Mosier OR, Train Derailment
August 16, 2016

Prepared for
Union Pacific Railroad/CH2M

Prepared by

FOOD SAFETY & ENVIRONMENTAL STEWARDSHIP LABORATORY
Peter Hoffman, Assistant Director.

ENVIRONMENTAL AND MOLECULAR TOXICOLOGY
1007 AGRICULTURE AND LIFE SCIENCES BUILDING
OREGON STATE UNIVERSITY
CORVALLIS, OR 97331-7301

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email: kim.anderson@oregonstate.edu
Executive summary

The Food Safety & Environmental Stewardship Program (FSES) provided sampling and analytical services at Columbia River sites associated with the June 3rd UPRR oil train derailment and fire, with oil contamination of the near-adjacent Columbia river. The analytical work and sampling plan was developed with consultation from UPRR representatives (CH2M) and personnel representing the Oregon Department of Environmental Quality (ODEQ), Environmental Protection Agency (EPA) and the National Oceanic and Atmospheric Administration (NOAA).

Under this agreement FSES provided the following services:

- Standard operating procedures (SOPs) for laboratory operations, analytical methods and field methods for finalization of the Mosier train derailment Sampling and Analysis Plan (SAP)
- Manufactured low-density polyethylene (LDPE) passive sampling devices (PSDs) with performance reference compound (PRC) infusion
- Field sampling equipment rental for:
  - 6 locations with surface water samplers, two each at upstream (Mosier East 1 and 2), wastewater treatment outfall (Mosier Mid 1 and 2) and downstream (Mosier West 1 and 2).
  - Mosier Mid 2 location sampled with triplicate, co-located samplers.
- Subsequent extraction of PSDs and measurement of 61 polycyclic aromatic hydrocarbons (PAHs).
- Analytical report (this document).
**Sampling and Analytical Plan**

A sampling and analytical plan (SAP) containing standard operating procedures (SOPs) covering the preparation, manufacture, pre- and post-deployment cleaning, field deployment and recovery, extraction and quantitative determination of parent and alkyl-substituted polycyclic aromatic hydrocarbons were provided and methodology for calculation of freely dissolved water concentrations of organic compounds were provided on July 7, 2016 (SAP attached).

**Field Operations**

After on-site examination, six field locations were chosen for sampler deployment. Two were significantly upstream to prevent wind-driven sampler contamination with surface sheen oil (East 1 and 2), two at the wastewater treatment outfall site, the apparent source of the oil sheen (Center 1 and 2) and two samplers downstream of the outfall sight (West 1 and 2). Triplicate samplers were deployed at Center 2. Deployment of PSDs occurred on June 24th and samplers were retrieved July 8th. During retrieval, sampler Center 1 was found entangled in beachside debris apparently deposited there by extreme wind-driven wave action during the week preceding retrieval. The three remaining Center 2 samplers were undisturbed.

Sampler locations (latitude and longitude) were as follows (refer to picture for identity):

<table>
<thead>
<tr>
<th></th>
<th>latitude</th>
<th>longitude</th>
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<tbody>
<tr>
<td>East 1</td>
<td>45.68658</td>
<td>-121.39647</td>
</tr>
<tr>
<td>East 2</td>
<td>45.68645</td>
<td>-121.39673</td>
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<tr>
<td>*Center 1</td>
<td>Sample lost</td>
<td>Sample lost</td>
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<tr>
<td>Center 2 (1, 2, and 3)</td>
<td>45.68742</td>
<td>-121.4044</td>
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<td>West 1</td>
<td>45.68729</td>
<td>-121.40589</td>
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<td>West 2</td>
<td>45.68737</td>
<td>-121.40601</td>
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</table>

*the second sampler placed at the center location was lost likely due to extreme wind and wave action.*
LDPE PSD Processing and Analysis

Upon arrival in the laboratory on July 8th, LDPE PSDs were stored at -20 °C and logged into the FSES laboratory information management system (LIMS) within 24 hours. PSDs were post-deployment cleaned and extracted on July 12, 2016. PSDs were analyzed for PAHs and data analyzed between July 12 and August 12, 2016.

PAH results

All PAH analysis achieved our desired data quality objectives (DQOs) as specified in the SAP. Summarized certificate of analysis (COA) attached.

Questions: If you have any additional questions, please call the Food Safety and Environmental Stewardship Laboratory Director at 541-737-8501.

