORTANT NOTE- When traveling to a sample station, keep the probe tip stored in the protective cap. This will keep the glass sensor hydrated.

1. Turn the probe on.

2. Set the probe 0.5 m beneath the surface is sampling in Maryland and 1 m beneath the surface in Virginia. Let the reading stabilize. This may take a little longer than the other probes.

3. Once the reading has stabilized record the reading on your datasheet.

4. Mark the depth that you took your sample on your data sheet.

5. Turn off the probe and replace the protective cap.

Field Sampling with Individual pH probe

IMPORTANT NOTE- When traveling to a sample station, keep the probe tip stored in the protective cap. This will keep the glass sensor hydrated.

1. Turn the probe on.

2. Set the probe 0.5 m beneath the surface of the water if sampling in Maryland and 1 m beneath the surface if sampling in Virginia. Let the reading stabilize. This may take a little longer than the other probes.

3. If measuring in a bucket, gently swirl the water with the probe and measure just beneath the surface.

4. Once the reading has stabilized record the reading on your datasheet.

5. Mark the depth that you took your sample on your data sheet.

6. Turn off the probe and replace the protective cap.

End of Day Calibration Check

To ensure the probe has maintained proper calibration, it is important to verify no significant probe drift has occurred. The procedures listed below will verify the probe did not drift outside QA/QC specifications. DO NOT CALIBRATE the probe during this check. Doing so will invalidate the data collected during the sample run.

1. Rinse off the probe and probe tip with distilled water and wipe dry using a soft cloth. Washing the probe will remove any material that may reduce probe life.

2. Place the probe into a container of pH 7.00 buffer. You may use the same buffer used during the morning calibration as long as the buffer was covered and appears clean.

3. Allow the probe to stabilize and record the temperature and pH reading in the "End of Day Temp C" and the "End of Day pH 7 Check" columns on the "pH Probe Calibration Form."

4. Rinse the probe and repeat the end of day check process using the 4.00 or 10.00 buffer.

If both buffer checks are within 0.20 units from the calibration values, the probe is within specifications. If the readings are greater than 0.20 units, flag all pH data collected during the sample run by typing "pH probe flag" in the "Additional comments" section when entering data into the online database. Also note "pH probe flag" at the top of the hard copy datasheet. This is because sometime during the sample run, the probe exceeded QA/QC specifications.

3.8.2 Colorimetric Kit

Equipment: LaMotte or Hach pH kits

Method:
Look on the front of black box to determine whether you have a wide range pH kit or a narrow range pH kit (i.e. cresol red, phenol red, bromthymol blue, thymol blue).

1. Rinse one sample test tube and cap twice with water from the stream or bucket.

2. Fill the sample test tube to the black line with water from the stream or bucket. The bottom of the meniscus should be even with the line. Use plastic dropper to add or remove water from test tube.

3. For wide range pH kit, add ten drops of the wide range indicator while holding the reagent bottle completely upside down. For narrow range kits, add 8 drops of the indicator while holding the reagent bottle completely upside down.

4. Cap the test tube and mix the sample thoroughly.

5. Slide the tube in the comparator slot, hold it up to the sunlight, and record the pH value from the color in the comparator that most closely matches the sample tube color. When the color observed is between 2 colors on the comparator, the value is reported to the nearest 0.5 unit (for wide range kit) or 0.1 unit for other pH kits.

3.9 Salinity, Conductivity, and Total Dissolved Solids

3.9.1 Salinity Measurement with a Refractometer

Equipment: Salinity refractometer

The refractometer must be calibrated before taking salinity measurement.

Calibration:

1. Check the refractometer with distilled water. If it does not read 0 o/oo, you must calibrate the instrument. DO NOT PERFORM CALIBRATION IN THE FIELD. Calibration must take place in controlled environment at approximately 20 oC (room temperature) using distilled water of the same temperature.

2. Lift the cleat plate and add 1-2 drops of distilled water to the oval blue prism. Hold the prism at an angle close to parallel so the water drops will not run off.

