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Collection, Processing, and Analysis of Stream Samples

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Purpose of this document

The Department of Ecology develops Standard Operating Procedures (SOPs) to document agency practices related to sampling, field and laboratory analysis, and other aspects of the agency's technical operations.

Washington State Department of Ecology

Environmental Assessment Program

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

Version 1.5

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Please note that the Washington State Department of Ecology's Standard Operating Procedures (SOPs) are adapted from published methods, or developed by in-house technical and administrative experts. Their primary purpose is for internal Ecology use, although sampling and administrative SOPs may have a wider utility. Our SOPs do not supplant official published methods. Distribution of these SOPs does not constitute an endorsement of a particular procedure or method.

Any reference to specific equipment, manufacturer, or supplies is for descriptive purposes only and does not constitute an endorsement of a particular product or service by the author or by the Department of Ecology.

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

Revision Date	Rev number	Summary of changes	Sections	Reviser(s)
2/9/2007	1.1	Editorial; formatting	All	Bill Ward
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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) for the collection, preservation, measurement, and analyses of 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. Program SOP methods for Instantaneous Temperature (EAP011), Dissolved Oxygen (EAP023), Metals (EAP029), Fecal Coliform Bacteria (EAP030), pH (EAP031), Conductivity (EAP032), and Invasive Species (EAP070) are also included.

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, Nitrogen Isotope, 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.

3.3 Ecology – Washington State Department of Ecology.

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 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.

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

3.9 MEL – Manchester Environmental Laboratory

3.10 MQO's – Measurement Quality Objectives

3.11 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.

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

3.13 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.

3.14 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, 2015) 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 2011), 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 bottle 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 PHC electrode
5.21 Hach pH 4, 7, & 10 Buffers.
5.22 Hach pH electrode filling solution.
5.23 pH 7 QC buffer (from another manufacturer - not Hach).
5.24 Hach 4-cell Conductivity electrode

Bridge Sampler W/sample bottles

Bacteria Sampler W/sample bottle

5.25 2 –100 µmhos/cm conductivity standards

5.26 2 – 1 L nutrient grab sample bottles[1] (marked up with black permanent ink and MSDS sticker)

5.27 1 – 1 L pH and conductivity grab sample bottle (marked w/red or green permanent ink)

5.28 DO box that has the following supplies:

5.29 300 mL BOD bottles (enough for the Run plus two spares)

5.30 Glass BOD stoppers

5.31 Plastic BOD bottle caps

5.32 3 mL graduated disposable transfer pipettes (one dedicated to each reagent)

5.33 Manganous sulfate monohydrate reagent bottle with MSDS sticker

5.34 Alkali-iodine-azide reagent bottle with MSDS sticker

5.35 Deionized water (DI water) used to rinse sampling bottles and equipment.

5.36 2-750 mL (or 500mL) plastic DI wash bottles

5.37 Metals sampling supplies:

5.38 Hand vacuum pump with hose and pressure gage

5.39 500mL Teflon FEP bottles pre-filled with de-ionized water by the lab

5.40 125 mL narrow mouth poly bottle containing H2S04 preservative for hardness sample disposable 0.45 micron cellulose acetate filter unit (pre-cleaned)

5.41 Small Teflon vials containing 5 ml concentrated nitric acid preservative

5.42 Powder-free vinyl or nitrile disposable gloves

5.43 Baking Soda

5.44 Eyewash Stations

5.45 Digital Camera

6.0 Summary of Procedure

6.1 Annual Run Preparation. This process typically begins in the winter (several months 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 stations[2] (see Attachment A for draft station selection guidance).

[1] These should contain about 200 mL of 10% HCL solution that is replaced every other Run

[2] These are sampled every other month.

6.1.2 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.

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(s) to the courier, and make hotel reservations.

6.2.1 Samplers should always prepare for a Run through the use of 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 should have been 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 Verify that the conductivity (and if needed DO electrode) soaks in tap or DI water (replace water monthly).

6.2.3 Field Work Plan & Contact Person Form.

6.2.3.1 Samplers must complete and post the Field Work Plan & Contact Person Form on SharePoint, along with links to the Run Directions and Run Times documents before beginning a run.

6.2.3.2 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.

