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ECOLOGY
State of Washington

Standard Operating Procedure EAP094, Version 1.0

Sampling Trace Contaminants Using Continuous Low-Level Monitoring Devices

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Purpose of this document

The Washington State 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.

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Standard Operating Procedure for Sampling Trace Contaminants using Continuous Low-Level Monitoring Devices

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SIGNATURES AVAILABLE UPON REQUEST

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SOP Revision History

Revision Date	Revision History	Summary of changes	Sections	Reviser(s)
4/5/2019	1.0	Complete re-edit of text, revised footer format	All	Arati Kaza
4/19/2019	1.0	Formatting and accessibility updates.	All	Ruth Froese

1.0 Purpose and Scope

- 1.1 This document is the Environmental Assessment Program (EAP) Standard Operating Procedure (SOP) for using Continuous Low-Level Aquatic Monitoring devices (CLAMs) to sample pesticides, herbicides, polyaromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs), pharmaceuticals, personal care products, and other trace contaminants in water.

2.0 Applicability

- 2.1 The CLAM is a submergible water sampling device that filters and concentrates contaminants onto solid phase extraction (SPE) media. It works as a continuous active sampler with a motorized pump to draw large volumes of water on site through a disk cartridge that houses the SPE media. The SPE disk (cartridge + media) is sent to the laboratory for analysis of the contaminant of interest. The volume of water filtered through the disk is measured on site to calculate the concentration of the water sample (Section 6.5).
- 2.2 The target analyte is collected as a time-integrated sample with user-defined deployment times. Typical deployments for EAP studies have been from 12–72 hours. Left unattended, the deployment time is limited by battery life (up to about 36 hours), turbidity of the water, and size of the water collection container. According to the manufacturer, the maximum volume capacity of one CLAM disk is about 100 L. Maximum deployment depth is limited by pumping and tubing capacities.
- 2.3 The use of CLAMs may be suitable for studies involving various organic contaminants (or other extractable analytes using the SPE procedure) expected to be at low levels in freshwater, marine, or man-made aquatic environments. Because the SPE disks tend to clog over time, CLAMs may be more effective in less turbid waters.
- 2.4 The CLAM was commercially introduced in 2007 by the manufacturer Aqualytical® (Visit [Aqualytical's website](#) for more information about the manufacturer). The technology uses the SPE procedure for testing organic analytes, which is a well-established extraction method approved by the Environmental Protection Agency (EPA 3535; EPA, 2007). The device is merely the physical unit housing the SPE media, onto which the analyte is sorbed from the water on site, rather than back in the laboratory. Instead of dealing with the transport and shipping of large volumes of water often need to be collected, the small SPE disk itself is sent to the laboratory for analysis—one of the benefits of the CLAM.
- 2.5 Through experimentation and learning, EAP completed several studies that tested the efficiency of using CLAMs to sample contaminants in rivers and streams of Washington (Table 1). This document outlines the procedures to be followed when using CLAMs to sample for trace contaminants in water for EAP studies.

Table 1. List of EAP studies using CLAMs to sample for contaminants in water.

Author (Year)	Contaminants Sampled Using CLAM	Study Title
Sargeant and Anderson (2010)	Pesticides and Herbicides (GCMS), Carbamates	Skagit-Samish Basin Intensive Surface Water Sampling for Pesticides in Salmon-Bearing Streams, 2009
Era-Miller (2014)	PCBs (EPA 1668, EPA 8082), PBDEs, Dioxins & Furans	Technical Memo: Spokane River Toxics Sampling 2012-2013 – Surface Water, CLAM, and Sediment Trap Results
Hobbs and Friese (2015)	Toxaphene	Pine Creek Toxaphene Source Assessment
Friese and Coots (2016)	PCBs (EPA 1668)	Little Spokane River PCBs: Screening Survey of Water, Sediment, and Fish Tissue
Hobbs and Friese (2016)	PCBs (EPA 1668), DDT	Wenatchee River PCB and DDT Source Assessment
Era-Miller and McCall (2017)	PCBs (EPA 1668), PBDEs	Spokane River PCBs and Other Toxics at the Spokane Tribal Boundary: Recommendations for Developing a Long-Term Monitoring Plan
Hobbs et al. (2019)	PCBs (EPA 1668), PBDEs	Assessment of Low-Level Sampling Methods for PCBs and PBDEs in Surface Waters

