Washington State Department of Ecology

Environmental Assessment Program

Standard Operating Procedures for the Collection, Processing, and Analysis of Stream Samples

Version 1.4

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Although Ecology follows the SOP in most instances, there may be instances in which Ecology uses an alternative methodology, procedure, or process.
### SOP Revision History

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Environmental Assessment Program

Standard Operating Procedure for the Collection and Processing of Stream Samples

1.0 Purpose and Scope

1.1 This document is the Environmental Assessment Program (EAP), Standard Operating Procedure (SOP) used to collect, preserve, measure, and analyze water quality at Freshwater Ambient Monitoring stations.

1.2 It describes the general stream monitoring procedures used for run preparation, sample collection, measurement, processing, preservation, and shipment. The document also addresses quality assurance and quality control procedures.

1.3 The standard set of samples collected, measured, or processed include: temperature, pH, conductivity, dissolved oxygen, turbidity, total suspended solids, fecal coliform bacteria, ammonia, nitrate plus nitrite, total nitrogen, total phosphorus, soluble reactive phosphorus, metals, and stage height.

1.4 Other samples that may also be collected and processed on a special study request basis include: alkalinity, dissolved organic carbon (DOC), total organic carbon (TOC), filtered total phosphorus, filtered total nitrogen, chlorophyll, and suspended sediment concentration (SSC).

1.5 All Ambient stations are typically monitored once a month and dissolved metals are also monitored every other month at only a few stations.

2.0 Applicability

2.1 This SOP is intended for long term ambient stream monitoring.

3.0 Definitions

3.1 Dissolved Oxygen (DO) – The concentration of dissolved oxygen (mg/L) in a water sample.

3.2 Conductivity – A measure of the ability of water to carry an electrical current. It is dependent upon the concentrations and types of dissolved ions and the water temperature. In general, a greater concentration of ions in the water will lead to a larger conductivity value.

3.4 EAP – Environmental Assessment Program.

3.5 EIM – Environmental Information Management System. A searchable database developed and maintained by the Washington State Department of Ecology.

3.6 Fecal coliform – A group of bacteria that inhabit the intestinal tract of warm-blooded animals and remain viable in freshwater for a variable period of time. The presence of fecal coliform bacteria in water indicates fecal contamination of the water by a warm-blooded animal; harmful bacteria and viruses associated with fecal contamination may also be present.

3.7 FEP – fluorinated ethylene propylene

3.8 Field Logbook – A weather resistant logbook containing “Rite in the Rain” ® writing paper used to document any and all field activities, sample data, methods and observations for each and all sample sites.

3.9 μmhos – micro mhos (mho = 1/ohm = 1 Siemen) per centimeter

3.10 MEL – Manchester Environmental Laboratory

3.11 MQO’s – Measurement Quality Objectives

3.12 MSDS – Material Safety Data Sheets provides both workers and emergency personnel with the proper procedures for handling or working with a particular substance. MSDS’s include information such as physical data (melting point, boiling point, flash point, etc.), toxicity, health effects, first aid, reactivity, storage, disposal, protective equipment and spill/leak procedures.

3.13 OC – Operations Center. The location of the program field equipment, boats, walk-in cooler and shop (where technicians repair or fabricate the equipment).

3.14 pH – A measure of the acidity or alkalinity of a solution, numerically equal to 7 for neutral solutions, increasing with increasing alkalinity and decreasing with increasing acidity. The pH scale ranges from 0 to 14.

3.15 Run – Monthly scheduled sampling event (usually lasting 2-4 days).
4.0 Personnel Qualifications/Responsibilities

4.1 Field operations require training specified in EAP's Field Safety Manual (Ecology, 2006) such as First Aid, CPR, and Defensive Driving.

4.2 Because the procedure requires the use of hazardous materials, training is required as per the Ecology Chemical Hygiene Plan and Hazardous Material Handling Plan (Section 1) (WA State Department of Ecology 2006), which includes Laboratory Safety Orientation, Job-Specific Orientation and Chemical Safety Procedures. The Standard Operating Procedures in Section 16 of the Chemical Hygiene Plan and Hazardous Material Handling Plan for handling chemicals must also be followed.

5.0 Equipment, Reagents, and Supplies

5.1 Bridge sampler (based on design presented in Figure 4500-0:1 of the 20th Edition of Standard Methods), 1 L Funnel, or Kemmerer/Van Dorn samplers
5.2 Sampling ropes 1 @ 10 ft., 1 @ 35 ft. and 2 @ 55 ft.
5.3 Extension pole with three prong stainless clamp
5.4 1-L funnel with tubing
5.5 Field Logbook or Field Data Report Form
5.6 Meter Calibration Log Form
5.7 Ambient Run Checklist
5.8 Sample tags
5.9 Sample coolers
5.10 Sample bottles
5.11 Cube ice
5.12 Gel-Ice (Blue Ice)
5.13 250 mL 10% HCl
5.14 Bacteria sampler
5.15 Long-line thermistor
5.16 Red-liquid thermometer
5.17 Weighted measuring tape
5.18 USGS gage keys
5.19 Peristaltic pump and filter holder
5.20 Hach pH meter with a three point calibration capability.
5.21 Approved Hach pH probe and backup probe.
5.22 Hach pH 4, 7, & 10 Buffers.
5.23 Hach pH probe filling solution.
5.24 pH 7 QC buffer (from another manufacturer - not Hach).
5.25 Hach 4-cell Conductivity probe
5.26  2 –100 μmhos/cm conductivity standards
5.27  2 – 1 L nutrient grab sample bottles¹ (marked up with black permanent ink and MSDS sticker)
5.28  1 – 1 L pH and conductivity grab sample bottle (marked w/red or green permanent ink)
5.29  DO box that has the following supplies:
5.29.1  300 mL BOD bottles (enough for the Run plus two spares)
5.29.2  Glass BOD stoppers
5.29.3  Plastic BOD bottle caps
5.29.4  3 mL graduated disposable transfer pipettes (one dedicated to each reagent)
5.29.5  Manganous sulfate monohydrate reagent bottle with MSDS sticker
5.29.6  Alkali-iodine-azide reagent bottle with MSDS sticker
5.30  Deionized water (DI water) used to rinse sampling bottles and equipment.
5.31  2-750 mL (or 500mL) plastic DI wash bottles
5.32  Metals sampling supplies:
5.32.1  Hand vacuum pump with hose and pressure gage
5.32.2  500mL Teflon FEP bottles pre-filled with de-ionized water by the lab
5.32.3  125 mL narrow mouth poly bottle containing H2SO4 preservative for hardness sample
disposable 0.45 micron cellulose acetate filter unit (pre-cleaned)
5.32.4  Small Teflon vials containing 5 ml concentrated nitric acid preservative
5.32.5  Powder-free vinyl or nitrile disposable gloves
5.33  Baking Soda
5.34  Eyewash Station
5.35  Digital Camera

6.0  Summary of Procedure

6.1  Annual Run Preparation. This process typically begins in the winter (almost a year ahead of the sampling schedule).

6.1.1  The first objective is to work with the regional watershed leads and other Ecology staff to prioritize and select new Basin Stations and metals sample stations² (see Attachment A for draft station selection guidance).

6.1.2  The next objective is to complete the “RunOrder” table in the “R&SNewWYPlanning” database. Then, notify the Ambient Database Administrator that the RunOrder table has been updated and he will use the database to generate the following documents: (1) Lab # (assigns lab numbers for each of the run stations), (2) Bottle Order (details the sample bottle needs, delivery, and pickup schedules for each Ambient Monitoring Run).

6.1.3  The administrator will then forward the finalized Lab # and Bottle Order documents to the Manchester Environmental Laboratory (MEL) and post them on the Y drive.

¹ These should contain about 200 mL of 10% HCL solution that is replaced every other Run
² These are sampled every other month.
6.1.4 The final objective is to draft and post the following two run documents on the Y drive (Y:\ambient) under the appropriate water year folder (WY___Docs) and run name by mid-September: (1) Run Times (details the planned daily time schedule) and (2) Run Directions (details driving and sample location directions).

6.2 Monthly Run Preparation. This should begin one week in advance of a run and requires: the completion and posting of a Field Work Plan & Contact Person Form, making sample tags, printing out the Field Data Report Form and the Lab Analyses Required Form (LAR), pre-booking air shipment(s), forward the air shipment confirmation e-mail to the courier, and making hotel reservations.

6.2.1 Samplers should always prepare for a Run by following a Run Checklist (see Attachment B) to ensure that all of the necessary tasks, sampling equipment, supplies, sample containers, and safety gear have been dealt with or loaded in the van. Note: Run sample bottles are delivered to the OC bottle storage room (or the designated regional location) by the lab courier the Wednesday before the scheduled run. The lab courier should be contacted if they are not there or the order is incorrect.

6.2.2 Field Work Plan & Contact Person Form.

6.2.2.1 Samplers must complete and post the Field Work Plan & Contact Person Form on SharePoint http://ecywbleyadx0/sites/eap/Field%20Schedules/Forms/AllItems.aspx along with links to the Run Directions and Run Times documents before beginning a run.

6.2.2.2 The information on the form enables family and program staff to call a sampler in case of an emergency or conduct a search if there was a mishap.

6.2.2.3 If plans change (lodging, cell phone number, etc.) the sampler must contact a supervisor or the section secretary to revise the information.