DIRECTOR: _________________________________ DATE: __________________________
Kim A. Anderson, PhD
Certificate of Analysis

Client Report For: Union Pacific Railroad

Project Name: Mosier Oil Spill River Water
Project Number: F16-24
Report Date: August 15 2016

QC Review Date

FSES Director Approval: Kim A. Anderson Date
Methodology:

SOP 418.00: Determination of Parent and Alkyl Substituted PAHs by Gas Chromatography-Tandem Mass Spectrometry

Unit Conversions:

ppb = parts per billion
ppm = parts per million
ppt = parts per trillion
ng/g = ppb
ng/L = ppt
ng/mL = ppb
ng/μL = ppm
ng/WB(Wristband) = ppb
pg/μL = ppb
μg/mL = ppm

Abbreviations:
J flag: Indicates lower precision in quantitation due to values near limits of detection or matrix effects.
B flag: The sample was background corrected.
< 123.45 U: Detection limit, indicates value was below limit of detection.

COA Notes:
CAS Registry Numbers are provided after chemical names on the following pages.
<table>
<thead>
<tr>
<th>Chemical Name</th>
<th>Concentration (ng/L)</th>
<th>Concentration (ng/L)</th>
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<tbody>
<tr>
<td>1,2-dimethyl-naphthalene, 573-99-8</td>
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<td>Concentration (ng/L)</td>
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<td>9,10-dimethylanthracene, 781-43-1</td>
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### Food Safety and Environmental Stewardship Program

**COA Report**

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<tr>
<th>Chemical Name</th>
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SAP for Mosier Train Derailment, prepared July 7, 2016.

The SAP is organized as follows:

- Section 1: Summary of Passive Sampling Approach
- Section 2: PRC Loading and LDPE PSD Preparation
- Section 3: Field Deployment and Retrieval Methods
- Section 4: Sample Identification and Handling Procedures
- Section 5: Post-Processing and Sampling of PSDs
- Section 6: Field QC Samples
- Section 7: Laboratory Analysis and Quality Control
- Section 8: Data Calculations
- Section 9: Schedule and Reporting

Supporting materials are provided in associated, tables, figures, and appendices as cited throughout.

1.0 Summary of Passive Sampling Approach

Eight LDPE water-samplers will be deployed at six sites as indicated on the associated map. Two sampler arrays will be deployed at the approximate location of the initiating point of the oil sheen with one array containing triplicate water cages and designated Source 1 a, b or c and Source 2. Two sites 0.6 km upstream of the source of the oil sheen will be sampled with single samplers to provide river-specific background analyte levels and designated Upstream one and Upstream 2 and two sites approximately 0.1 km downstream will also be sampled with individual samplers, and designated Downstream one and Downstream 2.

As discussed in associated conference calls and follow-up E-mails, the samplers are equipped with inert low-density polyethylene (LDPE) tubing, which essentially acts as a carbon sink such that PAHs sorb to the LDPE. Performance reference compounds (PRCs) will be impregnated into the LDPE as described in OSU’s Standard Operating Procedures (SOPs) for passive sampling provided in the Appendix. Pictures of a water sampler are provided in Figure 1.

Our intent is that passive samplers will be deployed by field staff from shore in shallow water (sampling strategies requiring boat deployment/retrieval will incur additional costs). Field deployment is further discussed in Sections 3.0 and 3.1.

2.0 PRC Loading and LDPE PSD Preparation

The passive sampling LDPE and DGT media will be prepared at the OSU laboratory before deployment to make PSDs. The following OSU SOPs, provided in the Appendix, describe the procedures for preparation of LDPE, infusion of PRCs onto the LDPE, and fabricating the PSDs before deployment.

- SOP 2120.06 – Cleaning of LDPE for Fabrication of PSD
- SOP 2131.00 – Drying of the Pre-Cleaned Organic PSDs
All LDPE used on this project will be prepared in the same batch to ensure comparability between sample results. Because all samples will be prepared in the same manner and at the same time and PSDs are loaded with a known volume and concentration of PRC fortification solution, tracking of time-zero concentrations by PSD is not necessary. The PRCs used on this project will be fluorene-d10, pyrene-d10, and benzo(b)fluoranthene-d12. The LDPE PSDs will be used for analysis of PAHs, as described in Section 7.1.

3.0 Field Deployment and Retrieval Methods

At the beginning of field work, a pre-sampling meeting will be held with all field staff to (1) discuss health and safety requirements; and (2) review sampling objectives and procedures as outlined in this SAP.

The OSU Aquatic Field Sampling Methods for PSDs SOP 100.03 (Appendix) describes the techniques for loading the PSDs into the surface water cages, and the deployment and retrieval techniques that should be followed in the field. Temperature loggers (“tidbits”) will be included to allow accurate calculation of free-water concentrations. A summary of project specific deployment and retrieval techniques is provided in this section and should be reviewed and followed by the field team to ensure data of sufficient quality and consistency are collected to meet project objectives.