3. Close the plate gently. The water drops should spread and cover the entire prism. Repeat the process if there are any gaps or if the sample is only on one portion of the prism.

4. Look through the eyepiece. If the scale is not in focus, adjust it by turning the eyepiece either clockwise or counterclockwise.

5. The reading is taken at the point where the boundary line of the blue and white fields crosses the scale.

6. If the reading is not at "0" turn the calibration screw with the included screwdriver while looking through the eyepiece until the boundary line falls on "0."

7. When the measurement is complete, the sample must be cleaned using tissue paper and distilled water.

NOTE: The refractometer needs to be at the same approximate temperature as the sample water. If the refractometer has been sitting in an air-conditioned environment prior to sampling, allow it to warm to the outside air temperature.

Method:

1. Rinse the refractometer with water sample.

2. Then apply drops of your water sample onto the refractometer and hold up to light to read salinity (right side of circle).

3. Record as parts per thousand (o/oo) using the scale located on the right hand side of

refractometer view scope.

3.9.2 Salinity, Conductivity, and TDS Probe

Equipment: Various models of conductivity probes and meters

Calibration
Most probes that test for conductivity and TDS use a pre-made calibration solution with a specific conductivity value. The probe is immersed in the solution and calibrated to the value of the solution. It is good to use a calibration solution concentration similar to what you may find in the field to ensure accuracy.

1. Record the date of calibration. Calibration must be done each day you perform samples.

2. Record the temperature of the water read by the probe while you are calibrating the probe.

3. Write down the conductivity listed on the probe when you immerse the probe into the conductivity solution and record the value prior to calibration.

4. Record the conductivity solution that you will use to calibrate the probe. The standard unit for these solutions is in microsiemens per centimeter (mS/cm) but probes may use different units.

5. Write down the conductivity reading after you have calibrated the probe in the solution. The probe should be very close to the calibrated buffer solution but may be off by a couple of units.

Measure salinity, conductivity & TDS

Depth Profile Sampling (>3m)
If you are measuring with a multi probe and have a long enough cord, you are encouraged to do depth profiles.

1. Measure 0.5 m above the bottom, then move your sensor up to the next whole integer depth, then proceed at 1 meter intervals up through the water column until you reach 1 m below the surface if sampling in Virginia, take an additional measurement at 0.5 m below the surface if sampling in Maryland. (Example: At a 3.4 m deep site, measure at 2.4, 2.0, 1.0 m and take an additional measurement at 0.5 m if in Maryland)

2. At each iteration allow the probe to stabilize before recording your salinity reading at the corresponding depth.

3. Measure DO and temperature at each depth as well.

4. Record depth, DO, temperature, and salinity on your data sheet for each depth.

Depth Profile Sampling (≤3 m)

1. Measure 1.0 m above the bottom, allow the probe to stabilize and record your result.

2. Measure 0.5 m below the surface if sampling in Maryland and 1.0 m below the surface if sampling in Virginia, allow the probe to stabilize and record your result.

Surface Sampling with Multiprobe

1. Prior to sampling, rinse the probe with deionized or distilled water.

2. Select the appropriate mode and range on the meter, beginning with the highest range and working down. Some probes will auto select the correct range.

3. Place the probe 0.5 m beneath the surface if sampling in Maryland and 1 m beneath the surface if sampling in Virginia, and read the salinity, conductivity or TDS of the water sample on the meter's scale.

4. Record the depth at which you took your sample on your data sheet.

NOTE: If your probe does not automatically select the appropriate measurement range, and the reading is in the lower 10 percent of the range that you selected, switch to the next lower range. If the reading is above 10 percent on the scale, then record this number on your data sheet.

5. Rinse the probe with distilled or deionized water between each sample and before post sampling calibration check. Replace the cap for storage and transport.

Surface Sampling with individual Salinity/TDS probe

1. Prior to sampling, rinse the probe with deionized or distilled water.

2. Select the appropriate mode and range on the meter, beginning with the highest range and working down. Some probes will auto select the correct range.