6.2.2.4 If the sampler fails to check in with the contact person, then the contact person needs to notify the supervisor to begin efforts to locate the sampler. Note: Van cell phones need to be kept on during work hours to allow the lab courier or other staff to get shipment information or to discuss other program related needs.

6.2.3 Making Sample Tags

6.2.3.1 Use the River and Stream Data Management Database to print the sample tag labels for the Run.

6.2.3.2 Stick the labels to the Rite in the Rain sample tags provided by MEL.

6.2.3.3 Rubber band the labeled tags by station and by the planned sampling order.
6.2.4 Printing Out the Field Forms.

6.2.4.1 Use the River and Stream Management Database to generate the Field Data Report and the LAR forms. Check the accuracy of the pre-entered information (run date, sampler…) on the forms before printing them (see Attachment C examples).

6.3 Pre-Run Procedure

6.3.1 Refill the DI water containers (2 L bottles and 5 gallon carboy). *Note: this task may also be done at the end of the Run if a DI water source is not available at the satellite office operation center.*

6.3.2 Turn on the cell phone.

6.3.3 Soak the conductivity probe in tap water for at least 30 minutes (overnight is better) before calibrating.

6.3.4 Put several scoops of ice into each sample cooler needed for the Run day and set the coolers into the van. If on a multiple day Run that includes an overnight stay, then consolidate the ice needed into a cooler for each day and top the cooler(s) off with several frozen Gel-Ice. If shipping by air cargo, pack one cooler with gel ice.

6.3.5 Calibrate the van barometer using the digital barometer located in the OC wet lab (or by another means such as a local weather station - but note that weather stations report BP corrected to sea level which must be converted back to absolute pressure).

6.3.6 Check the calibration of the long-line thermistor with an alcohol thermometer and note the result on the Meter Calibration Log Form.

6.3.7 Empty and refill the dedicated 4, 7, and 10 Hach pH buffer calibration bottles with fresh buffer solution that are the same temperature and at least 10°C.

6.3.8 Empty and refill the QC 7 pH buffer and conductivity standard bottles.

6.3.9 Clean the conductivity probe cells with a Q-Tip and rinse it with DI water.

6.3.10 Check the calibration of the long-line thermistor with an alcohol thermometer and note the result on the Meter Calibration Log Form.

6.3.11 Clean the inside of the filter apparatus by removing the hard plastic support from the base and cleaning underneath with a brush, if necessary. Re-assemble and pump (cycle) 10 % HCL through the filter stand followed by flushing it for at least 10 seconds with DI water from the 2 L storage bottle located in the sink.
6.4  **Every Morning**

6.4.1  Unplug the pH probe fill hole and slowly pull the attached probe soaker bottle down the probe about one half-inch to create a suction that will extract filling solution to clear the junction (this step can also be done when trouble shooting). Set the probe aside and upright (tip down) for about two minutes to allow the fill solution to be pulled through the junction.

6.4.2  Grasp the bottle cap and probe to keep them from moving and unscrew the soaker bottle. Put the bottle aside where it will not tip over.

6.4.3  Top off the probe fill chamber with filling solution. Remove the bottle cap and put it aside where it will not become contaminated. Rinse the probe with DI water and then shake off any water drops.

6.4.4  Do a three point calibration and measure each buffer a minimum of three times over the course of two minutes to ensure a stable reading was obtained (i.e., read the 4 buffer three times, then the 7 buffer three times, then the 10 buffer three times). Then push the done button and record the calibration slope and other calibration results on the Meter Calibration Log Form.

6.4.5  Compare the calibration Millivolt results and other calibration data to the expected pH calibration ranges shown in the lower left corner of the form.

6.4.6  If a buffer calibration result is beyond its expected buffer range by 2 mV, then suspect a bad buffer. Push the cancel button to go back into the calibration mode, and re-measure a freshly opened buffer for the suspect buffer, push the done button, and record the new result.

6.4.7  If the calibration results are within the expected ranges, then push the store button.

6.4.8  Check the calibration accuracy by measuring the pH QC 7 buffer and comparing the result to the true value of the buffer based on the buffer temperature. Record the buffer true value and measurement on the form.

6.4.9  Rinse probe with DI water, replace the bottle cap, put the end of the probe on the bottom of the quarter-filled probe soaker bottle, and slide the bottle cap down until it contacts the bottle threads.

6.4.10 Plug the fill hole, grasp the bottle cap and probe to keep them from moving, and screw on the soaker bottle (goal is to prevent pushing air bubbles in the fill chamber).

6.4.11 Store the probe upright (tip down).

6.4.12 Rinse the conductivity probe with DI water.
6.4.13 Calibrate the conductivity probe noting the probe I.D. number, standard, the initial and final cell constants, and any other required information on the Meter Calibration Log Form (see Attachment D for meter calibration log form). *Note: the conductivity standard is easily contaminated. Keep it tightly capped and avoid splashing other solutions or water into it. Also, freshly opened standard may be used for up to 5 days before discarding.*

6.4.14 Store the conductivity probe in tap water.

6.4.15 Insert a new filter into the filter stand and wet the new filter with DI water to help keep it in place. Reassemble the filter apparatus and turn the filter pump on for 10 seconds to further flush the apparatus.

6.4.16 Select an empty BOD bottle from the DO box, record its number on the Field Data Report Form, set it in the bridge sampler bucket, and secure the bucket lid.

6.4.17 Consolidate the 10% HCl solution from the two dedicated 1 L nutrient grab sample bottles (marked up with black permanent ink) into one of the bottles, triple rinse the empty bottle with DI water, and secure it in a bridge sampler bottle holder location.

6.4.18 Rinse a dedicated 1 L pH and conductivity grab sample bottle (marked with red permanent ink) with DI water and secure it in another bridge sampler bottle holder location.

6.4.19 Secure clean 1 L TSS and 0.5 L general chemistry (mostly used for turbidity analysis) sample bottles in the remaining bridge sampler bottle holder locations.

6.4.20 Secure a bacteria sample bottle in the bacteria sampler.

6.5 **Sampling Procedure.**

6.5.1 Deploy the Long-line thermistor (LLT) electrode and if warranted do an RP measurement.

6.5.2 Use one of the following three basic sample collection methods: bridge sampler (mostly used to collect samples from bridges), hand dip, and extension pole. *Note: Always survey the sample location for hazards (such as boating traffic or floating woody debris) that must be avoided when using the sampling gear. Also, if necessary, put on a high-visibility safety vest, turn on the amber strobe beacon light or vehicle emergency flashers, and put out the traffic cones and warning signs.*

6.5.3 Bridge Sampler Method. Carry the sampling gear to sample at the station (e.g., bridge sampler, sample bottles, bacteria sampler, sample ropes, and long-line thermistor) onto the bridge to a well mixed location such as the main part of the channel where representative stream samples may be collected.
6.5.3.1 Lower the thermistor probe into the water and let it equilibrate for at least two minutes while completing some of the other sampling tasks.

6.5.3.2 If called for, measure the stream stage height\(^3\) and record the result in the Yellow Field Logbook (Flow Book). Also, record the weighted measuring tape correction factor or check bar measurements. Note: The keys to the gage houses and wire weight gage boxes are located on the key ring stored in the van above the sampling ropes.

6.5.3.3 Attach the sampling rope to the bridge sampler\(^4\), remove all the bottle caps, and set the caps aside where they can remain clean.

6.5.3.4 Carefully lower the bridge sampler to the water surface, taking care to not dislodge any bridge debris onto it. Allow the bottom of the sampler to touch the water surface, and then raise the sampler off the water for a few moments to allow any debris from the bottom of the sampler to drop off and float away. Then rapidly lower the sampler about 0.5 meters to submerge it. Note: This minimizes the sampling of surface film and any debris from the bottom of the sampler.

6.5.3.5 When the bubbles from the bridge sampler bucket vent tube stop (bucket is full), retrieve the sampler taking care not to dislodge bridge debris into it. If a swift current carries the sampler downstream (before it can completely fill), then pull the sampler above the water, allow it to swing upstream, and then drop it back into the water. This action may need to be repeated a few times until the bucket is full.

6.5.3.6 Set the bridge sampler aside and replace the bottle caps.

6.5.3.7 Note: If alkalinity or other special study grab samples are needed, then collect them using the bridge or bacteria sampler. A sample bottle may be added to the bridge sampler through the use of a rubber tie down strap.

6.5.3.8 Memorize or record the water temperature, push the meter hold button to lock the result, retrieve the thermistor probe, and set the thermistor aside.

6.5.3.9 Attach the sampling rope to the bacteria sampler, remove the aluminum foil-covered stopper or cap from the bacteria bottle, and place the aluminum foil-covered stopper or cap where contamination can be avoided.

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\(^3\) Stream stage height measurements are obtained at some stations from a reference point (RP) by using a weighted measuring tape, a USGS weighted wire gage, or a staff gage.

\(^4\) The bridge sampler with sample bottle holders can simultaneously collect DO, turbidity, total suspended solids, pH, conductivity, and nutrient samples.
6.5.3.10 Move a few feet over from the location where the bridge sampler was retrieved and carefully lower the bacteria sampler to the water surface, taking care to not dislodge bridge debris or the bridge sampler retrieval water onto it. Allow the bottom of the sampler to touch the water surface, and then raise the sampler off the water for a few moments to allow any debris from the bottom of the sampler to drop off and float away. *Note: This minimizes the sampling of any debris from the bottom of the sampler.*

6.5.3.11 Lower the sampler part way into the water but do not submerge the lip of the sample bottle. Allow the current to re-orient the sampler so the sample bottle is on the upstream side of the sampler. Then rapidly lower the sampler about 0.5 meters to completely submerge it. *Note: This minimizes the sampling of surface film and prevents contamination from the bacteria sampler.*

6.5.3.12 Retrieve the bacteria sampler taking care to not dislodge bridge debris onto it.

6.5.3.13 Carefully replace the aluminum foil-covered stopper or cap in a way that avoids contamination to the inside of the bottle.