Passive samplers will be deployed from shore or shallow water by OSU personnel. GPS positioning data will be recorded at each deployment site. The actual sample coordinates will be recorded in the field logbook. Because of relatively shallow sampling sites (typically 3 to 4 feet), water cages will be set at depths of 2-feet from the bottom and sampling site depths estimated.

On-site inspection and on-going deployment ensure likely availability of indicated target sites. Should unanticipated changes in conditions occur which generate hazardous conditions (deep water and/or high wind-driven swells) at any site, deployment may be re-evaluated and target sites moved to ensure team safety.

Any site changes will still be selected to provide sufficient data to make upstream/source/downstream comparisons.

For shallow water stations where samples can be collected by field staff from shore:

- Field staff should be equipped with waders and the appropriate personal protective equipment (PPE).
- All in-water field staff will work in pairs (at least), using the buddy system to complete all tasks.
- On-shore support staff will maintain areas for preparing and processing samples on-shore will be set up so that as much of the work can be conducted from shore vs. in the water.
- Actual sample location will be collected after the sampler is installed.
3.1 Field Placement of Passive Samplers
The prepared LDPE PSDs (see Section 2.0) will be transported to the Site by OSU staff in coolers equipped with “blue ice.”

Before deployment, the LDPE PSDs will be inserted into the surface water cage as described in OSU Aquatic Field Sampling Methods for PSDs SOP 100.3. All personnel handling the PSDs should wear nitrile gloves and care should be taken to minimize any contact with the LDPE PSDs inside the samplers.

Surface Water Deployment:
- Place the PSD on the deployment hardware.
- Place the deployment hardware into the deployment cage and secure.
- Attach cage(s) to deployment gear, ensuring that the gear is adjusted for the appropriate water depth and so that the top of the sampler is approximately 24 inches above the mudline. The weight of the anchor and buoyancy of the support buoy should be appropriate for deployment conditions. If a top buoy is used as a visual marker, ensure sufficient slack line to account for changes in water depth.
- Submerge entire apparatus into the water and verify that deployment is correct before releasing the top buoy or rope.

3.2 Field Retrieval of Passive Samplers
Following the deployment period the field team will re-mobilize to the Site to retrieve the passive samplers. The top buoy should allow for visual relocation of the deployment gear. Teams will remove the water samplers and transport to a nearby onshore field team. Once onshore, OSU field personnel will remove the PSDs from the protective sampling cage, rinse them with water from the Site to remove sediment or biofouling (if necessary), and store the LDPE PSDs in labeled, clean, sealed jars for transport to the OSU laboratory within 24 hours of retrieval.

4.0 Sample Identification and Handling Procedures
This section describes procedures for sample identification and chain of custody that should be used for field activities. These procedures ensure sample quality is maintained during collection, transportation, storage, and analysis.

4.1 Sample Identification
Assign each sample a unique identification (ID) number describing the sample location. Record the sample number on a waterproof sample label and affix the label to the sample jar. To aid in data management, the field team should use consistent sample location numbers as described below. Each sample ID will consist of abbreviations reflecting the following six components:

1. Site Identification: MTD = Mosier train derailment
2. Sample Matrix: SW = surface water exposure, Sample Media: PE = polyethylene (i.e., LDPE)
3. Sample Month: e.g., 10 = Month of sample retrieval (e.g., October)
4. Sample Year: e.g., 15 = Year of collection (e.g., 2015)
5. Sample Station Number: An alphanumeric code indicating direction (East, West or Source) and sample number.

For example, MTDSWPE0716-E1 would be used to describe the surface water exposed LDPE sample collected in July 2016 at station East 1. Triplicate source samples will be appended with S1a, S1b and S1c respectively (ie MTDSWPE0716-S1a).

4.2 Sample Labels

Use sample labels to identify samples collected in the field. Once recovered, the PSDs will be placed in laboratory-cleaned amber bottles and a sample label will be firmly attached to the sample container and covered with clear packaging tape for protection. Information on the sample label should be sufficient to enable cross-reference with the field bench sheets and contain, at a minimum, the following information:

- Sample identification
- Date and time of collection

4.3 Sample Custody

OSU personnel will maintain custody of the samples for the duration of fieldwork, transport, and processing and will follow standard custody procedures to trace the possession and handling of a sample from collection to completion of all required analyses. Chain of custody procedures and forms, if required, will adhere to the requirements set forth in the OSU Chain of Custody for QAPP SOP 2021.01 (Appendix K).