3.0 Definitions

- 3.1 Blank – A synthetic sample, free of the analyte(s) of interest (Jones, 1998). The main purpose is to trace sources of inadvertently introduced contamination.
- 3.2 C18 Media Disk – SPE media that uses octadecyl bonded silica (C18) as a sorbent to retain predominantly non-polar compounds. It is the most well-established method for laboratory testing of non-polar compounds, including pesticides, PCBs, dioxins, furans, PAHs, and PBDEs.
- 3.3 Continuous Low-Level Monitoring device (CLAM) – A time-integrative continuous water sampling device that sequesters contaminants onto solid phase extraction (SPE) media by pumping and filtering water on site.
- 3.4 HDPP – The manufacturer-provided SPE disk is made of High Density Polypropylene (HDPP).
- 3.5 Hydrophylic/Lipophilic Balanced (HLB) Media Disk – SPE media that uses a modified styrene polymer as a sorbent to bind a variety of both polar and non-polar compounds. HLB media has been used to sample for many different pesticides, herbicides, pharmaceuticals, personal care products, and emerging contaminants.
- 3.6 Solid Phase Extraction (SPE) – Method by which the analyte of interest is separated and removed from a mixture of compounds by partitioning the compounds between solid (sorbent) and liquid (solvent) phases. It is often used to prepare samples prior to chromatographic analysis (EPA 3535).

4.0 Personnel Qualifications/Responsibilities

- 4.1 Personnel using CLAMs should have the training necessary to operate the equipment competently as well as knowledge and experience in general water quality field sampling procedures.
- 4.2 Typical users of this SOP have an Environmental Specialist or Natural Resource Scientist job classification.

5.0 Equipment, Reagents, and Supplies

- 5.1 A diagram of the CLAM and its components are shown in Appendices A and B. A field checklist template is provided in Appendix C.
- 5.2 CLAM Setup
 - 5.2.1 CLAM device. The details outlined in this SOP refer to an older model of the CLAM that is no longer manufactured by Aqualytical ®. However, the same general field application and data procedures can be used for all older and newer CLAM models. In regard to model-specific installation and operation, the manufacturer's instruction manuals can be referenced.

- 5.2.2 SPE Disk (SPE cartridge with pre-loaded laboratory-conditioned SPE media). The SPE media type to be selected (e.g., C18, HLB) depends on the analyte of interest. The cartridge provided by the manufacturer is made of high-density polypropylene (HDPP). A custom-made stainless steel cartridge should be used if the contaminant of interest is any of the PCB or PBDE congeners. For specifications of compatible stainless steel cartridges, see [ZenPure Stainless Steel Disc Holder Specifications](#). Section 8.2.4 discusses the use of stainless steel cartridges in place of HDPP cartridges due to contamination caused by the HDPP cartridge. Each SPE disk comes with an individual Mylar pouch for storage. The disk should be kept with its original labeled pouch to prevent potential cross contamination.
- 5.2.3 AA batteries (requires 4 per device).
- 5.3 Water Collection
 - 5.3.1 Vinyl tubing (Inner Diameter: 1/8-inch)
 - 5.3.2 Luer lock fittings. Luer lock fittings provided by the manufacturer are made of plastic. Stainless steel luer lock fittings should be connected to the CLAM's ports if the contaminant of interest is any of the PCB congeners.
 - 5.3.3 Water collection container (minimum 50 liter capacity recommended). The container can be customized with a luer lock fitting to connect the vinyl tubing. The exterior of the container should be appropriately marked (e.g., "Do Not Disturb: Research in Progress").
 - 5.3.4 Lock/key to secure the lid and bucket during deployment.
 - 5.3.5 Graduated cylinders. A range of cylinder volumes (e.g., 4000 ml, 100 ml, and 10 ml) is recommended for accurately measuring the volume of water pumped through the CLAM and periodic instantaneous flow rate measurements during deployment.
- 5.4 Deployment Setup
 - 5.4.1 Site-specific conditions should be evaluated to determine an adequate anchor and deployment setup. Typical anchor and deployment materials may include: cement block with eyelet, rebar, buoy, stainless steel cable, chain, anchor shackles, quicklinks, etc. Examples of different deployments are shown in Appendix D.
 - 5.4.2 Tools (e.g., pliers, diagonal cutters, zipties, rebar driver/hammer)
- 5.5 General Field Supplies
 - 5.5.1 Nitrile gloves
 - 5.5.2 Watch
 - 5.5.3 Field notebook

