

**Pollution Identification and Correction
Clean Water District: Trends Monitoring Project**



Standard Operating Procedures

**Created March 2015
Revised May 2021**

Purpose of this document

As the lead project entity, Clallam County division of Environmental Health Services 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. This SOP was created Clallam County Streamkeepers with the help of all PIC project partners.

Contact Information

Joel Green, Streamkeepers Coordinator (Trends Monitoring Program Lead)

Clallam County Department of Community Development

jgreen@co.clallam.wa.us

Streamkeepers@co.clallam.wa.us

360-417-2281

Heather Watts (Project Manager)

Clallam County Environmental Health Services

hwatts@co.clallam.wa.us

360-417-2415

Sue Waldrip (EH Lab Manager)

Clallam County Environmental Health Services

swaldrip@co.clallam.wa.us

360-417-2334

Jennifer Garcelon

Clallam County Environmental Health Services

jgarcelon@co.clallam.wa.us

360-417-2347

SOP Revision History

Author	Date	Section	Summary of Changes
Ed Chadd	March 2015	All	New SOP
Ed Chadd	February 2018	Unknown	Unknown
Ed Chadd	April 2019	Unknown	Unknown
Heather Watts	March 2021	All	Reformat
Heather Watts	May 2021	Post-sampling	Nutrient shipping details + equipment/supplies

* Original version by Ed Chadd, SK Coordinator & QA Officer until 2019

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Pre-Sampling

Complete this section 2 weeks in advance of the sampling event:

- The Project Coordinator determines sampling dates in consultation with the Project Manager, Lab Managers, and field team. Select dates at the end of the preceding year. Send copy of schedule to landowners.
- Check tides at <https://www.tides.net/washington/769/>. If possible, in order to avoid tidal influence, schedule such that all lower sites are sampled when tides are < 3 ft. (The critical ones are Cassalery 0.0, Cooper 0.1, and Meadowbrook 0.1.) Please make a note in the comments section of the data sheet when the site is not sampled due to tidal influence. This ensures it will be noted in the database as a failed-attempt observation.
- Check with labs& landowners if necessary.
- Poll for volunteer/staff availability; determine team, meeting time/place.
- Reserve county vehicle(s) if necessary.
- Assemble all equipment/supplies (see below).

Sampling Day

- Take all equipment & supplies (below)
- Place ice or ice packs in each cooler at least equivalent to the volume of sample to be collected (more may be needed in the summer)

Equipment/Supplies (make note of any supplies that are running low and notify SK manager to reorder)

PIC box(es):

- Bottles--one for each parameter/site/sample type, minimum 14 for Tier 1, and 12 for Tier 2, plus a few spares
 - Fecal coliform reusable pre-numbered 125 mL collection bottles. *Make sure none of these have duplicate numbers*
 - 500 mL nutrients grab bottles (16), pre-labeled for each site, plus spares.
 - 100 mL nutrients lab bottles (28), 16 narrow and 16 wide mouthed, unmarked; see below under "Laboratory Bottles"
- Foam bottle-holder for nutrients processing (should be in box with 500mL bottles)
- Disposable syringe filters .45 micron, for dissolved nutrients bottles – one for each sample set, plus ample spares;
- Empty Ziploc bags/grocery bags for used syringes, filters, decontaminating probes & pole-ends
- Syringes — separate labeled syringe for each site, plus a spare
- Short form laminated sampling and filtering procedure

PIC bag:

- SAFETY:
 - First Aid kit
 - High-visibility vests to wear at any sites along roads
- SAMPLING:
 - Cafeteria-style tray (for nutrients processing)
 - Clipboard
 - Engineer's measuring tape (in 0.01 ft increments)
 - Fiberglass 100+ ft. tape, with twin weights & rubber bands or ties
 - Disposable gloves to fit any size
 - Box with pencils, sharpener, grease pencils, markers, business cards
 - Purified water (filled day of or day before):
 - 1 wide-mouth 1-L bottle for drawing purified water into syringes

- 1 small bottle with spout for rinsing
- DECONTAMINATION KIT:
 - Stiff scrub brush
 - Spray bottle
 - Hydrogen peroxide (for decontamination—preferred because it won't cause interference in nutrients samples)
 - And/Or, disinfection agents containing quaternary ammonia compounds
 - Plastic bags of various sizes and ties

Paperwork (in PIC binder in PIC bag):

- Forms
 - Tier 1 COC
 - Tier 2 COC
 - Tour cover sheet
- Field Procedures
 - This SOP (PIC sampling procedures)
 - Nutrient collection SOP (<K:\Streamkeepers\Special projects\Dungeness River Management Team\Clean Water District\PIC project\Procedures\Nutrient sample process.docx>)
 - Bottle filling protocol
- Site information
 - Map of sites
 - Site access notes
- Project information
 - PIC QAPP
 - SK QAPP
 - PIC Plan
- Other
 - Noxious Weed Report forms
 - Volunteer hours sheet
 - Permits for any sites requiring them (none as of March 2021)
 - Material safety data sheets (MSDS) for all chemicals used (none)
 - Incident report sheets