3. Place the probe 0.5 m beneath the surface if sampling in Maryland and 1 m beneath the surface if sampling in Virginia and read the salinity, conductivity or TDS of the water sample on the meter's scale.

4. Record the depth at which you took your sample on your data sheet.

NOTE: If your probe does not automatically select the appropriate measurement range, and the reading is in the lower 10 percent of the range that you selected, switch to the next lower range. If the reading is above 10 percent on the scale, then record this number on your data sheet.

5. Rinse the probe with distilled or deionized water between each sample and before post sampling calibration check. Replace the cap for storage and transport.

Post sampling calibration check

1. Record the temperature of the probe at the end of the day when you are performing the calibration check.

2. Write down the conductivity listed on the probe when you immerse the probe into the conductivity solution and record the value.

3. Calculate the difference between the pre and post sampling calibration values.

4. Standard rule of thumb is if the probe difference is less than 10.00%, you should be confident of the probe values. To calculate the relative percent difference use the formula:

$$RPD\% = \frac{AbsoluteValue(Sample1 - Sample2)}{Average(Sample1 + Sample2)} \times 100\%$$

5. Initial the person calibrating and using the probe for your records. This is good to know in case something happens to the probe that you may not be aware of due to someone else is using it.

3.10 Nitrate – Nitrogen and Orthophosphate Kits

Equipment:

- Nitrate – Nitrogen kit w/ all chemicals and clean glassware (Hach NI-14 14161000, LaMotte, 3110, LaMotte 3354)
- Orthophosphate kit w/ all chemicals and clean glassware (Hach PO-19 224800, Hanna HI 38061, Hanna HI 713)
- Clean polypropylene sample bottle or scintillation vial (60 ml)

Method:

1. Rinse the sample bottle with sample water and dispose of downstream.

2. Repeat step 1 three times.

3. Fill the bottle with sample water from about 0.3 m beneath the surface and cap. Process the sample as soon as possible.

4. Make sure the sample is well mixed prior to analysis by shaking the sample bottle.

5. Follow the protocol for each nutrient type as outlined in the instructions accompanying the kit. Reagents should be maintained at about 20° C to yield best results.

6. Record your results on the data sheet.

3.11 Phosphate

Equipment:

- Hanna HI 713 Phosphate Low Range Checker
- Clean polypropylene sample bottle or scintillation vial (60 ml)

Method:

1. Rinse the sample bottle with sample water and dispose of downstream three times.

2. Fill the bottle with sample water from about 0.3 m beneath the surface and cap. Process the sample as soon as possible.

3. Make sure the sample is well mixed prior to analysis by shaking the sample bottle.

4. Turn the meter on by pressing the button. All segments will be displayed. When the display shows “Add”, “C.1” with “Press” blinking, the meter is ready.

5. Fill the cuvette with 10 mL of unreacted sample and replace the cap. Place the cuvette into the meter and close the meter’s cap.

6. Press the button. When the display shows “Add”, “C.2” with “Press” blinking the meter is zeroed.

7. Remove the cuvette from the meter and unscrew the cap. Add the content of one packet of HI 713-25 reagent. Replace the cap and shake gently for 2 minutes until the powder is completely dissolved. Place the cuvette back into the meter.

8. Press and hold the button until the timer is displayed on the LCD (the display will show the countdown prior to the measurement) or, alternatively, wait for 3 minutes and press the button.

9. The instrument directly displays the concentration of phosphate in ppm. The meter automatically turns off after 2 minutes.

10. Record your results on your datasheet.

4 Lab sample collection preparation and handling

4.1 Bacteria

Sample collection:

Note the amount of rainfall within 48 hours prior to sampling and record in the bacteria section of the datasheet.

Collecting by wading:

1. Wade into the main flow of the stream.
2. Take a few steps upstream with minimal disturbance;
3. Un-cap the sterile and pre-labeled bottle without touching the inside of the lid.
4. Using a U motion dip the bottle into the water down to approximately 0.3 m down and away from yourself allowing the bottle to fill ¾ full.
5. Cap the bottle and place sample on ice in cooler immediately (cooler temperature should be 1°C to 4°C). NOTE: Do not freeze your sample.