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

6.2.3.4 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.*

6.2.4 Make Sample Tags

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

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

6.2.4.3 Rubber band the labeled tags by station and by the planned sampling order.

6.2.5 Print Out Field Forms.

6.2.5.1 Use the River and Stream Management Database to generate the Field Data Report (FDR) and the Lab Analysis Request (LAR) forms.

6.2.5.2 Check the accuracy of the pre-entered information (run date, sampler…) on the forms before printing them (see Attachment C - Example FDR and LAR Forms).

6.3 Day One Procedures

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 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.

6.3.4 Calibrate check the van barometer using the OC digital barometer located in the 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). Adjust the van barometer to be within 0.10 in Hg (inches mercury) when needed (and if possible).

6.3.5 Check the calibration of the long-line thermistor to the NIST reference Onset HOBO U14 digital Thermometer, complete the calibration check log to determine if it can be used, and also note the results on the electrode Calibration Log Form.

6.3.6 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 15°C.

6.3.7 Replace the pH electrode filling solution, rinse electrode with DI water, carefully re-attach the half-filled electrode soaker bottle, plug the fill hole, and store the electrode upright.

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

6.3.9 Clean the conductivity electrode cells with a Q-Tip, rinse area with DI water, and store electrode in DI or tap water.

6.3.10 Verify that the meter times are in Pacific Standard Time and within 3 minutes to a cell phone or to the Naval Atomic Clock time.

6.3.11 Clean the inside of the filter stand 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 it followed by at least a 10 second flush with DI water from the 2 L storage bottle located in the sink.

6.4 Daily Pre-Departure Procedures

6.4.1 pH Electrode Calibration (Hach PHC electrode).

6.4.1.1 Clear the junction. Remove the filling-hole cap, and slowly pull the attached electrode soaker bottle down the electrode in half-inch increments until there is a noticeable drop in the volume of the electrode filling solution.

6.4.1.2 Remove the electrode storage bottle and top off the electrode fill chamber with filling solution.

6.4.1.3 Calibrate electrode following the electrode instruction manual for a three-point calibration (Note: Hach 4, 7, and 10 buffers must be used).

6.4.1.4 Check the calibration accuracy by reading the QC7 buffer.

6.4.1.5 Record all the calibration information on the calibration sheet. Then reattach the electrode storage bottle and store the electrode upright.

6.4.2 Conductivity Electrode Calibration (Hach CDC electrode).

6.4.2.1 Rinse the electrode with DI water and set it in fresh 100 umhos/cm conductivity standard. *Note: the conductivity standard is easily contaminated. Keep it tightly capped and avoid diluting it with DI or stream sample water. Also note: the accuracy of freshly opened standard can be affected if unused for over 15 days.*

6.4.2.2 Check the meter settings to ensure the meter reads in the non-linear function (nLF) mode for temperature compensation and the reference temperature setting is 25˚C.

6.4.2.3 Measure the 100 standard. If the result is within the acceptable range of ± 2 umhos of the standard (>98 and <102), then record the initial result and cell constant, sample ID number, and skip the following calibration steps. If the result is beyond the acceptable range, then remeasure a freshly opened standard. If this next result is beyond the acceptable range, then follow the calibration steps below.

6.4.2.4 Calibrate the electrode according to the electrode instruction manual.

6.4.2.5 Record the conductivity standard concentration, the electrode ID number, the initial and final cell constants, the sample ID number, and any other required information on the Electrode Calibration Log Form (see Attachment D).

6.4.2.6 Store the conductivity electrode in DI, tap, or stream sample water at all times. (Do not store with the pH electrode).

6.4.3 Pre-Sample Collection Preparations.

6.4.3.1 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.

6.4.3.2 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.

6.4.3.3 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.

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

6.4.3.5 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.

6.4.3.6 Secure a bacteria sample bottle in the bacteria sampler.

6.5 Sample Collection Procedures.

6.5.1 Deploy the Long-line thermistor (LLT) electrode and if warranted do a stream height reference point (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 electrode 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. Also note: A sample bottle may be added to the bridge sampler through the use of a rubber tie down strap.*

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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.

6.5.3.8 Memorize or record the water temperature, push the meter hold button to lock the result, retrieve the thermistor electrode, 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.

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 any 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 electrode 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 (or within view of driver).*

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.

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, push the meter hold button, or record the water temperature, and retrieve the thermistor electrode.

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

[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.

6.5.5.3 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.

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, push the meter hold button, or record the water temperature, and retrieve the thermistor electrode.