6.0 Summary of Procedure

6.1 Preparation

- 6.1.1 Ordering the SPE Disks. SPE disks are ordered directly from Aqualytical ®. Each disk comes in an individual clean Mylar pouch. Custom-made stainless steel cartridges can also be used in place of HDPP cartridges, but are ordered externally from Saint-Gobain Filtration Technologies.
- 6.1.2 Cleaning and Conditioning the SPE disk. The SPE disk must be cleaned and conditioned with solvents prior to conducting sampling. This is typically performed by the laboratory conducting the analysis.
 - 6.1.2.1 The cleaning step uses solvent to remove residual interferences that can result in contamination of the disks.
 - 6.1.2.2 The conditioning step uses solvent to activate (wet) the sorbent – this allows the analyte of interest to be retained on the sorbent, thus helping prevent the analyte from breaking through the sorbent.
 - 6.1.2.3 An equilibration step uses solvent that is similar to the sample environment in terms of solvent strength and pH to maximize retention of the analyte.
 - 6.1.2.4 In summary, the steps involve cleaning with 50 ml dichloromethane, conditioning with 50 ml methanol, and equilibrating with 50 ml deionized (DI) water. The disks are then capped and returned to the original Mylar pouch.
- 6.1.3 Testing the CLAM pump. Before field deployment, the CLAM should be tested in a clean bucket of DI water. The purpose is to determine that the device's pump is working and that the pump rates are efficient. The test can be performed with used (relatively clean) SPE disks that are kept only for testing purposes. Based on manufacturer specifications, expected pump rates during field deployments range about 5–60 ml/min. Based on previous tests performed by EAP staff, pump rates ranging about 30–70 ml/min have been observed when the CLAM is deployed in DI water. To perform a pump test:
 - 6.1.3.1 Fill a clean bucket with DI water.
 - 6.1.3.2 Insert SPE disk onto CLAM.
 - 6.1.3.3 Install batteries in CLAM.
 - 6.1.3.4 Connect one end of tubing to the CLAM's discharge port.
 - 6.1.3.5 Deploy CLAM in bucket of DI water.
 - 6.1.3.6 Wait for a consistent flow with no bubbles in order to purge the air from the tubing. Then, connect opposite end of tubing to the water collection bucket. Note the start time.
 - 6.1.3.7 Test the pump for at least one hour. If desired, the beginning, mid-point, and end instantaneous pump rates can be measured (Section 6.2).

6.1.3.8 At the end of the test, remove the CLAM from the water. Measure the volume of water in the water collection bucket. This can be done using a large graduated cylinder (e.g., 4000 ml), or similar apparatus.

6.1.3.9 Calculate the total pump rate in ml/min as:

$$\text{Total Volume Measured (ml)} / \text{Total Deployment Time (min)}.$$

6.1.3.10 The following information should be recorded: Date; Staff; CLAM Serial #; Disk Lot #; Deployment Start Time; Deployment End Time; Total Volume Measured; Calculated Total Pump Rate.

6.2 Guidelines for Checking Instantaneous Pump Rate

6.2.1 During field deployment, CLAM pump rates should be checked at the beginning, mid-point(s), and end of the deployment. The purpose is to assess any decline in pump rate due to clogging of the SPE disk or due to low batteries.

6.2.2 For each check, the volume of water pumped through the CLAM in exactly one minute is measured using a graduated cylinder and clock. The clock should be started when air is purged from the tubing and there is consistent flow (Section 6.1.3.6). Three instantaneous measurements are taken, and the average is calculated. All measurements should be recorded in a field notebook.

6.3 Field Deployment

6.3.1 Set up a secure anchor system for deploying the CLAM.

6.3.2 Place the water collection bucket in a stable spot where it won't tip over. If in a public location, hide the bucket as much as possible to prevent it from being tampered with.

6.3.3 Connect one end of CLAM tubing to luer lock fitting on bucket, and the other end to the CLAM's discharge port.

6.3.4 Wearing clean nitrile gloves, remove the SPE disk from the Mylar pouch and insert disk onto the CLAM. Label the Mylar pouch with the site name, date, and disk number.

6.3.5 Install batteries into CLAM. Once the batteries are installed, the device is in operation, and exposure time in the air should be minimized.

6.3.6 Secure CLAM to the anchor system. Backups should be used to ensure that the CLAM will not be lost.

6.3.7 Deploy CLAM in the desired location. The disk should ideally face downstream, or away from the direction of flow, to help minimize clogging of the disk. Position the CLAM so that there are no obstructions blocking the intake.