Other:

- Multimeter:
 - YSI ProDSS in its kit bag, minus the flow meter & flow parts box, with operating instructions or
 - YSI 85 with probe properly stored, with batteries installed & spares, with operating instructions
 - Bucket (low-flow season only)
 - Sampling pole(s) in sizes that will hold all sampling bottles, plus the water-quality probe
 - Back up purified water — 2 gallon bottles – one probably marked “used first” (see below)(filled day of or day before)
 - Coolers — one for each lab (nutrients, fecals), plus a small one to carry to sites
 - Ice packs — multiple packs for each cooler (6-8 for the large coolers)
 - Bubble wrap or similar to keep bottles from direct contact with ice packs
 - Insulated shipping box for nutrients samples (needed when samples come back to the County Courthouse)
- [Packing tape to wrap coolers prior to shipment—usually supplied by shipper]

Optional:

- Hand sanitizer (use with caution so as to not contaminate samples)
- Camera & photo log sheet or book to reference Site, Photo #, Subject, Photographer—if photos are being taken to document site/sampling locations
- [Binoculars (to read staff gauges from a distance)] – *supplied by a volunteer*

Laboratory bottles:

LABORATORY	ANALYTE(S)	BOTTLE, PREP	COLLECTION/ HANDLING	QUANTITY/ HOLDING TIME
Clallam County Environmental Health Lab	Fecal coliform	Sterilized PP ≥125 mL	Direct grab	100 mL, 8 hr
University of Washington Marine Chemistry Laboratory	Nutrients (dissolved): NO ₃ , NO ₂ , NH ₄ , PO ₄ , Si(OH) ₄	60 mL clear narrow-mouth HDPE	Indirect grab; use filter ; rinse	40 mL, 48 hr
University of Washington Marine Chemistry Laboratory	Total N and P	60 mL clear wide- mouth PP	Indirect grab; direct fill, rinse	40 mL, 7 days

Field Procedures (see “References” below for further details and documentation):

Bottle labeling Bring field-grab bottles to sampling site (large nutrients, fecal, extra in case of contamination). Label nutrient bottles LEGIBLY with permanent marker or grease pencil (preferred) to match number on fecal bottle. Cross out other markings/numbers on the nutrient bottles. *Make sure the number assigned is not one that you have already assigned at an earlier visit.* If you are doing replicates or blanks at this site, you will have two sets of bottles, each set numbered with the number on that set’s corresponding fecal bottle. Leave the small nutrients lab bottles at the car. Bring the larger grab bottle back to the car to process nutrient samples to reduce ground contamination.

Sample collection

- Wear high-visibility vests whenever sampling alongside a road.
- In general: follow Streamkeepers’ Grab-Sample protocol; use a sampling pole when possible; take a small cooler with you to keep the samples cold if there’s any distance to cover.
 - If the site has a noticeable layer of “gunk” on top, you can try to push it aside with the bottom of the bottle before dipping the bottle straight down into the water. Note any surface gunk on the data sheet.
 - At **tidally influenced locations**, sample from as shallow a depth beneath the surface as possible, to target the freshwater component of the water column.
- Collect the nutrients field-grab bottles first in order to get them back to the car for processing. Rinse the bottles and caps once with sample water prior to filling.
- **Replicates:** Repeat collection procedures for replicates. Indicate “R” on data sheet if not already indicated.
- **Blanks:** Process at the car

Water level Record water level per site notes, either:

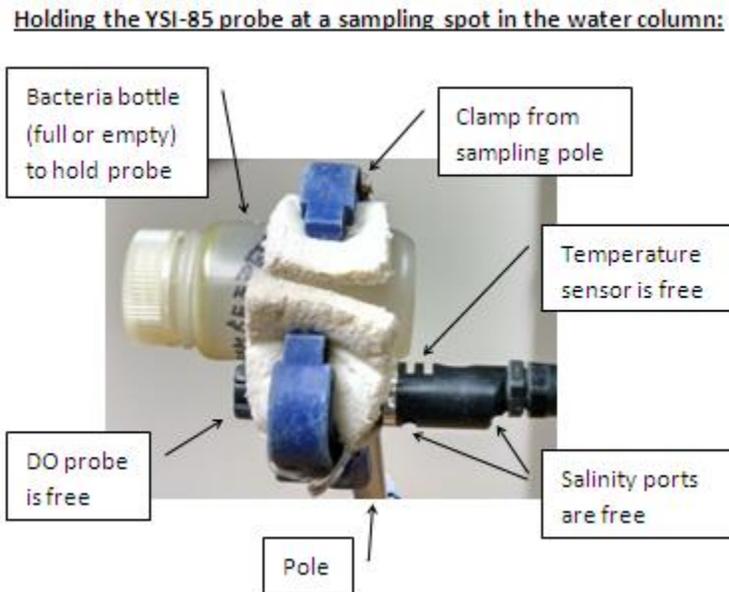
- Reading stream-gage height (GH)
- Measuring top-down (TD) from designated benchmark (this will be a negative number)
- Estimating (EST) flow per a method (floating object, bucket) in the Streamkeepers Handbook (see References)