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] 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.

6.6.4 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 exact 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 electrodes 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 electrode fill hole and carefully remove the pH electrode soaker bottle, rinse the electrode with DI water, and put it in the pH measurement sample cup. Turn on the meter and gently stir the pH electrode 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[8]. 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 fill a Hardness sample bottle 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*.

[8] Make sure there are a few drops of acid in each bottle.

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 electrode 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 gradual sample temperature changes may alter the pH or prolong the time it takes to obtain a stable result.*

6.6.16 If a Hach PHC281 electrode initial measurement is < 6.5 pH units, then clear the junction and remeasure the sample (not a current method for other Hach pH electrodes).

6.6.17 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[9].

6.6.18 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.

6.6.19 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 < 0.5, 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.

6.6.20 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).

[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.

6.6.21 ***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*!**

6.6.22 ***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.***

6.6.23 ***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.***

6.6.24 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.

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

6.6.26 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.

6.6.27 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.

6.6.28 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.

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

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

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

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

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

6.6.34 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[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.

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 Completely loosen the bottle cap while it is kept on the bottle opening. Gently lower the sampler lifting arm hose-clamp over the cap and then tighten the clamp to secure it.

6.7.3.6 Attach the sampling rope.

[10] Metals samples are collected at a few selected stations every other month.

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

6.7.3.8 Check to make sure the sampler lifting arm can move up freely.

6.7.3.9 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.*

6.7.3.10 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.*

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

6.7.3.12 Hold the bottle cap down on the bottle opening, carefully loosen the lifting arm hose-clamp, screw on the cap until it is tight, remove and tag the bottle, and place the bottle back in the Ziploc bags that it shipped in.

6.7.3.13 Repeat the procedure to obtain a second metals sample.

6.7.3.14 Put on a pair of gloves from the special Hg metals bottle bag and repeat procedures 6.7.3.1 – 6.7.3.4 to secure the bottle in the sampler.

6.7.3.15 Remove the gloves and follow procedures 6.7.3.5 – 6.7.3.10 to collect the sample.

6.7.3.16 Put on another pair of the gloves, hold the bottle cap down on the bottle opening, carefully loosen the lifting arm hose-clamp, screw on the cap until it is tight, remove and tag the bottle, and place it back in the Ziploc bags that it was shipped in. ***Note: Do not acidify this sample.***

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

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 procedure 6.7.4.1 – 6.7.4.6 to obtain a second metals sample.

6.7.4.7 Put on two pair of gloves from the special metals bottle bag, remove the cap, collect the New Hg Metals sample, remove one pair of the gloves, replace the cap, tag the bottle with the new Hg tag, and place it back in the Ziploc bags it shipped in. ***Note: Do not acidify this sample or set the cap down.***

6.7.4.8 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.

6.7.5.7 Repeat the procedure to obtain the second metals sample.

6.7.5.8 Put on two pairs of gloves from the special new Hg metals bottle bag, remove the cap, collect the New Hg Metals sample, remove one pair of gloves, replace the cap, tag the bottle with the new Hg tag, and place it back in the Ziploc bags that it shipped in. ***Note: Do not acidify this sample or set the cap down.***

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

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[11] 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.

6.8.11 Field Processing – New Hg Metals

6.8.11.1 Put it in the large Ziploc bag that contains the: tagged total metals sample, dissolved metals sample, and the empty Teflon bottle.

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[12].

[11]Any peristaltic pumps used for metals filtering must be checked to verify that the lowest setting will not create a vacuum greater than 20PSI.

[12] 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_-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.

6.9.1.1 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). ***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.***

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

6.9.2.2 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.

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

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

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

6.9.2.6 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[13].

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.

[13] One Metals blank is collected per Run per year.

6.9.3.2 Return to the van and follow the Dissolved Metals processing procedure (see procedure 6.8.10) 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 and place it on ice.

6.10 End of Day QC Procedures.

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

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

6.10.3 Check the calibration of the conductivity electrode. Record the result on the electrode calibration form Form. If the conductivity measurement is not within 5 µmhos/cm of the standard then troubleshoot the meter 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 Use a pen to fill out the Lab Analysis Required Form (LAR). The information required includes: sample times, field contact phone number, relinquished by, relinquish time, relinquished to "Walk in cooler", if necessary, number of coolers, and any helpful comments. Initial and date any changes made to the form in ink.