6.5.3.14 Return to the van with all the sampling gear.

6.5.4 Stream Side (1-L Funnel and hand dip) Method. This method is typically used to collect samples within reach of the water surface when standing in or near the stream.

6.5.4.1 Carry the funnel, thermistor, and needed sample bottles using vest pockets and an empty bucket to a well mixed location such as the deepest part of the active channel or another location where a representative sample may be collected. *Note: Do not contaminate the sample location by wading upstream of it or collect a sample from an eddy.*

6.5.4.2 Put the thermistor probe in the water and let it equilibrate for at least two minutes while completing some of the other sampling tasks.

6.5.4.3 If called for, measure the stream stage height^5 and record the measurement in the Yellow Field Logbook (Flow Book). Also, record the weighted measuring tape correction factor or check bar measurements. *Note: The keys to the gage houses and wire weight gage boxes are located on the key ring stored in the van above the sampling ropes.*

6.5.4.4 Rinse the funnel in the stream.

6.5.4.5 Invert the funnel or orient the open end of the funnel upstream and slowly submerge it until it and the funnel tubing completely fills avoiding any entrainment of air bubbles. Pinch the end of the funnel tubing and remove the funnel (top end first) from the water.

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^5 Stream stage height measurements are obtained at some stations from a reference point (RP) by using a weighted measuring tape, a USGS weighted wire gage, or a staff gage.
6.5.4.6 Insert the end of the funnel tubing into the bottom of a BOD bottle, allow the funnel to overfill the bottle until it is nearly empty, and then quickly withdraw the tubing (do not use any samples that were aerated by the final discharge from the funnel). Insert the glass stopper in the BOD bottle and cap it.

6.5.4.7 Hold the base of one of the sample bottles with one hand and remove the bottle cap. Then invert the bottle, reach upstream, and plunge the bottle into the water about 15 cm (6 inches), and then tip the bottle mouth up toward the water surface. Allow the bottle to fill, take it out of the water, replace the cap, and repeat the bottle filling process to fill the remaining sample bottles. *Note: The pH/conductivity bottle should be filled completely; the other bottles should be filled to the shoulder.*

6.5.4.8 Memorize or record the water temperature, retrieve the thermistor probe.

6.5.4.9 Return to the van with all the sampling gear.

6.5.5 Extension Pole Method. This method is typically used to reach a more representative or undisturbed sample location from the stream bank or to sample a shallow stream from a bridge.

6.5.5.1 Carry the extension pole, funnel, thermistor, and needed sample bottles using vest pockets and an empty bucket to a well mixed location such as the deepest part of the active channel or another location where a representative sample may be collected. Do not contaminate the sample location by wading upstream of it.

6.5.5.2 Put the thermistor probe in the water and let it equilibrate for at least two minutes while completing some of the other sampling tasks.

6.5.5.3 If called for, measure the stream stage height\(^6\) and record the measurement in the Yellow Field Logbook (Flow Book). Also, record the weighted measuring tape correction factor or check bar measurements. *Note: The keys to the gage houses and wire weight gage boxes are located on the key ring stored in the van above the sampling ropes.*

6.5.5.4 Secure one of the sample bottles in the extension pole clamp (Collect the FC sample last\(^7\)), remove the cap from the bottle, and place the cap where contamination can be avoided.

6.5.5.5 Use the extension pole to position the bottle just over the desired sample location.

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\(^6\) Stream stage height measurements are obtained at some stations from a reference point (RP) by using a weighted measuring tape, a USGS weighted wire gage, or a staff gage.

\(^7\) Collect the FC sample first in really slow moving streams. This avoids the potential of having the other sampling gear contaminate the sample location for the bacteria sample.
6.5.5.6 Invert the bottle and in one quick motion plunge the mouth of the bottle into the water about 15 cm (6 inches) and then tip the bottle mouth toward the water surface. Wait until the bottle has filled, then take it out of the water, replace the cap, and remove the bottle from the clamp.

6.5.5.7 Repeat this bottle filling process to fill the remaining grab samples.

6.5.5.8 The DO sample must be collected following 1L funnel procedure noted in 6.4.2 above or in combination with the extension pole.

6.5.5.9 Memorize or record the water temperature, retrieve the thermistor probe.

6.5.5.10 Return to the van with all the sampling gear.

6.6 Field Processing Procedure. Field processing fulfills three essential purposes: to preserve (fix) the DO sample, to prepare the individual samples for shipment to the lab, and to obtain field measurements for conductivity, pH, and barometric pressure. The typical field processing consists of the following procedure:

6.6.1 Put all the sampling gear into the van.

6.6.2 Tag the fecal coliform sample with the appropriate tag and place it in a cooler of ice.

6.6.3 Remove the BOD bottle from the bridge sampler bucket.

6.6.4 Remove the bottle stopper and fix the sample by adding two milliliters of manganous sulfate reagent followed by two milliliters of alkaline-azide reagent using the disposable pipettes reserved for each reagent. Add these reagents by dispensing them onto the inside neck of the bottle near the top of the sample (do not immerse the tip of the pipette). This should avoid splashing and entraining air bubbles into the sample and prevent any contamination of the reagents.

6.6.5 If necessary, tap the side of the BOD bottle to dislodge any air bubbles clinging to the inside of the bottle. Then insert a glass stopper in the BOD bottle and tip it to discard the displaced water.

6.6.6 Replace the stopper and invert the bottle a few times to mix the reagents into the sample.

6.6.7 Add a few milliliters of water around the stopper to form a water seal and cover the bottle top with a plastic BOD bottle cap.

6.6.8 Place the fixed sample into the DO box. Note: samples must be analyzed within four days.
6.6.9  Get into the van and record the sample time and the stream temperature on the Field Data Report Form. (Be sure to record accurate sample times at Hydrolab stations.)

6.6.10  Remove the pH and conductivity grab sample bottle (marked with red or green permanent ink), rinse the pH and specific conductivity measurement cups and probes with sample water, and gently over fill the pH and conductivity measurement cups with the sample water. Note: excessive agitation of the sample water will affect pH.

6.6.11  Unplug the pH probe fill hole and carefully remove the pH probe soaker bottle, rinse the probe with DI water, and put it in the pH measurement sample cup. Turn on the meter and gently stir the pH probe for several seconds every half minute (or so) for three to five minutes while completing some of the other field processing tasks.

6.6.12  Open a 125mL preserved nutrient bottle (contains 0.25 mL of sulfuric acid) and a 125 mL preserved nutrient bottle (contains 0.25 mL of hydrochloric acid) set them in the sink bottle holders. Avoid contact with the acid. Shake the 1 L nutrient sample to ensure it is thoroughly mixed and fill each of the preserved nutrient bottles to the bottle shoulder. Cap the bottles and tip them to mix the acid into the samples and set them aside. Also collect a Hardness sample if Metals samples are to be collected at the station. Note: special study samples such as dissolved organic carbon (DOC), total organic carbon (TOC), filtered total phosphorus, and filtered total nitrogen samples should also be sub-sampled out of the nutrient grab sample and processed at this time.

6.6.13  Turn on the filter pump and put the intake hose in the remaining 1 L nutrient sample. Allow the filtered sample water to run through the filter apparatus for 10-15 seconds to ensure that the DI water has been purged from it. Then fill a 125-mL amber bottle (no preservative) to the shoulder with filtered sample water, cap it, and set it aside.

6.6.14  Remove the intake hose from the 1 L nutrient sample bottle and rinse hose exterior with DI water. Then put the hose in DI water and let the pump run for 10-15 seconds to flush the interior of the filter apparatus.

6.6.15  Gently stir the pH sample with the pH probe for several seconds prior to and during the time it takes for the meter to indicate a stable sample measurement. Repeat this process until consecutive stable readings are within 0.02 pH units. Record the result and the sample temperature on the Field Data Report Form. Note: This process may take several minutes and that gradual sample temperature changes may alter the pH or prolong the time it takes to obtain a stable result.

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8 Make sure there are a few drops of acid in each bottle.
6.6.16 If the pH result equals 6.5 or less or 8.5 or higher, then check the calibration of the pH meter using the closest buffer (7 or 10). Record the calibration check result on the Field Data Report Form and if necessary, recalibrate meter, and re-measure the sample.

6.6.17 Check the calibration of the pH meter after the first, middle, and last station of the day using the QC 7 pH buffer. Record the check result on the Field Data Report Form and the Calibration Log Form. If necessary, recalibrate meter, and re-measure the sample.

6.6.18 Record the conductivity result on the Field Data Report Form or in the Field Logbook. The meter displays results to the nearest tenth, so round the result to the nearest whole number. If the tenths digit > 0.5, then round up; if it is < .5, then round down; and if it is = to 0.5 round to the nearest even number. For example, a conductivity result of 103.5 would be rounded to 104 and a result of 62.5 would be rounded to 62.