6.3.8 Conduct an initial pump rate (Section 6.2). Empty the contents of the graduated cylinder into the water collection bucket.

6.3.9 Before leaving the site, secure the bucket and lid with a lock.

6.3.10 During the CLAM's deployment, mid-point check(s) should be performed:

6.3.10.1 Check the pump rate (Section 6.2).

6.3.10.2 Check the device batteries.

6.3.10.3 Check the volume of water in the bucket. If the bucket is near full, measure and record the volume of water collected, and then dump it out to prevent the bucket from overflowing.

6.3.10.4 Check the anchor system to ensure that it's still secure.

6.3.11 At the end of the CLAM's deployment:

6.3.11.1 Conduct the end pump rate (Section 6.2). Empty the contents of the graduated cylinder into the water collection bucket.

6.3.11.2 Pull the CLAM from the water. Wearing clean nitrile gloves, remove the SPE disk from the device and immediately place into its original labeled pouch. Seal pouch and store in a cooler on ice.

6.3.11.3 Remove the batteries from the CLAM and stop the pump. Measure the volume of water in the bucket. This (plus the volume of any water measured and dumped out during the mid-point check) is the total amount of water pumped through the CLAM during its deployment. It is used to calculate the sample concentration.

6.3.11.4 Return CLAM to its Pelican case.

6.3.11.5 Remove all equipment from site.

6.4 Sample Processing

6.4.1 SPE disk samples are shipped to the analytical laboratory in a cooler on ice ($\leq 6^{\circ}\text{C}$). The holding time for the SPE disks is 14 days, or 1 year if the disks are stored frozen. When filling out the chain of custody form, a matrix of "Other" is used.

6.5 Calculating Analyte Concentrations

6.5.1 The result provided by the laboratory is the analyte mass on the SPE disk. The final concentration of analyte in the water sample is calculated as:

$$\text{Analyte Mass} / \text{Sample Volume}$$

For example, if the result for toxaphene reported by the laboratory is 10.1 ng/sample, and the total volume of water pumped through the CLAM is 44.1 L, then the final concentration of toxaphene in the water sample is $10.1 \text{ ng} / 44.1 \text{ L} = 0.23 \text{ ng/L}$.

6.5.2 Both the final analyte concentration and the sample volume are entered into Ecology's Environmental Information Management System (EIM). The laboratory reported result (analyte mass on the disk) is not entered into EIM.

7.0 Records Management

7.1 A field notebook should be kept to record notes for CLAM deployments. At minimum, the following information should be recorded:

7.1.1 Site name and coordinates

7.1.2 Sample name

7.1.3 Field staff

7.1.4 Field conditions including weather, site details, and turbidity of water

7.1.5 CLAM serial #

7.1.6 SPE disk #

7.1.7 Deployment start date and time

7.1.8 Midpoint check date and time

7.1.9 Retrieval date and time

7.1.10 Initial pump rate (average of 3 measurements)

7.1.11 Mid-point pump rate (average of 3 measurements)

7.1.12 End pump rate (average of 3 measurements)

7.1.13 Total volume of water pumped through CLAM

7.2 Table 2 provides guidance on entering data for selected fields in EIM that are especially relevant for CLAM data. Guidance for entering information for all EIM fields is provided in Ecology's EIM Help Documents.

7.3 Each sample should have two types of results, which should be entered as separate Result Parameter Names in EIM: the analyte concentration and the sample volume. To enter the analyte concentration, select the analyte's name as the Result Parameter Name. To enter the sample volume, select Sample Event Volume Flow as the Result Parameter Name.

Table 2. Guidance on values to enter for selected fields in EIM.

EIM Field	Value to Enter for CLAM Data
Field Collection Type	Sample
Field Collection Start Date	(Date of CLAM deployment)
Field Collection Start Time	(Time of CLAM deployment)
Field Collection End Date	(Date of CLAM retrieval)
Field Collection End Time	(Time of CLAM retrieval)
Field Collection Comment	(May want to include comments such as turbidity and flow of the water)
Sample Composite Flag	Yes
Sample Collection Method	INSITU-SPE
Result Parameter Name	(Choose the analyte's name to enter the analyte concentration) (Choose "Sample Event Flow Volume" to enter the sample volume)
Result Value	(Final concentration of the analyte in water)
Result Value Units	(Units of concentration: mass/volume)
Result Method	(Analytical method for the analyte)