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. Put a tag on the handle of the cooler indicating it goes to MEL to make identification easier.

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.*

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[14].

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.*

6.13.6 Contact the lab courier with any changes to the planned air shipment and the **air waybill number** (already noted in the forwarded airline confirmation) after the cooler(s) have been shipped.

[14] Dry ice freezes Gel-Ice colder and some samples could be frozen if several of them are used.

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 Move the meters, electrodes, a filled DI water wash bottle, pH buffers, and conductivity standard 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 Laboratory Analysis - *Note: Save all Winkler chemical waste resulting from any analysis (in a pail or bucket) for treatment (See 6.15.7 Winkler Waste Treatment and Disposal Methods). Also Note: the titration procedures are also documented in a Winkler training video in the Training area of EAP SharePoint.*

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 empting 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[15] 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.

[15] This is a slight modification of azide modification method presented in SM 20th Edition, 1998, which calls for the addition of 1 mL of manganous sulfate and alkali-iodine azide instead of 2 mL. The excess reagents are accounted for by using 203mL volumetric flasks rather than 201mL flasks.

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[16] and it should be sharp and distinct[17]. 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 needs 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[18]. 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[19] if the back-titration requires fewer than or equal to 8 drops and record the result without qualification[20]. 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[21].

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.

[16] The volume of sodium thiosulfate used to titrate 203 mL of a sample equals the DO of the sample in mg/L.
[17] If the end point was not sharp and distinct or the sample contains purple flakes, then replace the starch solution (it may have gone bad – this is rare). Record the result with a "J" qualification to indicate the result is an estimate and note that the starch was bad and was replaced on the Field Data Report Form.
[18] The mL of Sodium thiosulfate used to analyze a 200mL sample with this method is equal to the DO concentration in mg/L.
[19] The corrected final value is the final value - (number of drops used x 0.05 mg/L). For example, if 8 drops were used and the final value was 10.3 mg/L, then the corrected final value is 9.9 mg/L (10.3 mg/L - (8 x 0.05 mg/L or 0.4 mg/L)).
[20] Justification: Our MQOs specify 0.2 mg/L; 8 drops is equivalent to 0.4 mg/L which leaves a generous allowed error of 50% for miscounting, imprecise drop size, etc. to still be within MQOs.
[21] Justification: Results with a potential error of 50% of 1 mg/L, or 0.5 mg/L, should not be recorded at all.

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.

6.15.5 Record the volume of the sodium thiosulfate needed for each normality check on the field notebook or worksheet and on the titration log located next to the titration station (The average of the two normality checks is used as a correction factor for the field data). *Note: These normality checks should be very close, within 0.2 mL. If they are not, then do at least two more until you have three consecutive results (within 0.2 mL of each other) to use to calculate a correction factor*.

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

6.15.5.2 Eliminate air from the tip of the Potassium Biiodate bottle-top dispenser to ensure it dispenses a 10.0 mL.

6.15.5.3 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,

6.15.5.4 Rinse the inside flask wall with starch solution to ensure that all of the Potassium Biiodate is in the titrated solution, and eliminate Sodium Thiosulfate drops/residue from the outside of the refillable burette tip and tube connection.

6.15.6 Correcting Titration End Point Results with Normality Check (NC) Results[22].

6.15.6.1 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.

6.15.6.2 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).

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

[22] The Ambient database automatically does this.

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

6.15.6.5 Corrected Result = (measured DO × CF) = (11.5 mL × 1.01CF) = 11.6 mL. *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.*

6.15.7 Waste Treatment Procedures. Follow procedure depicted in Figure 4 below, record final pH on the Winkler Waste Treatment Record (Attachment E), and rinse the treated waste down the drain with copious amounts of tap water.

Figure 4. Winkler Waste Treatment.

6.15.8 Lab Clean Up Procedure

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

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

6.15.8.3 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.

6.15.8.4 Thoroughly rinse the used flasks and stir bar(s), and give them a 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 the conductivity electrode with DI water.

6.16.4 Store the meter(s), electrodes, pH buffers, and conductivity standards in a warm and dry area in the regional lab or operation center.

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

6.16.6 Empty the van of trash and vacuum it out.

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

6.16.8 If warranted, get the van oil changed.

6.16.9 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.