6.6.19 Record the barometric pressure, stream stage height, and any other measurements on the Field Data Report Form. Then record any weather or unusual site specific observations, and equipment issues (spend some time on this as these narrative observations can help explain any anomalous data on the form).

6.6.20 **Note:** if you observe any unusual or suspicious looking colored water in or entering the stream, or other potential environmental hazards (drums, dead animals, or new invasive plants or benthic macro invertebrates), then take some pictures and make notes about the observation and your exact location. If the suspicious looking colored water or potential environmental hazard is dangerous, then do not approach!

6.6.21 If the suspicious looking colored water is obviously not dangerous, then take some precautions and collect two water samples (500mL bacteria and 1L - TSS) to send to the lab. Also, if warranted, collect any potential new invasive plant samples for later identification. Send to Jenifer Parsons (Program plant specialist) or other agency staff that can do the identification.

6.6.22 **In addition,** immediately report these observations to the appropriate Ecology contacts (Ecology’s Spills Hotline, regional office staff, and/or watershed lead) and indicate that there are samples being sent to the Manchester Lab for potential analysis if it is warranted.

6.6.23 Label the all sample bottles with the appropriate sample tags, double check the station ID on the tag, and place them in ice in a cooler.

6.6.24 Remove and discard the used filter from the filter apparatus, rinse the inside of the apparatus with DI water, and insert a new filter.

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9 If the difference between the pH meter result and the standard is greater than or equal to 0.10 pH units then recalibrate the meter, if the difference between the pH meter result and the standard is greater than or equal to 0.15 pH units, then recalibrate the meter, re-read the sample, and "J" data since last calibration check.
6.6.25 Wet the new filter with DI water to keep it in place, reassemble the filter apparatus, and then turn the filter pump on for 10-15 seconds to flush the apparatus with DI water.

6.6.26 Select an empty BOD bottle from the DO sample box, record its number on the Field Data Report Form, place it in the stainless bridge sampler bucket, and secure the bucket lid.

6.6.27 Rinse the used nutrient sample bottle with DI water and pour the 10% acid solution from the spare bottle into the newly rinsed bottle. Cap it, shake it, and set it aside in the sink to soak until the next station.

6.6.28 Triple rinse the newly emptied nutrient sample bottle with DI water, and secure it in a bridge sampler bottle holder location.

6.6.29 Rinse the dedicated 1 L pH and conductivity grab sample bottle with DI water and secure it in another bridge sampler bottle holder location.

6.6.30 Secure clean 1 L and 0.5 L sample bottles in the remaining bridge sampler bottle.

6.6.31 Rinse probe with DI water, carefully re-attach the quarter-filled probe soaker bottle, plug the fill hole, and store the probe upright.

6.6.32 Decontaminate all field gear and equipment following the “Standard Operating Procedures to Minimize the Spread of Invasive Species” (Parsons, et. al, 2012).

6.6.33 Repeat the Sample Collection and Processing Procedures (see procedures 6.4, and 6.5 above) at the rest of the sampling stations.  

Note: the calibration of the pH meter must be checked against a QC 7 pH buffer (not used for calibration purposes) after the first, middle, and last stations of the day. The conductivity meter needs to be checked after the last station of the day. Record the results on the Field Data Report Form and on the Meter Calibration Log Form.

6.7 Metals Sampling Procedure. If called for, return to the sample location, and collect the metals samples\(^\text{10}\).

6.7.1 This sampling procedure generally follows EPA Method 1669. Samples are collected as single grabs in a 500ml Teflon FEP bottle using the stainless steel metals sampler or by hand. Care must be used at all times when collecting and processing metals samples to avoid contaminating the inside of the sample bottle or cap with debris and to minimize the contact with ambient air.

\(^{10}\) Metal samples are collected at a few stations every other month.
6.7.2 Metals samples should be processed (filtered, preserved, and placed on ice) within 15 minutes after having been collected. If the metals processing requirement was not met then make a note to the lab on the field sheet (and in the remarks) indicating how long it took to process the sample. The lab may “J” qualify the data. Note: the holding time prior to analysis for all metals, except mercury, is six months and the holding time for mercury is 28 days.

6.7.3 Metals Sampler Method. This method is typically used to collect samples from a bridge or from the stream bank through the use of a rope.

6.7.3.1 Move to a well-mixed location such as the deepest part of the active channel where a representative sample may be collected.

6.7.3.2 Invert the Teflon sample bottle, remove the cap, and rinse the sampler with the “ultra pure” water that empties out of the bottle.

6.7.3.3 After the bottle empties, set the sampler down and replace the bottle cap.

6.7.3.4 Then fit the sample bottle into the base of the stainless steel metals sampler.

6.7.3.5 Remove the Ziploc bag covering the designated metals sampler cap\(^{11}\) and set aside the bag, remove the sample bottle cap and place the cap in the clean plastic bag the bottle shipped in.

6.7.3.6 Lower the sampler bottle cap lifting arm until the sampler cap covers the bottle opening (make sure the lifting arm can move up freely).

6.7.3.7 Attach the sampling rope.

6.7.3.8 Carefully lower the sampler to the water surface, taking care to not dislodge bridge debris onto it. Allow the bottom of the sampler to touch the water surface, and then raise the sampler off the water for a few moments to allow any debris from the bottom of the sampler to drop off and float away. Note: This minimizes the sampling of any debris from the bottom of the sampler.

6.7.3.9 Lower the sampler about 15 cm (6 inches) into the water. Allow the current to re-orient the sampler so the sample bottle is on the upstream side of the sampler. Then rapidly lower the sampler about 0.5 meters to completely submerge it. This minimizes the sampling of surface film. Note: At about 25 cm under the water surface, the sampler should automatically raise the bottle cap and allow the bottle to fill. Also, it may take more than 45 seconds for the bottle to fill.

6.7.3.10 Retrieve the filled bottle taking care to not dislodge bridge debris onto it or the sampler.

\(^{11}\) The cap must be kept clean and replaced before sampling the first metals station of a run or when contaminated.
6.7.3.11 Cap the filled sample bottle with the original cap from the clean plastic bag, remove it from the sampler, and place the bottle in the Ziploc bag it shipped in.

6.7.3.12 Repeat the procedure to obtain a second metals sample.

6.7.3.13 Return to the van with the samples and sampling gear.

6.7.3.14 Cover the metals sampler cap with a Ziploc bag.

6.7.4 Hand Dip Method. This method is typically used to collect samples from a small or shallow stream, or near the bank of a large stream.

6.7.4.1 Move to a well-mixed location such as the deepest part of the active channel or another location where a representative sample may be collected. *Note: Do not contaminate the sample location by wading upstream of it or collect a sample from an eddy.*

6.7.4.2 Grab the base of the sample bottle with one hand, invert the Teflon sample bottle, remove the cap, and let the “ultra pure” water empty out of the bottle.

6.7.4.3 Reach upstream and plunge the bottle into the water about 15 cm (6 inches) and then tip the bottle mouth up toward the water surface.

6.7.4.4 Allow the bottle to fill and then take it out of the water.

6.7.4.5 Replace the cap in a way that avoids contamination to the inside of the bottle and place the bottle in the Ziploc bag it shipped in.

6.7.4.6 Repeat the procedure to obtain a second metals sample.

6.7.4.7 Return to the van with the samples and sampling gear.

6.7.5 Extension Pole Method. This method is typically used to reach a more representative or undisturbed sample location from the stream bank or slow moving stream.

6.7.5.1 Secure the metals sample bottle in the extension pole clamp.

6.7.5.2 Move to a well-mixed location where a representative sample may be reached with the pole. *Note: Do not contaminate the sample location by wading upstream of it and do not collect a sample from an eddy.*

6.7.5.3 Invert the Teflon sample bottle, remove the cap, and let the “ultra pure” water empty out of the bottle. Also, put the cap into the Ziploc bag the bottle shipped in and put the bag in a location that will prevent contamination to the inside of the cap.

6.7.5.4 Position the bottle over the desired sample location.
6.7.5.5 Invert the bottle and in one quick motion plunge the mouth of the bottle into the water about 15 cm (6 inches). Then slowly move the bottle upstream with the bottle mouth tipped toward the water surface until the bottle has filled.

6.7.5.6 Take the filled bottle out of the water and then replace the bottle cap in a way that avoids contamination to the inside of the cap and bottle.

6.8 Metals Field Processing Procedure.

6.8.1 Total Recoverable Metals and Total Mercury.

6.8.2 Close the vehicle door to minimize drafts

6.8.3 Put on powder-free vinyl or nitrile disposable gloves.

6.8.4 Remove the disposable filter unit from the large Ziploc bag and set the bag and filter unit aside.

6.8.5 Unscrew the cap from the first sample bottle (but leave it on the bottle).

6.8.6 If necessary, gently squeeze the side of the sample bottle to displace about 5 ml of sample to make room for the Nitric acid preservative.

6.8.7 Carefully uncap the small Teflon vial containing 1:1 Nitric acid, lift the cap from the sample bottle and add the acid to the sample. Screw the cap on the sample and then re-cap the empty Nitric acid vial.

6.8.8 Attach the Total Metals and Total Recoverable Mercury sample tag to the sample bottle.

6.8.9 Place the tagged sample in its original Ziploc bag along with the empty (capped) Teflon vial, eliminate air from the Ziploc bag, seal it and then put it in the large Ziploc bag that contained the filter unit.