8.0 Quality Control and Quality Assurance Section

8.1 Volume Measurements

- 8.1.1 There are three different ways that the water collected by CLAMs has been estimated in past EAP studies: (1) Measure the actual total volume; (2) Estimate volume by regression; (3) Estimate volume using the device's "totalizer."
- 8.1.2 The accepted method for EAP studies is to measure the actual total volume of water pumped through the CLAM during its entire deployment. This method provides the most accuracy because it is a direct measurement – it does not rely on a derived estimation by calculation.
- 8.1.3 Estimation by linear regression using the start and end pump rates has shown to produce biased high volumes in the field, which likely resulted in biased low analyte concentrations (Hobbs and Friese, 2015; Friese and Coots, 2016). Because of its inaccuracies, linear regression is no longer used to estimate volumes from CLAMs for EAP studies.
- 8.1.4 Clogging of the disks is the main reason for the decline in pump rates over time. During CLAM deployments in three different river systems in Washington, Hobbs et al. (2018) observed *exponential* declines in pump rates at all sites, with >90% declines over deployment periods of ~30 hours at the more turbid sites (Appendix E). Half of the decline occurred within about 10 hours, suggesting that sample collection is weighted toward the beginning ~10 hours of deployment.
- 8.1.5 The CLAM's totalizer uses optics to continuously measure the volume of water as it is pumped through the CLAM. However, the totalizers were found to be unreliable, consistently producing biased high volumes (Friese and Coots, 2016). Because of this, EAP studies no longer use the device's totalizer to estimate volume.

8.2 Blank Samples

- 8.2.1 Blank samples are used to assess the level of inadvertent contamination in field and laboratory equipment, or in exposure of the SPE media to the atmosphere. The type and number of blank samples should be determined by the project manager.
- 8.2.2 Types of blank samples typically collected for CLAM studies are described below.
 - 8.2.2.1 A field blank is collected to assess and quantify potential contamination that occurs during the time that the SPE disk is exposed to the air when the CLAM is deployed/retrieved and possible exposure during travel and shipping. This is performed by removing the end caps of a clean unused disk, exposing the disk to the air for the approximate duration a typical CLAM deployment and retrieval was exposed to air, then replacing the caps.
 - 8.2.2.2 A raw media blank is used to assess and quantify potential contamination of the SPE media itself. The SPE media is analyzed separately from the cartridge during the solid phase extraction procedure.
 - 8.2.2.3 A disk blank serves as the laboratory method blank and is used to assess and quantify potential contamination of the SPE media + cartridge during the solid phase extraction procedure.

- 8.2.3 The approach to handling the laboratory method blank sample results is either to apply data censoring (qualifications) or blank corrections to the analytical results. The appropriate method is determined by the project manager and may depend on specific project objectives. Censoring or blank corrections for CLAM data are applied to the result analyte mass on the sample media, rather than to the analyte concentration in the water sample.
- 8.2.4 Several EAP studies using CLAMs to sample for organic analytes in the water observed blank contamination (Friese and Coots, 2016; Hobbs and Friese, 2016; Era-Miller and McCall 2017). It was determined that the HDPP cartridges housing the SPE media were the cause for blank contamination in PCB and PBDE samples. As a result, EAP now uses customized stainless steel cartridges for studies of PCBs and PBDEs using CLAMs in order to reduce the problem of background contamination in samples (Hobbs et al., 2018). For studies of other analytes, collection of blank samples can help determine whether the HDPP cartridges contribute to background contamination. Nevertheless, collection and testing of blank samples is especially important for all CLAM studies.

8.3 Replicate Samples

- 8.3.1 Replicate samples are two or more samples taken from the environment at the same time and place, using the same protocols. Replicates are used to estimate the random variability of the material sampled (Jones, 1998). For CLAM deployments, replicate samples are collected by deploying/retrieving two or more CLAMs at the same location and time.

8.4 Data Results

- 8.4.1 The data package from the analytical laboratory includes the sample results and a case narrative. The project manager reviews the results and case narrative to ensure there are no errors, or that any errors are identified and examined.