6.16.10 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 webpage www.ecy.wa.gov/programs/eap/fw_riv/.

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

10.1 APHA (American Public Health Association), 2015. Standard Methods for the Examination of Water and Wastewater-. No: 4500-O C. Winkler Method, Azide Modification, American Public Health Association, 22nd Edition. Washington D.C.

10.2 Ecology, 2015. Environmental Assessment Program Safety Manual. Washington State Department of Ecology. Olympia, WA.

10.3 Ecology, 2011. Chemical hygiene plan and hazardous materials management plan. Washington State Department of Ecology. Olympia, WA

10.4 EPA, 1996. Method 1669, Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels. Washington, D.C.

10.5 Hallock, D., 2007. Addendum to Quality Assurance Monitoring Plan: Stream Ambient Water Quality Monitoring: Correction of Objectives, Responsibilities, and Addition of Analytes. Washington State Department of Ecology, Olympia, WA. 11 pp. Publication No. 03-03-200Add2. https://fortress.wa.gov/ecy/publications/summarypages/0303200addendum2.html

10.6 Hallock, D. and W. Ehinger, 2003. Quality Assurance Monitoring Plan: Stream Ambient Water Quality Monitoring. Washington State Department of Ecology, Olympia, WA. 27pp. Publication No. 03-03-200. https://fortress.wa.gov/ecy/publications/summarypages/0303200.html

10.7 Hallock, D., 2010. River and Stream Water Quality Monitoring Report for Water Year 2010. Washington State Department of Ecology, Olympia, WA. 40 pp. + appendices. Publication No. 11-03-037. http://www.ecy.wa.gov/biblio/1103037.html

10.8 Parsons, J., D. Hallock, K. Seiders, W. Ward, C. Coffin, E. Newell, C. Deligeannis, K. Welch. 2012. Standard Operating Procedures to Minimize the Spread of Invasive Species. EAP_SOP - 070.

Attachment A – Draft Station Selection Guidance

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

Date	Task
June	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.)
Late July	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.
August	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 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.
Late August	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.
Early September	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.
Mid September	Database administrator will submit required reports to MEL.
Late September	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.
October 1	New water year begins.

[*NOTE: Ambient regional staff includes all ambient staff responsible for the Runs in each of the four Ecology regions (currently six Runs) and the database administrator/coordinator.*

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.

Standard Constituents		
Ammonia	nitrate plus nitrite	phosphorus, total
conductivity	nitrogen, total	suspended solids, total
fecal coliform bacteria	oxygen	temperature
flow (at most stations)	ph	turbidity
metals & hardness (bimonthly, 12 stations)	Phosphorus, soluble reactive	

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 www.ecy.wa.gov/programs/wq/303d/.)
- Category "2" (Needs more data. See www.ecy.wa.gov/programs/wq/303d/.)
- Support Ecology's permitting system (See https://fortress.wa.gov/ecy/wqreports/public/f?p=110:300:3631029519474507:::::.)
- Never been there, suspect impairment (See www.ecy.wa.gov/programs/eap/fw_riv/)
- 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 EAP Safety Manual, 2012), 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

Attachment B – Run Checklist

Pre-Run Preparation

___ Hotel Reservations
___ Pre-Booked Air Shipment
___ Field Work Plan in SharePoint
___ Sample Tags
___ Meter Calibration Log Form
___ Lab Analysis Report Forms
___ Field Data Report Forms
___ Waterproof Field Notebook
___ Run Directions Binder
___ Van Binder and keys
___ Cell Phone
___ Gas Van
___ Sample Bottles
___ Submit Timesheet
___

Standards & Sampling Supplies

___ pH 4, 7, & 10 Buffers
___ pH Probe Filling & Storage Solutions
___ Conductivity Standard
___ Filters
___ Pipettes
___ Deionized Water
___ D.O. Reagents
___ 250 mL 10% HCl
___ Disposable Powder Free Gloves
___ Soak Probes in Tap Water
___ Tape
___ Scissors
___ Bags for small bottles
___ Clipboard
___ Baking Soda
___ Flagging

Sampling Equipment

___ Gage & Gate Keys
___ Stainless D.O. Bucket Sampler
___ Fecal Coliform Sampler
___ Metals Sampler
___ Weighted Measuring Tape
___ Ropes
___ D.O. Sample Box
___ Filter Apparatus
___ Hand Vacuum Pump with Hose
___ Map/Gazetteer/Thomas Guide pages
___ Gloves
___ Knee Boots
___ Rain Gear

Van/Safety Equipment

___ Yellow Hazard Beacon
___ Flares or Reflectors
___ Tire Chains
___ Jumper Cables
___ Tool Chest
___ Flashlight
___ Shovel
___ Safety Vests
___ Hardhats
___ First Aid Kit
___ Foil Blanket
___ Emergency Eyewash
___ Hand Towels
___ Hand Truck?
___ Step Ladder?