6.8.10 Dissolved Metals.

6.8.10.1 Attach the hand pump (or peristaltic pump) hose to the metals filter unit.

6.8.10.2 Remove the cap from the second sample bottle; lift up one side of the filter unit lid about 3 cm (1 inch), and pour the sample into the top of the unit. Note: Avoid touching or contaminating the inside of the filter unit.

6.8.10.3 Cap the empty sample bottle and put it into the large Ziploc bag that also contains the tagged total metals sample.
6.8.10.4 Hold onto the filter unit with one hand and use the other hand to squeeze and release the 
hand pump lever (or turn on the peristaltic pump on the lowest setting) to create a 
vacuum no greater than 20 PSI\textsuperscript{12} to filter the sample.

6.8.10.5 Filter as much of the collected sample as possible (at least half).

6.8.10.6 Empty “ultra pure”-water from an unused Teflon bottle and set the cap on the bottle 
opening.

6.8.10.7 Unscrew the bottom of the filter apparatus, remove the cap from the top of the unused 
Teflon sample bottle (do not set the cap down), pour the filtered sample into the Teflon 
bottle, and set the cap on the bottle opening.

6.8.10.8 Carefully uncap the small Teflon vial containing 1:1 Nitric acid, lift the cap off the 
bottle containing the filtered sample, and add the acid to the sample. Screw the cap on 
the sample and then re-cap the Nitric acid vial.

6.8.10.9 Attach the Dissolved Metals sample tag to the sample bottle.

6.8.10.10 Place the tagged sample in its original Ziploc bag along with the empty (capped) Teflon 
vial.

6.8.10.11 Eliminate air from the Ziploc bag, seal it, and put it in the large Ziploc bag that contains 
the tagged total metals sample and the empty Teflon bottle.

6.8.10.12 Eliminate air from the large Ziploc bag and place the bagged samples on ice in a cooler.

6.9 Quality Assurance / Quality Control Sampling Procedures. Stations for Quality 
Assurance / Quality Control (QA/QC) samples are assigned at random prior to the water 
year. A typical Run has two field blank stations and ten field replicate/field split 
stations per year. One QA sample station is assigned per Run per month. This 
sampling follows the regular sampling process for the station.

6.9.1 Field Replicate/Field Split Samples\textsuperscript{13}.

6.9.1.1 Repeat the normal sample collection and processing procedures (See sections 6.4 and 
6.5) to collect a second set of field grab samples at the station. Then collect two 
samples out of the of the same 1 L nutrient grab sample (instead of one set). \textit{Note: the 
split samples for the station are usually just nutrient samples but they may also include 
non-nutrient samples such as hardness, TOC, and DOC.}

\textsuperscript{12}All pumps that are used for metals filtering must be verified that the lowest setting will not  ...

\textsuperscript{13}Replicate samples are collected after the normal set of samples have been collected, processed, and the sampling 
equipment has been set up to sample another station. The QA \textsubscript{-1} samples are used to assess variability from short-
term instream processes and field and lab processing. The QA-2 samples are used assess variability from only the 
field and lab processing.
6.9.1.2 Label the first set of collected samples with the QA_-1 (field replicate) tags and label the second samples with the QA_-2 (field split) tags. Note: There is no need to split any sample that is collected directly in the bottle and sent to the lab. Also note that the QA_-3 tags is are to be used if any QA samples are collected at a station other than the station associated with the QA_-1 and QA_-2 samples.

6.9.2 True Process Field Blank Samples. The purpose of this procedure is to subject the blank samples to all the typical sample collection contamination sources.

6.9.2.1 Load the bridge sampler with all the normal plastic sample bottles (TSS, general chemistry, nutrient, and pH/conductivity). Go to the sample site, remove the bottle caps, and set the caps in the typical location you would use at that site (such as on the road or bridging). Lower the bridge sampler to the water surface (do not immerse anything into the stream), retrieve the sampler, and cap the bottles.

6.9.2.2 Return to the van and fill all the containers except the stainless bucket with the Lab provided DI water.

6.9.2.3 Fill the conductivity measurement cup with water from the pH/conductivity grab sample bottle, allow the conductivity probe to stabilize, and record the measurement.

6.9.2.4 Go through the normal process of obtaining the preserved nutrient bottle samples and filtered nutrient samples from the nutrient grab sample bottle.

6.9.2.5 Do not collect fecal coliform or DO samples or take pH or temperature measurements.

6.9.2.6 Label the bottles with the appropriate QA_-1 tags, place them in ice in a cooler, and note the time and conductivity measurement on the Field Data Report Form.

6.9.3 True Process Field Metals Blank Samples\textsuperscript{14}.

6.9.3.1 Load the sampler with a metals bottle (do not empty the special “ultra-pure” DI water out of the bottle). Go to the sample site, remove the bottle cap, and put the cap in a dry Ziploc bag to avoid any contamination. Lower the Metals Sampler to the water surface (do not immerse anything into the stream), retrieve the sampler, and cap the bottle.

6.9.3.2 Return to the van and follow the Dissolved Metals processing procedure (see procedure 6.7.2) and filter the ultra-pure de-ionized water from the sample bottle. Then pour the filtered DI water sample back into the same bottle the water came from, cap it, label it with a QA_-1 tag\textsuperscript{15}, and place it on ice.

\textsuperscript{14} One Metals blank is collected per Run per year.
\textsuperscript{15}If the QA sample is collected at a station other than the one associated with the QA_-1 blank samples, then tag it with a QA_-3 tag.
6.10 **End of Day QC Procedures.**

6.10.1 Check the calibration of the pH meter using the QC 7 pH buffer. Record the result on the Field Data Report Form and if necessary, recalibrate meter, and re-measure the last sample.

6.10.2 Rinse probe with DI water, carefully re-attach the quarter-filled probe soaker bottle, plug the fill hole, and store the probe upright.

6.10.3 Check the calibration of the conductivity meter. Record the result on the Field Data Report Form. If the conductivity measurement is not within 5 µmhos/cm of the standard then troubleshoot the meter\(^\text{16}\) and if necessary re-measure all of the samples using the general chemistry sample.

6.10.4 Review the information recorded on the Field Data Report Form for completeness.

6.10.5 Transfer the sample times to the Lab Analysis Required Form (LAR). Also enter the field contact phone number, relinquished by, relinquish time, number of coolers, and any comments.

6.11 **OC Walk-in Cooler Shipping Procedures.**

6.11.1 Drain the ice water from the sample cooler(s), top the samples off with a couple scoops of ice, and set the cooler(s) in the walk-in cooler.

6.11.2 Put in the completed LAR in the courier’s inbox tray located near the walk-in cooler.

6.12 **Greyhound or motor freight (truck) Shipping Procedures.** *Note: If possible, avoid shipping on Greyhound because this method can delay the receipt of the samples by the lab.*

6.12.1 Fold the completed LAR, put it in a plastic sandwich bag, and tape the bag under the sample cooler lid.

6.12.2 Drain the coolers of ice water, and top them off with some additional ice or frozen Gel-Ice (Blue-Ice). *Note: do not overload the cooler with Gel-Ice because this can freeze the samples. Also, all sample coolers used to ship samples must be in good condition and not leak.*

\(^{16}\) If the meter is in the non-linear function (nLF) and the temperature coefficient is 25, then use an unopened conductivity standard to verify the meter calibration (the standard is easily contaminated). If the meter needed to be changed to the non-linear function, temperature reference 25, or be recalibrated, then re-measure all of the samples.
6.12.3 Tape the cooler drain plug and lid using ¾ or 1 inch reinforced tape. It works best to tape over the drain plug first and then wrap tape twice around that end of the cooler and cooler lid.

6.12.4 Check the sample cooler(s) in at the package service counter of the shipper and provide Ecology’s account number along with any other necessary information.

6.12.5 If the shipper indicates any problems with the shipment schedule, then notify the courier.

6.13 **Airfreight Shipping Procedures.** GoldStreak – Alaska Airlines/Horizon Air Cargo is the current provider of this service for the sample cooler shipments. *Note: The airline may require a 24 hour advance notification procedure. The shipment can be booked online the week before the run.*

6.13.1 Fold the completed LAR, put it in a plastic sandwich bag, and tape the bag under the lid of an empty (dry) sample cooler lid of a cooler that is in good condition and will not leak. Tape the cooler drain plug using ¾ or 1 inch reinforced tape.

6.13.2 Transfer the iced samples into the empty (dry) sample cooler and be sure that the all the sample container lids are tight.

6.13.3 Top off the samples with several frozen Gel-Ice. The amount of Gel-Ice may need to be increased during hot weather to ensure that the samples remain at or below 4°C during shipment. If the Gel-Ice were frozen or kept frozen with dry ice, then use only a few of them to top off the samples.

6.13.4 Hold off taping the cooler(s) but take the tape with you so it can be done after check-in and TSA inspection.

6.13.5 Check the sample cooler(s) in at the airline airfreight office or ticket counter. They will need Ecology’s Customer ID number, your personal and Ecology ID, and possibly other necessary information. Request that they attach a Keep Cool Sticker to the cooler lid or side and have the officer from the Transportation Security Administration (U.S. Department of Homeland Security) tape the cooler lids down after the cooler contents have been inspected. If possible watch the process to be sure they remember to secure the cooler lids down with tape. *Note: The process allowed to get the cooler lids secured with tape varies at each airport. Some airport staff will let us tape the coolers using our tape, others will tape them using our or their tape (ask if you can watch for chain-of-custody reasons), and sometimes they will tape the lids but not allow you to watch.*

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17 Dry ice freezes Gel-Ice colder and some samples could be frozen if several of them are used.
6.13.6 Contact the lab courier with any changes to the planned air shipment or **air waybill number** (already noted in the forwarded airline confirmation), after the cooler(s) have been shipped.