Collecting using a bucket:

1. Make sure not to touch inside of bucket with your hands.
2. If sampling from a dock or pier, go as far as possible to the end of the pier to collect your sample.
3. Throw the bucket out as far as possible in the main channel, and try not to disturb the bottom.
4. Rinse the bucket three times with stream water collected downstream of your sampling location.
5. Fill the bucket with the sample water to 1/3 full.
6. Un-cap the sterile and pre-labeled bottle without touching the inside of the lid.
7. Using a U motion dip the bottle into the water down and away from yourself allowing the bottle to fill ¾ full.
8. Cap the bottle and place sample on ice in cooler immediately (cooler temperature should be 1°C to 4°C). NOTE: Do not freeze your sample.

Collecting using a sampling pole (from boat or dock):

If sampling from a boat make sure that the boat motor has not stirred up the water. If the water is shallow, sampling should be done through wading.

1. Un-cap your sterile and pre-labeled bottle and secure it to the end of the pole.

2. Extend the pole outward and dip at approximately 0.5 m below the surface if sampling in Maryland and 1.0 m below the surface if sampling in Virginia.

3. Cap the bottle and place sample on ice in cooler immediately (cooler temperature should be 1°C to 4°C). NOTE: Do not freeze your sample.

After sampling bacteria wash your skin that came in contact with the water with disinfectant or soap to reduce your chances of becoming sick.

4.2 Chlorophyll A

Example field supplies:

- 500-mL polypropylene (PP) sample bottles
- 50-mL syringes
- filter bodies with filter caps
- 25-mm 0.7-µm porosity GF/F filter membranes
- Handheld vacuum pump
- Opaque towels
- Aluminum foil
- Filter forceps

Before going out to collect samples, prepare equipment and supplies according to the recommended sampling procedure of the laboratory where the samples will be analyzed. This can include syringe filtering or a handheld vacuum pump and filters.

Method:

1. Using the sampling pole, rinse the 500-mL labeled site specific bottle and syringe three times.

2. Drain the bottle until it is empty, put the cap on, lower it one forearm's length under water (about 0.3 meters) then remove the cap. Wait for the bottle to fill, then cap it and return it to the surface.

3. Follow the recommended filtering procedure by the analytical laboratory where the samples will be analyzed. Color on the filter generally indicates a sufficient sample for analysis.

4. Record the volume of water pushed through the filter on the data collection sheet.

5. Store samples in cooler. Samples must be kept cool and out of sunlight for the duration of field sampling.

6. Cap 500-mL bottle retaining sampled water and store in dark location to bring back to lab. This sample will serve as a back-up sample should there be a filter problem.

Laboratory preparation:
Following the procedures laid out by the analytical lab that will process the samples is important. Here are a few general steps:

1. Prepare pieces of aluminum foil.

2. Fold in half again, then unfold, creating a crease.

3. Create labels using labeling tape noting site number, date, and volume pressed through

filter.

4. Place filter in aluminum foil with the center of the filter centered on the crease, with side containing the intercept chlorophyll up (should have slight color to it). Folding foil and gently assisting with forceps if necessary by pressing on filter fold the filter in half.

5. Double over edges of fold, displacing air and create a little pocket in which the folded filter is located.

6. Repeat for all samples.

7. Label foil packets.

8. Place foil packets in locking plastic bag and then double bag with another locking plastic bag.

9. Place in freezer to await shipment to the analytical laboratory.

10. Rinse all filter holders and 500-mL bottles with tap water and allow to air dry.

NOTE: It is critical that the chlorophyll water samples and foil packets remain dry. The samples in foil should be double bagged and packed with ice in portable Styrofoam transport coolers with surrounding cardboard box. Samples should be mailed overnight to arrive at the analytical laboratory as soon as possible. If properly packaged and frozen (sampled filters should be stored frozen, at least -20˚C, in the dark), chlorophyll a samples can be stored for up to three and a half weeks. The package should also be marked to indicate “chlorophyll samples” as contents.