Personal Gear

___ Sun Glasses
___ Watch
___ Extra Clothing
___ Hat
___ 2 Gallons Drinking Water

Meters/Instruments

___ pH Electrode
___ Conductivity Electrode
___ Long-line Thermistor
___ Barometer
___ Camera (and GPS?)
___ Meter Manuals
___
___

Pre-Departure Preparation

___ Check Road Conditions
___ Acid Wash Filter Apparatus
___ Calibrate Check Barometer[1]
___ Change pH Probe Solution
___ Clean conductivity cells
___ Change pH & Conductivity standards
___ Calibrate Conductivity Electrode[1]
___ Calibrate pH Electrode[1]
___ Check Thermistor Calibration[1]
___ Load Ice Chests, Gel-Ice, and Ice
___
___
___

[1]Enter Observations on Meter Calibration Log Form

Sampler: Bill Ward

SRM FIELD DATA REPORT FORM

Date: 9/17/2012

Station	Station Name	Time	Temp ºC	DO mg/L	DO #	Temp / pH	True / Meter	Cond uS/cm	Press in.Hg	Stage Height	ChkBr/ Corr.	Comments
23A160	Chehalis R @ Dryad											
24B090	Willapa R nr Willapa											
24F070	Naselle R nr Naselle											
25F060	Mill Cr nr mouth											
25E060	Abernathy Cr nr mouth											
25D050	Germany Cr @ mouth											
QAS-1	Quality Control Sample											
QAS-2	Quality Control Sample											

WEATHER, etc:

Bi-Iodate: 10.0/10.0 Thiosulfate: _____/_____

Laboratory Analyses Required

Project Name: **SRM - 1209005**

SIC: **DWF03** Program: **EAP**

Send Results to: **David Hallock** Mail Stop: **47600** ☑ Monitoring Reference QAPP: **0503202 and Addendum**

Date Year 12 mm dd	Time (Military, e.g., 1625)	Field Station ID	Manchester Lab Sample Number	Matrix Code	Source Code	General Chemistry: RUNS parameter group*	Conductivity	Turbidity	Total Suspended Solids	Ammonia	Nitrate-Nitrite	Total Pers. Nitrogen	Total Phosphorus TP8	Orthophosphate, Mix 11	TOC	DOC	Chlorophyll	Suspend. Sed. Conc.				Micro: Fecal Coliform (MF)				Metals: Met Amb parameter Tot	Met Amb Parameter Dis	Hardness	Total Mercury Only			
0917		23A160	- 01	10	12	X	X	X	X	X	X	X	X	X		X						X										Chehalis R @ Dryad
0917		24B090	- 02	10	12	X	X	X	X	X	X	X	X	X		X						X										Willapa R nr Willapa
0917		24F070	- 03	10	12	X	X	X	X	X	X	X	X	X		X						X										Naselle R nr Naselle
0917		25F060	- 04	10	12	X	X	X	X	X	X	X	X	X	X	X		X				X										Mill Cr nr mouth
0917		25E060	- 05	10	12	X	X	X	X	X	X	X	X	X	X	X		X				X										Abernathy Cr nr mouth
0917		25D050	- 06	10	12	X	X	X	X	X	X	X	X	X	X	X		X				X										Germany Cr @ mouth
			-		10	12																										
			-		10	12																										
0917		QAS-1	- 12	10	12	X		X	X	X	X	X	X	X	X	X		X				X										Quality Control Sample
0917		QAS-2	- 13	10	12					X	X	X	X	X	X	X																Quality Control Sample

Project Officer

Name: **David Hallock**

Phone: **3604076681**

Sampler

Name: **Bill Ward**

Field Phone #:_____

Chain of Custody Record					
Relinquished by	Received by	Date	Hr	Mn	Comments (temp, preserv, No. of coolers, etc.)
		09/17/12			Shipped _____ coolers.