6.14 **End of Day Procedures**

6.14.1 Call the contact person noted on the Field Work Plan & Contact Person Form.

6.14.2 Lift the tube out of the DI water for the filter apparatus, lay the tube across the top of the apparatus, turn on the pump, and pump the filter apparatus dry.

6.14.3 If the overnight air temperatures will be below 10°C (50°F), then move the meters, probes, DI water wash bottle, pH buffers, and conductivity standards into a heated room (hotel room, regional lab, or operation center).

6.14.4 If the overnight air temperatures will be at or below freezing, then also move the DI water, and DO box containing DO samples into a heated room to prevent freezing or loss to breakage.

6.15 **DO Analysis** - Note: Always dilute chemicals going into the sink with a continuous stream of tap water to prevent damage to the plumbing. *Note: these procedures are also documented in a Winkler training video: [http://teams/sites/EAP/videos/default.aspx](http://teams/sites/EAP/videos/default.aspx)*

6.15.1 Initial Cleaning Procedure:

6.15.1.1 Put on a plastic apron and Nitrile gloves.

6.15.1.2 Thoroughly rinse the flask and stir bar with deionized water.

6.15.1.3 Check and if necessary fill the Potassium bi-iodate dispenser and starch squirt bottle.

6.15.1.4 Fill the Sodium thiosulfate reservoir and loosen the reservoir cap. Note: it is best to do this a few hours before the titrations, so the solution may reach room temperature and there are no chemical reaction delays during the titration process.

6.15.1.5 Open the volumetric burette stopcock to a fill position.

6.15.1.6 Raise and lower the sodium thiosulfate storage bottle reservoir above and below the volumetric burette a few times to flush the burette and to mix the sodium thiosulfate in the reservoir.

6.15.1.7 Clamp the reservoir onto the workstation lab-frame above the volumetric burette.
6.15.1.8 Set a small beaker under the burette tip and turn the stopcock to the drain position to dispense the old thiosulfate from the burette but not the burette tip. Refill the burette and then drain it a second time to also rid any old thiosulfate from the tip. Avoid emptying the burette tip, because the resulting air bubble is difficult to eliminate.

6.15.2 Titration Procedure:

6.15.2.1 Remove the plastic cap from the BOD bottle.

6.15.2.2 Pour off the water seal and invert the bottle several times to mix the floc.

6.15.2.3 Allow the floc to settle to the lower half of the bottle.

6.15.2.4 Put on the face shield.

6.15.2.5 Remove the bottle-top sulfuric acid dispenser from the acid storage cabinet. The dispenser should already be pre-set to dispense 2 mL of acid.

6.15.2.6 Remove the glass stopper of the BOD bottle. Dispense 2 mL of the acid into the DO sample and put the acid bottle back into the cabinet. **Note:** Concentrated sulfuric acid is a very dangerous chemical and should be handled very carefully. Never add water to it and always immediately rinse and dispose of gloves that get any acid on them.

6.15.2.7 Re-stopper the BOD bottle and invert it several times over the sink until the precipitate has completely dissolved. The sample should have a clear yellowish color. If some floc remains in BOD bottle, then invert the bottle several times to mix the floc and allow 5-6 minutes for the precipitate to dissolve. If the floc still has not dissolved then add a few drops of sulfuric acid from the sulfuric acid dispenser until floc completely dissolves.

6.15.2.8 Slide a magnetic stir bar into an empty 500 mL Erlenmeyer flask.

6.15.2.9 Fill a 203 mL volumetric flask with the DO sample, transfer the sample to the Erlenmeyer flask, and set the flask in the sink.

6.15.2.10 Refill the volumetric burette with sodium thiosulfate (make sure the sodium thiosulfate escapes from the top nipple).

6.15.2.11 Place the Erlenmeyer flask containing the sample on the magnetic stirrer and turn on the stirrer to the lowest setting.

6.15.2.12 Titrate the sample with the Sodium thiosulfate from the volumetric burette until it turns to a pale yellow color.
6.15.2.13 Squirt 1 to 2 mL of the starch solution into the sample. Note: the addition of the starch solution earlier than this can cause a less distinct titration endpoint or overshooting the end point.

6.15.2.14 Continue the titration process by adding the sodium thiosulfate by quickly twisting the burette stopcock past the discharge point (or by slowly adding individual drops) until the purple color of the sample just disappears. This is the titration end point and it should be sharp and distinct. Care should be taken to avoid an end point overrun.

6.15.2.15 Check the titration end point of any sample that was possibly overrun by adding a drop of bi-iodate from a 3 mL graduated disposable transfer pipette to the titrated sample. If the end point is correct, a faint purple color should reappear. If more than one drop of bi-iodate is required to get a faint purple color, then the end point was overrun and a Back-Titration need to be done to correct the result (see 6.14.3 – Back-Titration).

6.15.2.16 Record the titration result or corrected titration result in the proper column on the Field Data Report Form or in the field notes as mg/L of DO. If the value is between the 0.1 mL marks on the burette, round the even numbers down and the odd numbers up (e.g., 10.25 to 10.2 and 10.35 to 10.4).

6.15.3 Back-Titration Procedure

6.15.3.1 Back-titrate an overrun end point sample using bi-iodate drops from a 3 mL graduated disposable transfer pipette (1 drop = 0.05 mg/L). Correct the final value if the back-titration requires fewer than or equal to 8 drops and record the result without qualification. If the back-titration requires more than 8 drops but less than or equal to 20, correct the final value and record the result with a "J" qualification (twenty drops are equivalent to 1 mg/L). If the back-titration requires more than 20 drops, do not record a result, but make a comment on the Field Data Report Form indicating the titration error.

6.15.3.2 If a graduated burette or pipette is available, then carefully back-titrate to the overrun end point sample using a measured quantity of bi-iodate and subtract the amount used to correct the final result.

6.15.4 Sodium Thiosulfate Normality Check. The test is done to verify the strength of the Sodium Thiosulfate solution and get a data correction factor. The normality check result should almost always be between 9.95 and 10.05 mL if the Sodium Thiosulfate has been stored properly. The result should also be very similar to those that others have recently recorded in the Titration Log.

6.15.4.1 After the first sample has been titrated to its end point, add exactly 10 mL of the bi-iodate standard using: a 10 mL volumetric burette, w/3-way stopcock, 10 mL bottle-top dispenser, or glass volumetric pipette. Rinse the inside wall of flask with starch solution to ensure that none of the standard is on it and re-titrate.
6.15.4.2 Repeat this procedure mid-way through the batch of samples to be titrated.

6.15.4.3 Record the volume of the sodium thiosulfate needed for each normality check on the field note book or worksheet and on the titration log located next to the titration station. Enter the average of the two normality check results into the database and the database will compute a correction factor and adjust the data (if warranted).

6.15.4.4 Note: These normality checks should be very close, within 0.1 mL. If they are not, then do at least two more until you have three consecutive results (within 0.1 mL of each other) to use to calculate a correction factor.

6.15.4.5 If you get less than a 9.95 mL result, then repeat the normality check on another sample but do the following first:

6.15.4.6 Eliminate air from the tip of the Potassium Biiodate bottle-top dispenser to ensure it dispenses a 10.0 mL volume (If warranted, verify the dispensed volume with a 10mL volumetric flask (or another accuracy method).

6.15.4.7 Gently dispense the Potassium Biiodate into the titrated solution in the bottom of the Erlenmeyer flask and avoid getting any on the inside flask wall,

6.15.4.8 Rinse the inside flask wall with starch solution to ensure that all of the Potassium Biiodate is in the titrated solution, and

6.15.4.9 Eliminate Sodium Thiosulfate drops/residue from the outside of the refillable burette tip and tube connection.

6.15.5 Correcting Titration End Point Results with Normality Check (NC) Results

6.15.5.1 Note: If using the ambient database, these corrections will be done automatically; simply enter the mL of thiosulfate needed into the database “correction factor” field.

6.15.5.2 Divide the average of the two or more normality check results into 10 to get the correction factor (10/NC avg.), and then multiply the measured result by the correction factor (CF) to get the corrected result (Corrected DO = measured DO × CF).

6.15.5.3 For example, if the average of the normality checks was 9.9 mL and the sample titration result was 11.5 mL, then:

6.15.5.4 Correction Factor Multiplier = (10/NC avg.) = (10/9.9 mL) = 1.01CF

6.15.5.5 Corrected Result = (measured DO × CF) = (11.5 mL × 1.01CF) = 11.6 mL

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18 The Ambient database automatically does this.
6.15.5.6  Note: The corrected result is the volume, in mL, of sodium thiosulfate used to titrate a 200mL sample. This volume is equivalent to the concentration of DO in mg/L.

6.15.6  Clean Up Procedure

6.15.6.1  Move the sodium thiosulfate reservoir back to its storage area on the counter.

6.15.6.2  Open the volumetric burette stopcock to a fill position (this allows the thiosulfate in the volumetric burette to return to the reservoir).

6.15.6.3  Tighten the reservoir cap, drain thiosulfate from the burette to a level just above the stopcock (leave thiosulfate in the tip), and leave the stopcock in a closed position.