9.0 Safety

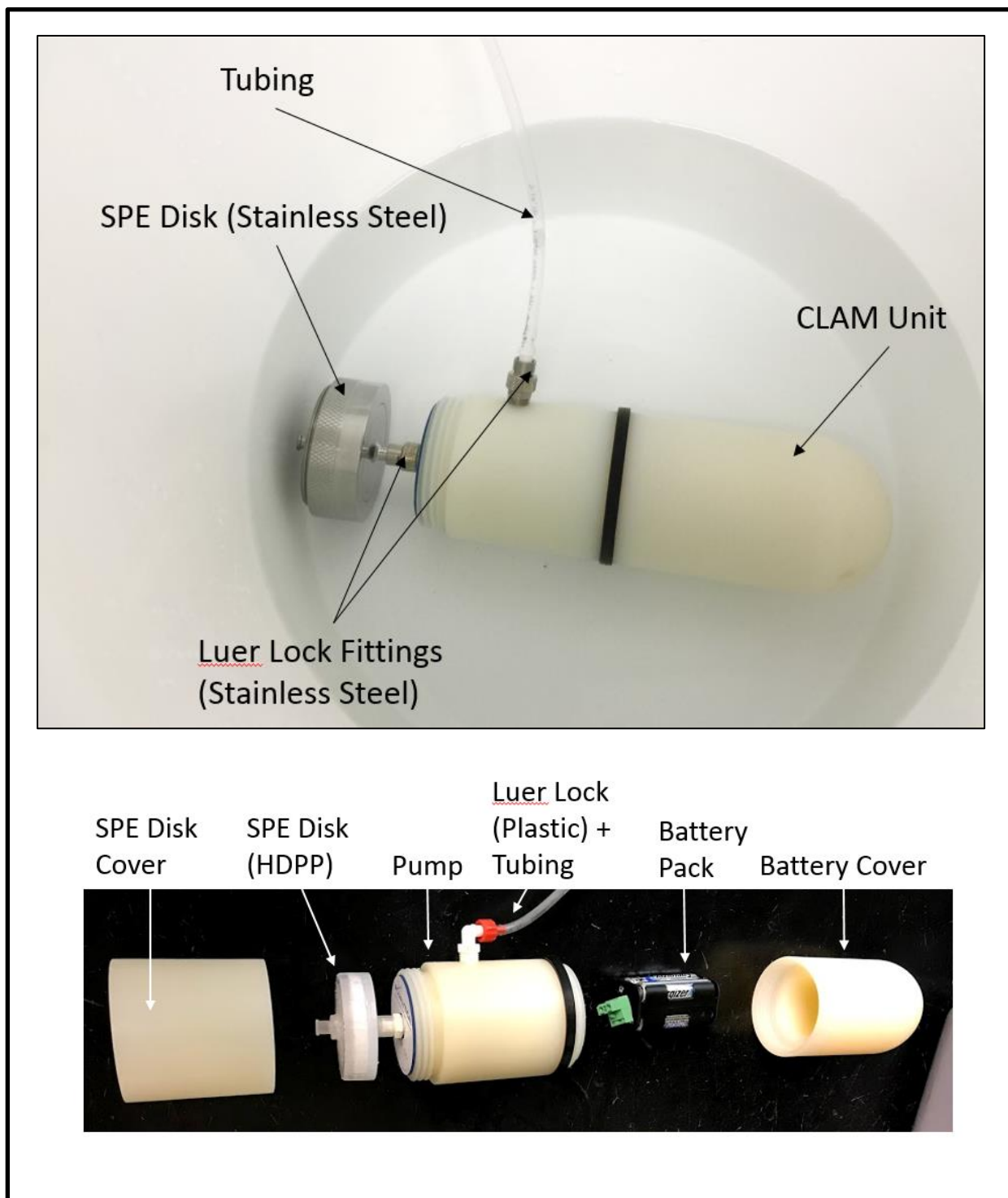
- 9.1 All fieldwork related to sample collection using CLAMs should follow the protocols in the EAP Safety Manual (Ecology, 2019). Special attention should be paid to sections describing working in rivers and streams, working near traffic and from bridges, and operating vehicles.