4.3 Nutrient and Grab Samples

Collecting from a boat:

1. Facing upstream, extend the pole and bottle, rinse the bottle out three times, and take the sample the fourth time from a depth of 0.3 m beneath the surface.

2. After samples are taken, immediately place the sample on ice up to the shoulders of the bottle. The lid should not be immersed under the ice, in case ice water leaks into the sample bottle, diluting the concentration of the sample.

3. On the field data sheet, record the time, date, and any other information about the water sampling event.

Collecting by wading:

1. Wade into the main flow of the stream.

2. Take a few steps upstream with care not to disturb the sediment;

3. Un-cap the pre-labeled bottle.

4. Using a U motion dip the bottle into the water approximately 0.3 m down and away from yourself allowing the bottle to fill to the shoulder.

5. After samples are taken, immediately place the sample on ice up to the shoulders of the bottle. The lid should not be immersed under the ice, in case ice water leaks into the sample bottle, diluting the concentration of the sample.

Collecting with a sampling pole from the shore or a dock:

1. Attach the sample bottle to the sampling pole, making sure that the clamp is tight.

2. The sampling point in the stream or river should have a low to medium flow and not be in eddies or stagnant water.

3. Facing upstream, extend the pole and bottle, rinse the bottle out three times, and take the sample the fourth time from approximately 0. 5 m beneath the surface if sampling in Maryland and 1.0 m beneath the surface if sampling in Virginia.

4. Fill the bottle up to the shoulders and immediately cap and place on ice. The lid should not be immersed under the ice, in case ice water leaks into the sample bottle, diluting the concentration of the sample.

4.4 Chemical preservatives and reagents

The nutrient sample bottles contain a small amount of sulfuric acid as a preservative. When sampling it is important to fill the bottle to the needed level and not pour out the preservative or excess sample from the bottle.

The bacteria sample bottle contains a dechlorinating tablet. When collecting non-chlorinated water, discard the tablet. Samplers should discard the tablet just prior to collecting a bacteria sample at the site. Discard the tablet by dumping out of the bottle without touching the lip or inner wall of the sample bottle. The tablets are harmless to the environment and may be left at the site.

4.5 Sample container handling and preservation

Proper sample containers and sample preservation are essential to sample integrity. Samples not preserved properly may be rejected by the laboratory.

a) Sample containers should be inspected and any torn, punctured or cracked sample containers discarded.

b) After collecting the sample, make sure the lids are secured tightly to prevent contamination from water seepage in or out of the container.

c) Sample containers and coolers should be stored with the tops securely fastened. Containers with loose fasteners should be replaced or taped to prevent loss of sample containers during transport.

d) In the field, unless specified otherwise, all samples should be placed in an ice filled cooler immediately after collection. To ensure samples do not exceed the 4°C holding temperature, sample containers shall be placed upright and if possible, covered with ice in such a manner that the container openings are above the level of ice. Bacteria sample bottles should be stored in bags, placed in coolers and surrounded with wet ice.

e) Glass sample containers should be packed in bubble wrap or other waterproof protective materials to minimize accidental breakage.

f) The laboratory will provide temperature bottles that they use to determine sample temperature upon arrival at the lab. Make sure that every cooler used to ship samples to the lab contains one of these bottles.

g) Store sample containers in a clean area away from fumes, smoke, gasoline containers, etc. A trunk of car or a garage may not be appropriate.

4.6 Sample Bottle Identification

Each sample container must include a label with the following information:

a) Station ID or description

b) Date and time of sample collection

c) Collector's initials

d) Sample depth in meters (surface samples are reported as 0.3 m)

e) Parameter name and/or group code

f) Container number

g) Preservative used and volume filtered, if applicable.