Water-quality measurements Probe (or thermometer if only collecting temperature data) should be as close as possible (both location and depth) to where you are collecting your grab samples. For the ProDSS meter:

- If collecting in deeper, slower water, dangle the probe from the cable at the end of the sampling pole.
- If collecting in shallow water, set it between clean rocks in mid-column.

- Using the sampling pole is often preferable to standing in the stream. You can give semi-neutral flotation to the bulkhead on the end of the pole by sticking a piece of wood (a.k.a. “The Whale”) in with it.

Here is an example of semi-neutral flotation for the YSI-85 probe:



If water level is too low to deploy the probe, collect water in a bucket and keep the bucket down in the water. If you work quickly, you should be able to get accurate readings.

Comments Note any activity upstream of the sampling site that might influence sampling results: pets, livestock, wildlife tracks, septic smells, kids playing in the creek, vehicle tracks, plowing, compost application, etc.

Anti-Contamination Procedures (more details in the Streamkeepers Handbook):

Invasive Species It is important to avoid potential introduction of invasive species. Avoid entering the water, and avoid touching the bottom with poles or probes. Anything that enters the water needs to be treated as follows when leaving the site:

- Thoroughly inspect whatever has contacted the water and use a brush and DI water to remove any sign of dirt, debris, or organisms. If you suspect pathogenic compounds in the water, rinse even more thoroughly.
- If you're not sure you've removed everything or have felt boot bottoms, you may use the cleaning agent (Formula 409, etc.) provided with your decontamination kit:
 - **NOTE: The use of quaternary ammonia compounds might contaminate your nutrients samples and blanks. This can cause the data to be downgraded. In addition, you may not use quaternary ammonia compounds on any electronic probe—they may cause damage. The use of hydrogen peroxide is preferred.**
 - Thoroughly spray and then enclose the potentially-contaminated items in a plastic bag while traveling to your next site:
 - At least 10 minutes for quaternary ammonia compounds
 - At least 15 minutes for hydrogen peroxide
 - Thoroughly rinse the items (with the purified water supplied with your kit) away from the stream before sampling at the next site.

Glove up to process nutrients samples Skin products can contaminate nutrients samples easier than you might think. Wear clean, disposable gloves when processing nutrients samples.

Pouring from nutrients grab bottle Cap and invert field-grab bottle three times to mix sample.

Non-filtered nutrients lab bottle (wide mouth) Following instructions above, pour ~8 mL of sample into lab bottle. Cap, rinse, and discard. If there is any question about the sterility of the lab bottle, rinse twice more. Then re-agitate the field-grab bottle and fill the lab bottle.

Filtered nutrients lab bottle (narrow mouth)

- Check purified-water bottle: Inspect the bottle from which you will be filling your syringe for clean rinses. If you see any signs of contamination inside, shake and discard the contents, then rinse 3x with ~100 mL of purified water from a clean jug before refilling.
- Purified-water rinse: Find the syringe labeled for your site/sample type in the “clean rack” and draw it full with lab-purified water. Attach a fresh filter. Expel all 60 mL through this filter.
- Syringe/bottle sample rinse: Remove filter temporarily, holding in such a way as not to contaminate it (such as in the upturned purified-water 1-L bottle cap). Fill syringe from agitated field-grab bottle. Use this water to rinse the lab bottle 3x, using 1/3 of the water in the syringe each time.
- Syringe/filter/bottle sample rinse: Fill syringe again from agitated sample bottle. Re-attach filter. Inject at least 8 mL of sample in lab bottle. Cap, rinse, and discard.
- Filtered sample fill: Now fill the bottle, through the filter, with the 50 mL of sample remaining in the syringe.

Field blanks At designated site(s), run an extra set of samples at the car, using lab-purified water instead of stream water, but otherwise following the same procedures as above.

Finishing up nutrients samples

- After filling, cap laboratory bottles tightly and put on ice in cooler.
- Empty the field-grab bottle & rinse with purified water. Discard and then refill with a bit more purified water for transport.

Nutrients lab blanks For troubleshooting purposes, we will sometimes perform these, back at the County Lab. Assign a number different from the already-assigned grab sample bottle numbers, and record these, along with times, on your data sheet.