10.0 References

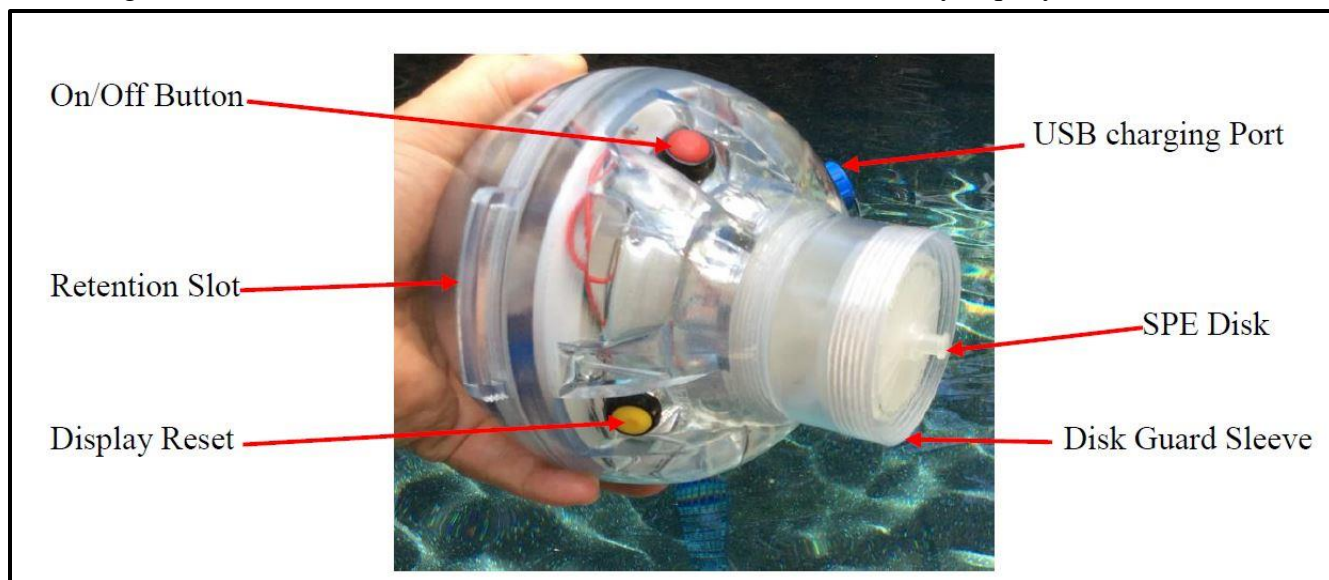
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<https://fortress.wa.gov/ecy/publications/SummaryPages/1003043.html>

Appendix A: Diagrams of CLAM

A-1. Diagrams of older model of CLAM.