Samples will not be analyzed if this information is missing. If more than one container is needed for a parameter (such as a duplicate sample), each container collected for that parameter must have a label with identical information in addition to an indication of 1 of 3, 2 of 3, 3 of 3, etc., as required. Split samples should be designated as S1 and S2.

Please remember to fill out the labels on the bottle with a waterproof pen before taking the samples.

It is essential that the actual sampling site match the labeling information. Always check the labeling information against the actual site. Samples not labeled properly may be rejected by the laboratory.

4.7 Transport of Samples

After collecting the samples at the site:

1. Place the bottles in the cooler filled with ice. Coolers should have enough ice to come up to the necks of the sample bottles.

2. Place any chain of custody forms in the Ziploc bag taped to the inner lid of the cooler.

3. Transport the cooler with samples to the designated drop off point or laboratory by the specified time.

5 Lab Procedures

Labs should be either NELAP, state, federal certified, or recognized by the CBP. Labs that do not maintain a certification or are not recognized by the CBP will be considered for inclusion on a case by case basis. The following are the approved methods and their corresponding SOPs for reference for laboratories. It is expected that laboratories will be in compliance with these methods and will already be in possession of the procedural documentation for these methods.

Parameter	Method	Appendix
Bacteria - Enterococcus	US EPA method 1600	Appendix III
Bacteria - Enterococcus	ASTM Method #D6503-99	See manufacturer's manual
Chlorophyll & Pheophytin	CBP IV-12.0	Appendix IV
Chlorophyll & Pheophytin	US EPA method 446.0	Appendix V
Chlorophyll & Pheophytin	US EPA method 445.0	Appendix VI
Silicate	US EPA method 366.0	Appendix VII
Nitrate - Nitrogen	US EPA Method 353.2	Appendix VIII
Nitrite - Nitrate	US EPA Method 353.2	Appendix VIII
Ammonia - Nitrogen	US EPA Method 350.1	Appendix IX
Orthophosphate	US EPA Method 365.5 or 365.1	Appendix X
Total Nitrogen	Standard Methods 4500-N C-2011 or 4500-P J-2011	Appendix XI
Total Phosphorus	EPA Method 365.1 or Standard Methods 4500-P J-2011	Appendix XII

Laboratories will perform QA/QC measures including: method blanks, matrix spikes, replicates, check standard.

6 Cleanup and Storage of Water Monitoring Equipment

a) Rinse the thermometer in tap water and store upright.

b) Pour contents of DO sampling bottles and chemical kits into the sink. Rinse all the bottles and containers thoroughly with tap water. Put all equipment away until next sampling time.

c) Store all chemical reagents in a dark, cool place and out of the reach of children and pets!

d) Save expired chemicals and give them to your monitoring coordinator or trainer at the next recertification event for proper disposal.

NOTE: If you conduct the analytical procedures away from home or on a boat, you need a special container for safe disposal of the test samples. A plastic milk jug or jar works well and is easy to obtain. Fill this container about ½ to ¾ full with kitty litter to absorb the moisture. When the litter is saturated, place the closed jar in double plastic garbage bags and dispose of in the trash.

6.1 Maintenance for pH meter

Follow maintenance and care guidelines as specified by the manufacturer manual. Below are some general day to day care tips.

1. Ensure the probe is cleaned and well maintained. After each sample run, rinse off the probe with distilled water. Use a soft cloth and gently dry the probe and glass sensor.

2. Store the probe tip in the cap provided by the manufacturer. Inside this cap, place a small cotton ball or piece of paper towel soaked with pH 4.00 buffer (or probe storage solution). This will keep the probe in working condition until the next field sampling event.

3. If you see any biological growth (mold, algae, etc.), use mild soap or warm (~30o C) pH 4.00 buffer to clean. Rinse with distilled water and dry.

4. If the calibration or end of day check indicates there is a problem with the probe, and standard cleaning does not produce acceptable results, replacement of the sensor cap may be necessary. Contact a Project Team Member to get a replacement sensor cap.