Comments:_____

_____

_____

* RUNS Parameter Group: Turb, TSS, NH3, NO2+NO3, TPN, FCMF.

Attachment D- Electrode Calibration Log Form

Electrode Calibration and QC Checks				Draft version 1.1		
Date (dd/mm/yy)	Time	Run		Sampler(s)		
Thermistor #	a) HOBO Logger/Thermometer (º			Van pressure (Pre-cal.)		
Meter #	b) Thermistor (ºC)			Lab pressure		
pH Elect. #	Correction (a minus b)			LDO Electrode pressure		
Cond Elect. #	Was that corr. expected?	Y/N		Adjusted?	Y/N	
LDO Elect. #	Comments:					

pH Electrode Calibration[a]						Calib. Millivolts (Mv)				
Date/Time	Slope	%	Offset	r2	Temp °C	4	7	10	QC 7 True	QC7 reading

LDO Electrode	Pre calibration[b]			Calibration[c]				Post calibration		
Date/Time	1) Expected mg/L (USGS)	2) Reading mg/L	Diff of 1 & 2d	Slope	Offset	Temp	inHg	1) Expected mg/L (USGS)	2) Reading mg/L	Diff of 1 & 2

Conductivity Electrode Calibration[d]						Temp˚C	Hach pH7	Hach pH10	NIST pH7	NIST pH 10
Date/Time	Initial Cell Constant	Initial Reading	Final Cell Constant	MV W/O temp corr.	Final Standard Reading					
						8	7.08	10.19	7.07	10.21
						10	7.07	10.17	7.06	10.18
						12	7.06	10.14	7.05	10.16
						14	7.05	10.12	7.04	10.13
Daily Electrode QC Check Day #1[e]						16	7.04	10.1	7.03	10.11
Date:	**Sample ID**	**Time**	**True pH**	**Reading**	**Recal?**	18	7.03	10.08	7.02	10.08
pH QC Check #1					Y/N	20	7.02	10.05	7.01	10.06
pH QC Check #2					Y/N	22	7.01	10.03	7.01	10.04
pH QC Check #3					Y/N	24	7	10.01	7	10.02
pH QC Check #4					Y/N	26	7	10	6.99	10.01
End day QC check Cond Stand 100, Reading _____					µS/cm	Comments				
Daily Electrode QC Check Day #2[e]										
Date:	**Sample ID**	**Time**	**True pH**	**Reading**	**Recal?**					
pH QC Check #1					Y/N					
pH QC Check #2					Y/N					
pH QC Check #3					Y/N					
pH QC Check #4					Y/N					
End day QC check Cond Stand 100, Reading _____					µS/cm					
Daily Electrode QC Check Day #3[e]										
Date:	**Sample ID**	**Time**	**True pH**	**Reading**	**Recal?**					
pH QC Check #1					Y/N					
pH QC Check #2					Y/N					
pH QC Check #3					Y/N					
pH QC Check #4					Y/N					
End day QC check Cond Stand 100, Reading _____					µS/cm					

Footnotes

[a] *See bottom right corner for expected ranges.*

[b] *See O_2 Solubility Table below.*

[c] *Recalibrate if difference is ± 0.1 mg/L.*

[d] *If electrode conductivity is >± 5µs/cm, recalibrate, re-read sample, & "J" data since last calibration.*

[e] *If electrode pH is >± 0.10 units, recalibrate; if > ± 0.15 units, recalibrate, re-read sample, & "J" data since last calibration*

LDO Electrode QC Check Post Run							Slope #: -57.5 to -58.8 (<0.7)	pH4: 165 to 178 (<5)
Date/time	Sample ID	Van Pressure	Meter Pressure	1) Expected mg/L	2) Reading mg/L	Diff of 1 & 2	Slope %: 98 to 100	pH7: -5 to +6 (<5)
							Slope r^2: >0.9995	pH10: -168 to +179 (<5)
							Offset: -3 to +8 (<4)	Cond: 0.375 to 0.425 (<0.02)

Winkler Waste Treatment Record

Name	Date	Volume	Initial pH	pH after treatment

Measure and record waste volume and initial pH. Then sprinkle in about two tablespoons of Baking Soda per ½ gallon (or one scoop), stir it to mix, wait about five minutes, and then re-measure and record the final pH.