A-2. Diagram of the CLAM 4000/5000 (newer CLAM models). Photo by Aqualytical ®



Appendix B: CLAM Components



B-1. SPE Disk – Stainless Steel



B-2. SPE Disk – HDPP



B-3. CLAM tubing



B-4. Example water collection container



B-5. Mylar pouch for SPE Disk



B-6. Luer Lock
Fittings –
Plastic



B-7. Luer Lock
Fitting –
Stainless Steel



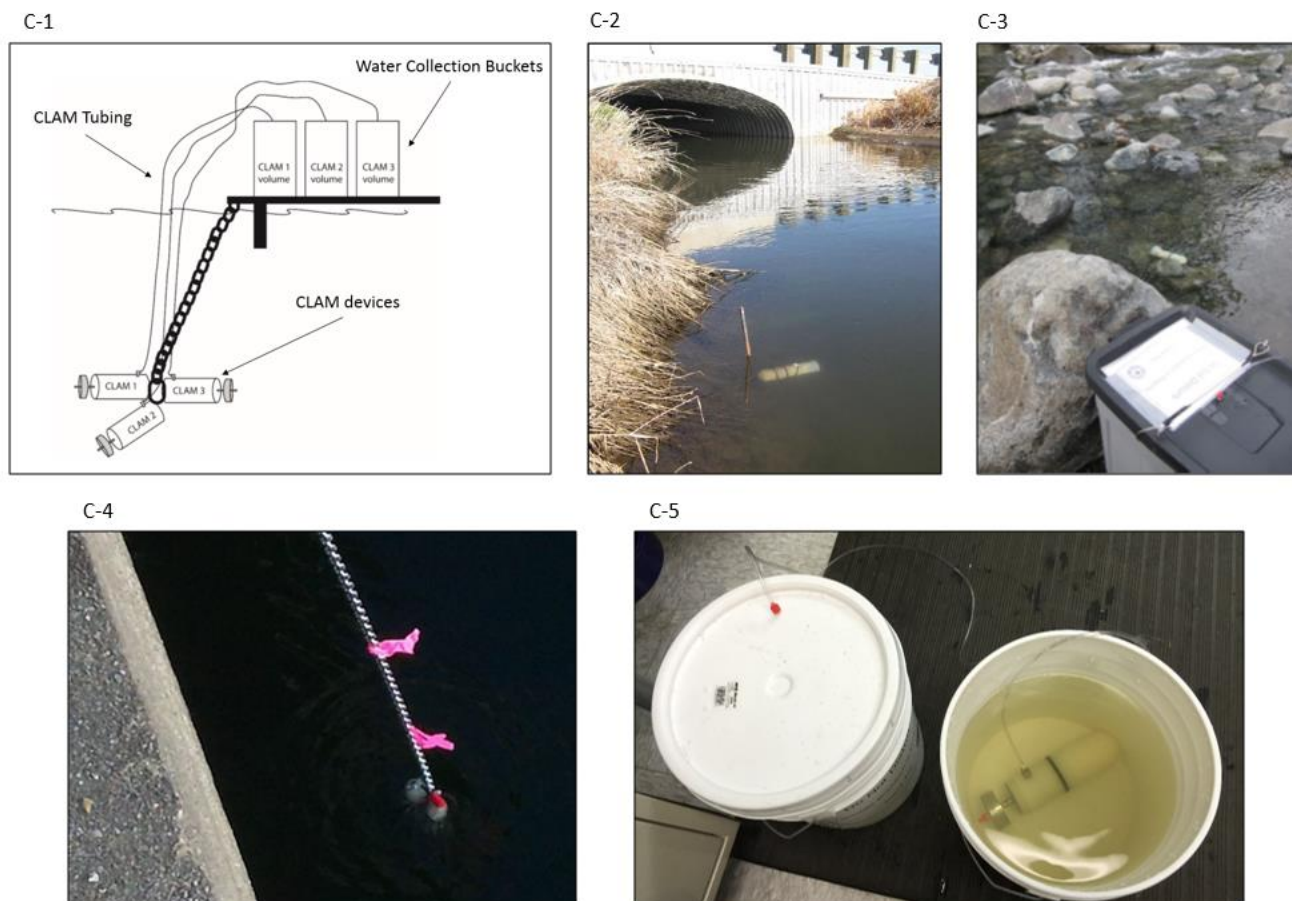
B-8. SPE Media – C18

Appendix C: Field Checklist Template

CLAM Field Checklist

- ☐ CLAMs (stored in Pelican cases)
- ☐ SPE disks in Mylar pouches
- ☐ Cooler with ice
- ☐ Water collection containers (customized to fit tubing and locking mechanism)
- ☐ Locks & keys for water collection containers
- ☐ AA batteries for CLAMs (4 per unit, plus extra)
- ☐ CLAM tubing
- ☐ Luer lock fittings
- ☐ Nitrile gloves
- Graduated cylinders:
 - ☐ Large (4000 mL)
 - ☐ Medium (100 mL)
 - ☐ Small (10 mL)
- ☐ CLAM deployment/anchor setup:
- ☐ Miscellaneous tools (hammer, screwdriver, diagonal cutters, pliers, zipties, anchor shackles, quicklinks)
- ☐ High profile, low profile cement blocks with steel eyelet
- ☐ Rebar sections with cable eyelets/attached steel cable
- ☐ Rebar driver/mallet
- ☐ Extra steel cable
- ☐ Chain
- ☐ Storage bucket
- ☐ Watch

Appendix D: Field Deployment Setup Examples



C-1. Schematic of deployment used in the Spokane River, WA. The three CLAMs are suspended off of a platform using chain. The CLAMs pump water through the tubing and into separate water collection buckets, which sit on a platform. Source: Adapted from Hobbs and McCall (2016).

C-2. CLAM deployed in the Burlingame Canal near Pine Creek, WA. The CLAM is weighted with a cement block. Cable or chain connecting the CLAM to rebar staked to the bottom serves as a backup. Source: Hobbs and Friese (2015).

C-3. CLAM deployed in a tributary of the Wenatchee River, WA. The CLAM is weighted with a cement block. The CLAM pumps water through the tubing and into the water collection bucket, which is secured on the bank.

C-4. CLAM suspended off a bridge and deployed in Ballona Creek, CA. Source: [Low Level Detections of Organochlorine Pesticides: Ballona Creek, Los Angeles, CA - Summer 2011](#)

C-5. CLAM pump test. A CLAM is deployed in a bucket of field site water (right bucket). Water is pumped through tubing into a water collection bucket (left bucket).

Appendix E: Exponential Declines in CLAM Pump Rates

The plots show exponential declines in CLAM pump rates while deployed over a period of 24–36 hours in three different river systems in Washington (Hobbs et al. 2018). The total percent decline in pump rate is given for each plot, as well as the time it took for the CLAM's pump rate to drop by half.