6.15.6.4  Thoroughly rinse the used flasks and stir bar(s), and final rinse them with DI water.

6.16  End of Run Procedures.

6.16.1  Brush and DI rinse the pH and conductivity sample cups and store them upside down.

6.16.2  DI rinse the filter apparatus and pump the lines dry.

6.16.3  Rinse pH probe with DI water, carefully re-attach the quarter-filled probe soaker bottle, plug the fill hole, and store the probe upright.

6.16.4  Rinse the conductivity probe with DI water.

6.16.5  Store the meters, probes, pH buffers, and conductivity standards in a warm and dry area in the regional lab or operation center.

6.16.6  Refill the manganous sulfate monohydrate and alkali-iodine-azide reagent containers in the DO box.

6.16.7  Empty the van of trash and vacuum it out.

6.16.8  Top off the gas tank (tank must be at least ¾ full).

6.16.9  If warranted, get the van oil changed.

6.16.10  Turn any malfunctioning equipment into the Operation Center Technician along with a completed Equipment Problem Report Form for repair at the end of each Run. Malfunctioning equipment may result in unsafe sampling conditions and lost sampling opportunities.
6.16.11 Enter the field data results and comments into our Access-based database, review the entries for accuracy, and turn in the printout of the Run Field Data sheet along with the other documentation to the database manager. Note: The run isn’t considered complete until the field data have been entered and finalized in the database. This means that normally you would do the run, analyze the DO samples, clean up your gear, and enter data before doing any other non-run-related tasks.

7.0 Records Management

7.1 All hardcopy documentation of the data, such as completed Field Logbook and Field Data Report Forms are kept and maintained by the project lead. These documents are organized in binders or in expanding files. After about six years, hardcopies are boxed and moved to EAP archives.

7.1.1 The data are entered into our Access-based database, reviewed and verified following the Quality Control and Quality Assurance procedures, uploaded into EIM, and posted on our web page [http://www.ecy.wa.gov/programs/eap/fw_riv/rv_main.html](http://www.ecy.wa.gov/programs/eap/fw_riv/rv_main.html).

8.0 Quality Control and Quality Assurance Section

8.1 The data QA program for field sampling consists of three parts: (1) adherence to the SOP procedures for sample/data collection and periodic evaluation of sampling personnel, (2) consistent instrument calibration methods and schedules, and (3) the collection of a field quality control (QC) sample during each sampling run. Our QA program is described in detail in Hallock and Ehinger (2003) and Hallock (2012).

8.2 The field QC samples are collected as a duplicate (sequential) field sample. This consists of the collection of an additional sample approximately 15-20 minutes after the initial collection at a station. This sample represents the total variability due to short-term, in-stream dynamics, sample collection and processing, and laboratory analysis.

8.3 The annual field QC metals sample is a filtered field blank sample. This sample captures potential contamination from sample processing and laboratory analysis.

8.4 A two-tiered system is used to evaluate data quality of individual results based on field QC. The first tier consists of an automated evaluation of the data. Results exceeding pre-set limits are flagged. The second tier QC evaluation is a manual review of the data flagged in the first tier. Data are then coded from 1 through 9 (1 = data meets all QA requirements, 9 = data are unusable). Criteria for assigning codes are discussed in more detail in Hallock and Ehinger (2003) and Hallock (2012). We do not routinely use or distribute data with quality codes greater than 4.
8.4.1 The overall quality of data collected during the sampling year are evaluated in our annual reports (e.g., Hallock, 2011)

9.0 Safety

9.1 Safety is the primary concern when collecting samples. Since most sample sites are located on highway bridges, road and pass conditions should always be checked before departure (especially in winter). If roadside hazards, weather, accidents, construction, etc. make sample collection dangerous, then skip that station. Note the reason on the Field Data Report Form and notify your supervisor of the hazard when you return to the office. If the hazard is a permanent condition, relocation of the station may be necessary. Review Ecology’s Safety Program Manual periodically to assist with these safety determinations.

10.0 References


10.2 Ecology, 2006. Chemical hygiene plan and hazardous material handling plan. Olympia, WA.


Attachment A – Run Checklist

Draft Water Year Planning and Basin Station Selection Guidance

We have had problems with final station selection not happening until late September or even into October, after the new Water Year has already begun. As a result, scoping gets neglected, location metadata collection may be sloppy or overlooked, samples may be missed, stations get moved after sampling has begun, and data management is convoluted, which risks data being compromised.

Sometimes there are legitimate reasons for delaying station selection, but too often the reason is that we are all too busy with other things. To help shepherd the station selection process, this document includes some milestones for preparing the ambient runs for a new water year, as well as some guidance for identifying suitable basin stations.

**Milestones**

<table>
<thead>
<tr>
<th>Date</th>
<th>Task</th>
</tr>
</thead>
<tbody>
<tr>
<td>June</td>
<td>Ambient regional staff will work with stakeholders (regions, TMDL staff, TMDL effectiveness staff, watershed leads, local governments, etc.) and each other to develop a list of basin stations for the coming water year. (See selection criteria, below.) Identify any supplemental parameters (and funding sources), metals stations, flow-critical stations, etc. to the ambient coordinator. (Some scoping at questionable stations may be required at this time.)</td>
</tr>
<tr>
<td>Late July</td>
<td>Ambient regional staff will submit lists of basin stations (final, pending scoping) directly to stakeholders and, via the ambient coordinator to the flow group and EAP managers. Include supplemental parameters, reasons for sampling each station, etc. Also include any proposed stations that were not selected, and the reason they were not selected.</td>
</tr>
<tr>
<td>August</td>
<td>Ambient staff will scope basin stations. Look for safe parking and bridge access, safe and representative (e.g., well-mixed) bank sample location. Consider high-flow conditions (and and high-tide condition, where applicable). Record cross-section temperatures and conductivities. Take notes for developing run directions (road names, etc.). Take photographs (upstream and downstream) and GPS coordinates (NAD83). The ambient coordinator will provide a sampling schedule for the upcoming water year to MEL and the flow group. The flow group will identify stations where flows may not be available.</td>
</tr>
<tr>
<td>Late August</td>
<td>Ambient regional staff will submit the final list of basin stations directly to stakeholders and, via the ambient coordinator to the flow group and EAP managers. Ambient regional staff will indicate the availability of flows at stations where flows are not expected.</td>
</tr>
<tr>
<td>Early September</td>
<td>Ambient staff will plan the new water year run. Enter day/order/lab number information, parameters for each station, the coming year's sampling schedule, etc., into a temporary database, complete run directions, etc.</td>
</tr>
<tr>
<td>Mid September</td>
<td>Database administrator will submit required reports to MEL.</td>
</tr>
<tr>
<td>--------------</td>
<td>-----------------------------------------------------------</td>
</tr>
<tr>
<td>Late September</td>
<td>Ambient staff must enter September field data on time (the Thursday after the run). After the last run is entered, the database administrator will switch the database over to the new water year's schedule.</td>
</tr>
<tr>
<td>October 1</td>
<td>New water year begins.</td>
</tr>
</tbody>
</table>

[NOTE: I am defining "ambient regional staff" as the four ambient staff responsible for each of the four Ecology regions (Bill, Chris, Dan, and Craig). "Ambient staff" includes the Western Run person (Troy). I am both the "database administrator" and the "ambient coordinator," for lack of a better term.]

**Sampling Design**

Our standard monitoring design consists of monthly sampling for the constituents listed in the table, below. We are usually willing to collect additional constituents when the analysis is funded by a stakeholder.

Our funding is sufficient to sample a total of 82 stations (plus quality control samples). We have divided these into 62 long-term stations that we monitor every year and 20 basin stations that can change from year to year. If logistics allow, we are usually happy to monitor additional basin stations, provided a stakeholder funds the analyses. (Lab analyses for standard constituents at one station for a year costs $1,320.) We may also establish a series of additional stations in cases where a stakeholder has been able to fund staff time and travel, as well as analyses.

<table>
<thead>
<tr>
<th>Standard Constituents</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ammonia</td>
</tr>
<tr>
<td>conductivity</td>
</tr>
<tr>
<td>fecal coliform bacteria</td>
</tr>
<tr>
<td>flow (at most stations)</td>
</tr>
<tr>
<td>metals &amp; hardness (bimonthly, 12 stations)</td>
</tr>
</tbody>
</table>

**Basin Station Selection Criteria**

Ideally, basin stations will be selected with the consensus of all stakeholders. But if there are too few stations identified by early July, ambient monitoring staff may need to identify additional stations. Conversely, if too many stations are identified, ambient staff will need to prune the list or get commitments from stakeholders to fund the extra stations. Ambient staff will also need to decide if proposed stations meet our basic requirements.