- **Wet_Lab_Blank:** Follow field blank procedures as above, but fill the grab bottle directly from the water purifier device.
- **Direct_Blank:** No grab bottle; fill lab bottle (totals) or syringe (dissolved) directly from the spigot of the water purifier device.
- **Trip_Blank:** Bottle is filled in the lab prior to the sampling round and then travels in the cooler with the rest of the samples.

Post-Sampling

Processing Procedures

Delivery of fecal samples to Environmental Health Lab

- Samples are generally due by 2:30 PM. Check in with front counter; bring samples in cooler back to the lab. If lab tech is not in, put samples in the fridge. Sign the Chain of Custody (CoC) sheet and make 2 copies: 1 for you and 1 to ship with the nutrient samples (if applicable). Leave original CoC on the lab counter.
- Temperature checks: The Lab director has a temperature gun to check temperatures of sample bottles upon receipt. Test the first and last sample of the day, as well as the sample that was warmest when collected. If all are <10°C, or if the latter show signs of chilling, they are within acceptable limits.

Shipment of nutrients samples to UW Marine Lab Using insulated shipping box for nutrients samples, layer samples in the bottom of the cooler, bubble wrap or other separator to keep samples from freezing, and 5-6 frozen water bottles in cooler. Include a copy of the master data sheet (CoC) on top. Ship from Lincoln St. Station to UW lab by 4:30pm the same day to the following address:

COVID-19 ALTERNATE ADDRESS

UW-Oceanography
Aaron Morello
Marine Sciences Bldg Rm G
Seattle, WA 98195

Ship nutrient samples via priority overnight and charge to CC HHS-Env Health, "PIC Trends Sampling DOE PIC Task 3." Email Aaron Morello at UW Marine Lab to let him know samples are on the way and include the tracking number (aaronm@uw.edu). Route shipping receipt to Celia Thompson in HHS via interoffice mail.

Cleanup Procedures

- Discard used filters and make sure 20 clean filters are available for the next PIC outing. Streamkeepers keeps a stock of these with our PIC supplies. If resupply is needed, inform the project manager.
- Assess whether there are enough clean nutrients lab bottles (18 of both small- and large-mouthed) for the next round of PIC sampling, and notify the Streamkeepers coordinator of quantities of each type of bottle which need to be ordered (as well as syringes, if not available in stock—see below).
- Purified water containers:
 - Gallon jugs: if they've been used:
 - Empty them.

- Rinse 3x with ~200 mL of fresh purified water, capping, shaking vigorously, and then rinsing out the inside of the cap as you shake out the water.
- Refill with fresh purified water.
- Jugs that have not been used: mark “Use First!” with blue tape.
- Smaller purified water containers: if they’ve been used:
 - Empty, fill ~1/4 full and rinse 3x, rinsing the threads and any spouts as described above.
 - Let air dry.
- Field sample bottles and syringes: See EH Water Lab’s SOP at (same document, different drives)
 - J:\eh\water\Streamkeepers\Field Bottle wash and Acid Wash_August 2020.docx
 - <K:\Streamkeepers\Special projects\Dungeness River Management Team\Clean Water District\PIC project\General info\EH Lab PIC Bottle wash and acid wash Aug 2020.docx>

References

Clallam County Health and Human Services. 2016. Addendum 1 to 2015 Quality Assurance Project Plan: Sequim-Dungeness Clean Water District Pollution Identification & Correction, Trends, and Project Monitoring. Port Angeles, Washington, March 2016.

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http://www.ecy.wa.gov/programs/eap/qa/docs/ECY_EAP_SOP_ManuallyObtainingSurfaceWaterSamples_v1_2EAP_015.pdf

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Ohio EPA, Division of Surface Water. 2013. Surface Water Field Sampling Manual –Appendix II. Final Version 4.0, 1/31/13. Section B: Critical Cleaning Protocol for Orthophosphate Syringes.
http://www.epa.ohio.gov/Portals/35/documents/SW_SamplingManual_AppendixII.pdf

Streamkeepers of Clallam County Volunteer Handbook. Current edition. Clallam County Department of Public Works-Roads. Port Angeles, Washington. http://www.clallam.net/streamkeepers/html/volunteer_handbook.htm
[NOTE: This handbook is updated frequently. Revisions are not expected to bias results when compared to earlier data, but rather explain the procedures better, deal with additional special circumstances, and make it less likely that data will be rejected.]

U.S. Geological Survey. 2004. "Cleaning of Equipment for Water Sampling," Chapter A3, *National Field Manual for the Collection of Water-Quality Data*. Ed. Franceska D. Wilde. Version 2.0, 4/2004. Techniques of Water-Resources Investigations, Book 9: Handbooks for Water-Resources Investigations. Reston, VA: U.S. Dept. of the Interior.
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