Appendix I

Field Data Sheet

Appendix II

Theoretical DO Calculation

How to Calculate Theoretical Dissolved Oxygen Values

From: Virginia Citizen Water Quality Monitoring Program Methods Manual - October 2007

Proper calibration of Dissolved Oxygen (DO) probes is important to collect accurate data. An easy way to see if a probe is calibrated correctly is to compare the probe's results against a theoretical DO value. This value is what the DO level should be based on temperature and barometric pressure.

DO Level based on temperature

The top table on the attached chart allows users to find the DO level based on temperature. The top and side axis of the table corresponds to the temperature that the probe is reporting. The intersection of the two axes displays the DO reading. Write this number down to start calculating the theoretical DO level.

Correction factor for barometric pressure

Barometric pressure is a way to tell how much atmosphere is pressing down on a surface. Weather systems and elevation above (or below) sea level can change this value. The bottom table of the attached chart will help compensate for these changes in pressure. Dissolved oxygen probes normally show pressure in millimeters of mercury (**mmHg**) or millibars (**mBar**).

Having a barometer on hand is a good way to get pressure data. A weather station can also provide pressure data. Websites such as www.weatherunderground.com are useful to find local weather stations. Please note that most barometers and weather stations report pressure in inches of mercury (**inHg**).

Note about using weather station pressure readings

Weather stations compensate pressure readings to make it appear as if the station is at sea level. To account for this, subtract the barometric pressure by 1.01 inHg per 1,000 feet in elevation of the weather station. This final value is known as absolute barometric pressure.

Example: Find the absolute barometric pressure of a station located 222 feet above sea level that reported 30.12 inHg.

$$30.12 \text{ inHg} - \frac{1.01 \text{ inHg}}{1000/ 222 \text{ feet}} \rightarrow 30.12 - \frac{1.01}{4.50} \rightarrow 30.12 - 0.22 == 29.90 \text{ inHg absolute barometric pressure}$$

Once finding the absolute pressure, use the bottom table found on the attached chart to find the proper correction factor to use. The formulas at the bottom of the chart will help in converting inHg barometric pressure readings into **millibars** (mBar) or **millimeters of mercury** (mmHg) that are commonly used to calibrate a dissolved oxygen probe. Use this value to find the correction factor to use in the final calculation.

Example: A barometric pressure of 970 millibars you would use a correction factor of 0.96 (second column, bottom row).

Theoretical DO Calculation

To find the theoretical DO value, use the following formula.

Theoretical DO = (DO level based on temperature) x (barometric pressure correction factor)

Example: If a probe had a temperature of 18.4 C and the barometric pressure was 970 mBar, the theoretical DO value would be 9.00 mg/L (9.37mg/L x 0.96 correction factor).

Dissolved Oxygen Saturation

Directions- To determine theoretical DO saturation, multiply the O2 concentration value (found in the top chart) by the barometric pressure correction factor (bottom chart).

Example: Find the DO saturation for at a temperature of **18.4 C** at **730 mmHg** pressure: 9.37 x 0.96**= 9.00 mg/L**