**Basin Station Selection Criteria**
- Category "5" (303(d) listed. See [http://www.ecy.wa.gov/programs/wq/303d/index.html](http://www.ecy.wa.gov/programs/wq/303d/index.html))
- Support Ecology’s permitting system (See [http://ecydbleyorwq06/webwplcs](http://ecydbleyorwq06/webwplcs). Click on Permits/Permit Dates Report. You can filter on permits in your region coming due in the next year or two. You can’t see the receiving water, though.) Also: Contact Gary Bailey (Permit Writers Workgroup) or Nancy Kmet (through Dave Hallock).
• Never been there, suspect impairment (See http://www.ecy.wa.gov/programs/eap/fw_riv/rv_main.html#4)
• Never been there, need to broaden coverage (especially in supplemental spawning areas)
• Supplement local efforts
• Pre-TMDL
• Contribute to an active TMDL
• Post-TMDL/effectiveness

Basic Requirements
• Safe to park, access bridge/bank, and sample (see http://www.ecology.wa.gov/programs/eap/Safety/FieldOpsandSafetyManual2009.docx, Working near traffic and from bridges, Working in Rivers and Streams, and Fall Protection, among others; remember, you must be able to park and sample outside the fog line.)
• Stream flows in one direction (i.e., no tidal influence)
• Representative samples can be collected (well-mixed, no upstream tributary or other source)
• Active stream flow gage recommended but not required (see https://fortress.wa.gov/ecy/wrx/wrx/flows/regions/state.asp)

Metals Stations
• Permit writers want data upstream of their facilities, even if no problems are expected
• Basin stations where we don’t have data
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## Freshwater Run Checklist

<table>
<thead>
<tr>
<th>Type</th>
<th>C</th>
<th>E</th>
<th>NW</th>
<th>SW</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>250 mL unpres. nutr (brown)</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
</tr>
<tr>
<td>250 mL pres. nutr. (clear)</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
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<tr>
<td>500 mL (general)</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
</tr>
<tr>
<td>1000 mL (TSS)</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
</tr>
<tr>
<td>250 mL FC/Enterococcus</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
</tr>
<tr>
<td>Acid (metals)</td>
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<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
</tr>
<tr>
<td>500 mL Teflon (metals)</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
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<td>Metal Filters Units</td>
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<td>·</td>
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<td>Chlorophyll or other</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
<td>·</td>
</tr>
</tbody>
</table>

### Pre-Run Preparation

- Hotel Reservations
- Sample Tags
- Field Data Report and LAR Forms
- Site Visit forms (for Hlab sites)
- Meter Calibration Log Form
- Yellow Flow Book
- Field Sampling Notification Form
- Contact Person Designation Form
- Run Directions
- Set Calendar “out of office”
- Gas Van
- Submit timesheet, if required

### Meters/Instruments

- Hach Meter
- Conductivity and pH probes
- Thermistor
- Alcohol Thermometer
- Barometer
- Thumb drive and data card to D/L hab station

### Standards

- pH 4, 7 & 10 Buffers (and spares)
- pH 7 QC Buffer (and spares)
- pH Probe Fill Solution
- Conductivity Standard

### Sampling Equipment & Supplies

- Deionized Water
- Stainless D.O. Bucket Sampler
- Fecal Coliform Sampler
- Metals Sampler
- Ropes 1 @ 35 ft. & 2 @ 75 ft.
- D.O. Sample Box
- D.O. Reagents
- Pipettes
- Ice Chests
- 250 mL 10% HCl (and spare)
- Filters
- Filter Apparatus
- Extra batteries 4 - AA, 1 - 9V
- Weighted Measuring Tape
- USGS Keys; flow station key
- Fiberglass Tape

### Van/Safety Equipment

- Yellow Hazard Beacon
- Flashlight
- Tool Chest
- Tire Chains
- Jumper Cables
- Flares or Reflectors
- First Aid Kit
- Foil Blanket
- Orange Vests
- 2 Gallons Drinking Water
- Hand Towels

### Personal Gear

- Rain Gear
- Sun Glasses
- Watch
- Gloves
- Knee Boots
- Extra Clothing
- Hat
- Map/Gazetteer

### Pre-Departure Preparation

- Check Road Conditions
- Acid Wash Filtering Apparatus
- Check Barometer Calibration
- Change pH & Conductivity standards
- Suction ⅛” pH fill solution and refill
- Check Thermistor Calibration
- Calibrate pH meter
- Calibrate Conductivity Meter
- Van binder, gas card, cell phone
- Ice and, if shipping, gel ice

---

1. See bottle order report for current water year (take extras!)
2. Edit at SharePoint and y:\ambient\WY## DOCS#_Run.
3. Enter Observations on Meter Calibration Log Form
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## Attachment C – Laboratory Analyses Required Form

### Laboratory Analyses Required

<table>
<thead>
<tr>
<th>Date</th>
<th>Year</th>
<th>Time</th>
<th>Field Station ID</th>
<th>Manchester Lab Sample Number</th>
<th>Source Code</th>
<th>General Chemistry</th>
<th>Micro</th>
<th>Metals</th>
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<tr>
<td>09/17</td>
<td>12</td>
<td>23A100</td>
<td>011012</td>
<td>X X X X X X X X X X</td>
<td>011012</td>
<td>X X X X X X X X X X</td>
<td>X X</td>
<td>Chehalis R @ Dryad</td>
</tr>
<tr>
<td>09/17</td>
<td>12</td>
<td>24H000</td>
<td>021012</td>
<td>X X X X X X X X X X</td>
<td>021012</td>
<td>X X X X X X X X X X</td>
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</tr>
<tr>
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<tr>
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<td>25F050</td>
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<tr>
<td>09/17</td>
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<td>X X</td>
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</tr>
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<td>12</td>
<td>QAS-3</td>
<td>131012</td>
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<td>131012</td>
<td>X X X X X X X X X X</td>
<td>X X</td>
<td>Quality Control Sample</td>
</tr>
</tbody>
</table>

### Project Officer
- **Name:** David Hallock
- **Phone:** 3604076681

### Sampler
- **Name:** Bill Ward
- **Field Phone #:** __________

### Chain of Custody Record

<table>
<thead>
<tr>
<th>Relinquished by</th>
<th>Received by</th>
<th>Date</th>
<th>Hr</th>
<th>Mn</th>
<th>Comments (temp, preserv, No. of coolers, etc.)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>09/17/12</td>
<td></td>
<td></td>
<td>Shipped __________ coolers.</td>
</tr>
</tbody>
</table>

* RUNS Parameter Group: Turb, TSS, NH3, NO2+NO3, TPN, FCMF.
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### Attachment C-2

**SRM FIELD DATA REPORT FORM**

<table>
<thead>
<tr>
<th>Station</th>
<th>Station Name</th>
<th>Temp (°C)</th>
<th>DO (mg/L)</th>
<th>DO #</th>
<th>Temp (°C)</th>
<th>True pH</th>
<th>Cond (μS/cm)</th>
<th>Press in Hg</th>
<th>Stage Height</th>
<th>ChkBr/Corr.</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>23A160</td>
<td>Chehalis R @ Dryad</td>
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<td></td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>24B090</td>
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</tr>
<tr>
<td>24F070</td>
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<td></td>
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<td></td>
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<td></td>
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</tr>
<tr>
<td>25E060</td>
<td>Abernathy Cr nr mouth</td>
<td></td>
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<td></td>
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<td></td>
<td></td>
<td></td>
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<tr>
<td>25D050</td>
<td>Germany Cr @ mouth</td>
<td></td>
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<td>QAS-1</td>
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<td></td>
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<td>Quality Control Sample</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**WEATHER, etc:**

Bi-lodeate: 10.0/10.0  Thiosulfate: ___/___
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Attachment D
**Meter ID Numbers**
- Meter
- Cond probe
- pH probe
- Thermistor

**Pre-Run Calibration**
- (a) Thermometer (°C)
- (b) Thermistr (°C)
- Van pressure (Pre-cal.)
- Lab pressure
- Correction (a minus b)
- Was that corr. expected? Y/N

**DAY 1**

<table>
<thead>
<tr>
<th>Cond Meter</th>
<th>Standard:</th>
<th>Initial Reading:</th>
<th>Initial Const:</th>
<th>Final Const:</th>
<th>Stnd. reading:</th>
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<tbody>
<tr>
<td>pH Meter</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Slope (#):</td>
<td>(%)</td>
<td>Time:</td>
<td>r²:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>mv @ pH 4:</td>
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<td>Offset:</td>
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</tr>
<tr>
<td>mv @ pH 7:</td>
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<td></td>
<td>QC7:</td>
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<td>Buffer Temp:</td>
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<td>QA Check #2</td>
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<td>Y / N</td>
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<td>QA Check #3</td>
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<td>Y / N</td>
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<td></td>
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**DAY 2**

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</tr>
<tr>
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<td>(%)</td>
<td>Time:</td>
<td>r²:</td>
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<td>Offset:</td>
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<td>Y / N</td>
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**DAY 3**

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<th>Initial Const:</th>
<th>Final Const:</th>
<th>Stnd. reading:</th>
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<tr>
<td>Slope (# &amp; %):</td>
<td>(%)</td>
<td>Time:</td>
<td>r²:</td>
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<td>Y / N</td>
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</tbody>
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**Footnotes**
- a See bottom right corner for expected ranges.
- b If > 45 seconds try 1) cleaning probe, 2) changing cable, 3) new probe
- c If meter pH is ± 0.10 units, recalibrate; if > ± 0.15 units, recalibrate, re-read sample, & "J" data since last calibration.
- d If meter conductivity is > ± 5μs/cm, open and read a fresh standard. If conductivity standard reading is still > ± 5μs/cm, then recalibrate and re-read all samples.

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**Expected Cal. Ranges (and w/in run range)**

- Slope #: -57.5 to -58.8 pH4: 165 to 178 (<5)
- pH4: 165 to 178 (<5)
- Slope %: 98 to 100 pH7: -5 +6 (<5)
- Slope r²: >0.9995 pH10: -168 to +179 (<5)
- Offset: -3 to +8 (<4) CondStd: 0.375 to 0.425 (<0.02)