Temp in ^{O}C	O_2 concentrations in mg/l									
	0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
5	12.75	12.71	12.68	12.65	12.61	12.58	12.55	12.52	12.48	12.45
6	12.42	12.39	12.36	12.32	12.29	12.26	12.23	12.2	12.17	12.14
7	12.11	12.08	12.05	12.02	11.99	11.96	11.93	11.9	11.87	11.84
8	11.81	11.78	11.758	11.72	11.69	11.67	11.64	11.61	11.58	11.55
9	11.53	11.5	11.47	11.44	11.42	11.39	11.36	11.33	11.31	11.28
10	11.25	11.23	11.2	11.18	11.15	11.12	11.1	11.07	11.05	11.02
11	10.99	10.97	10.94	10.92	10.89	10.87	10.84	10.82	10.79	10.77
12	10.75	10.72	10.7	10.67	10.65	10.63	10.6	10.58	10.55	10.53
13	10.51	10.48	10.46	10.44	10.41	10.39	10.37	10.35	10.32	10.3
14	10.28	10.26	10.23	10.21	10.19	10.17	10.15	10.12	10.1	10.08
15	10.06	10.04	10.02	9.99	9.97	9.95	9.93	9.91	9.89	9.87
16	9.85	9.83	9.81	9.79	9.76	9.74	9.72	9.7	9.68	9.66
17	9.64	9.62	9.6	9.58	9.56	9.54	9.53	9.51	9.49	9.47
18	9.45	9.43	9.41	9.39	9.37	9.35	9.33	9.31	9.3	9.28
19	9.26	9.24	9.22	9.2	9.19	9.17	9.15	9.13	9.11	9.09
20	9.08	9.06	9.04	9.02	9.01	8.99	8.97	8.95	8.94	8.92
21	8.9	8.88	8.87	8.85	8.83	8.82	8.8	8.78	8.76	8.75
22	8.73	8.71	8.7	8.68	8.66	8.65	8.63	8.62	8.6	8.58
23	8.57	8.55	8.53	8.52	8.5	8.49	8.47	8.46	8.44	8.42
24	8.41	8.39	8.38	8.36	8.35	8.33	8.32	8.3	8.28	8.27
25	8.25	8.24	8.22	8.21	8.19	8.18	8.16	8.15	8.14	8.12
26	8.11	8.09	8.08	8.06	8.05	8.03	8.02	8	7.99	7.98
27	7.96	7.95	7.93	7.92	7.9	7.89	7.88	7.86	7.85	7.83
28	7.82	7.81	7.79	7.78	7.77	7.75	7.74	7.73	7.71	7.7
29	7.69	7.67	7.66	7.65	7.63	7.62	7.61	7.59	7.58	7.57
30	7.55	7.54	7.53	7.51	7.5	7.49	7.48	7.46	7.45	7.44

Barometric Pressure Correction factor:

mmHg (mBar)	Corr. Factor	mmHg (mBar)	Corr. Factor	mmHg (mBar)	Corr. Factor	mmHg (mBar)	Corr. Factor
775-771 (1033-1028)	1.02	750-746 (1000-995)	0.987	725-721 (967-961)	0.953	700-696 (934-928)	0.92
770-766 (1027-1021)	1.014	745-741 (994-988)	0.98	720-716 (960-955)	0.947	695-691 (927-921)	0.914
765-761 (1020-1014)	1.007	740-736 (987-981)	0.973	715-711 (954-948)	0.94	690-686 (920-915)	0.907
760-756	1	735-731	0.967	710-706	0.934	685-681	0.9

(1013-1008)		(980-975)		(947-941)		(914-908)	
755-751 (1007-1001)	0.993	730-726 (974-968)	0.96	705-701 (940-935)	0.927	680-676 (907-901)	0.893

Appendix III

Laboratory Method – Bacteria – Enterococcus

Appendix IV

Laboratory Method – Chlorophyll & Pheophytin CBP IV-12.0

Appendix V

Laboratory Method – Chlorophyll & Pheophytin – US EPA 446.0

Appendix VI

Laboratory Method – Chlorophyll & Pheophytin – US EPA 445.0

Appendix VII

Laboratory Method – Silicate – US EPA 366.0

Appendix VIII

Laboratory Method – Nitrate - Nitrite - Nitrogen – US EPA 353.2

Appendix IX

Laboratory Method – Ammonia – Nitrogen US EPA 350.1

Appendix X
Laboratory Method – Orthophosphate
US EPA 365.5 and US EPA 365.1

Appendix XI

Laboratory Method – Total Nitrogen –
Standard Methods 4500-N C-2011 and 4500-P J-2011

Appendix XII

Laboratory Method – Total Phosphorus

EPA Method 365.1 and Standard Methods 4500-P J-2011

Tidal Monitoring Quality Assurance Project Plan
Alliance for the Chesapeake Bay
CB96334901 for Citizen-Based/Non-Traditional Monitoring Grant