4.4 Sample Handling and Packaging

All PSDs will be transported between the field and the OSU laboratory by OSU field staff and thus shipping of the samples or additional transfer of custody is not anticipated. PSDs will be stored in clean amber containers in coolers containing “blue ice” to maintain refrigeration before arrival at the laboratory. Coolers will be packaged carefully to avoid breakage or contamination. Given that inter-agency custody transfers are not anticipated, custody seals will not be used.

4.5 Field Logbooks and Data Forms

Field deployment/recovery bench sheets and data forms are necessary to document daily activities and observations. Documentation should be sufficient to enable participants to accurately and objectively reconstruct project events. Entries should be made in waterproof ink, dated, and signed.

4.6 Photographs

Photographs will be taken to document field activities. Photos will be downloaded into a photo log file, and kept with the electronic project files.

5.0 Post-Processing and Sampling of PSDs

Upon arrival at the OSU laboratory, samples will be logged in, and the LDPE PSDs will be frozen until extraction. The procedures for processing the LDPE PSDs before analysis are described in OSU’s passive sampling SOPs and summarized below by PSD media.
5.1 LDPE Cleaning, Sub-sampling, and Extraction
The LDPE PSDs will be cleaned to remove biofouling, water, and other potential analytical interferences. If frozen, samples will be brought back to room temperature before cleaning. Cleaning will be conducted under a solvent hood at the OSU laboratory following the procedures outlined in the OSU Cleaning Field Deployed Polyethylene PSDs SOP 108.01. This procedure includes submerging the PSDs sequentially in three different solvents. Once cleaned, the PSDs are cut into pieces using clean scissors and stored in amber jars until extraction. The OSU chemist will fill out a bench sheet documenting the cleaning process and will include a post-deployment cleaning blank with each batch of samples.

The method for extracting organic compounds (i.e., PAHs and PCP) from the PSDs by use of a two-step hexane dialysis is described in the OSU Extraction of Organic Compounds from Polyethylene PSDs SOP 406.02 (Appendix K). Once dialysis is completed, the extracts are transferred and concentrated in hexanes. The OSU chemist will fill out a bench/QC summary sheet documenting the extraction process and will include all field and/or laboratory QC samples to ensure batch extraction quality.

6.0 Field QC Samples

Field Triplicate (FT) The FT is a triplicate sample collected at a target sample location (i.e., three samples collected independently at a sampling location during a single act of sampling). The purpose is to evaluate the precision of sampling procedures. FT samples will be collected at one of the two source sample sites. Additionally, trip, construction and field blanks will be generated and analyzed.

7.0 Laboratory Analyses and Quality Control

7.1 Chemical Analyses
Chemical analysis of the surface water samples will be completed by the OSU laboratory as described in the following SOPs, provided in the Appendix:

- SOP 418.00 – Determination of Parent and Methyl Substituted Polynuclear Aromatic Hydrocarbons (PAHs) using Gas Chromatography Tandem Mass Spectrometry (GC-MS/MS)

For the purpose of this project, the 62 PAHs will be reported as listed in the associated SOP. Additionally, limits of detection (LODs) and limits of quantitation (LOQs) are provided within the SOP.

7.2 Laboratory QA/QC Procedures
Laboratory QA/QC will be maintained through the use of standard EPA methods and other accepted methods and standard analytical procedures for the target analytes. The method-specific and other analytical and laboratory QC procedures and protocols followed are detailed in the laboratory’s method-specific SOPs. These procedures incorporate the collection and analysis of the following laboratory QA/QC components:

- Internal QC samples
- Continuing calibration verification (CCVs)
8.0 Calculations of Water Concentrations from PSD Results

The methods for estimating porewater and surface water concentrations from concentrations measured in PSDs, are described for LDPE extraction results.

8.1 LDPE Calculations

In order to assess an analyte’s in situ LDPE-water exchange kinetics performance reference compounds are added. Labelled compounds will be used ensuring that the PRCs do not occur in the environment. By determining the depletion of PRC compounds during the deployment period, the sampling rate of target constituents in LDPE can be estimated and used in conjunction with literature-based partitioning coefficients to calculate the sampler water partitioning coefficient. Water concentrations can then be calculated based on the equations provided in Section II.C of the OSU Calculation of PAH Water Concentrations Derived from PSD SOP 407.01.

OSU personnel will perform all of the calculations necessary to convert the raw LDPE results and present the final surface water and porewater results in their report (Section 4.8.2).

8.0 Schedule, Reporting and Data Sharing

8.1 Schedule

To be determined

8.2 Reporting

A preliminary report, including field placement and retrieval methods, sample preparation and transport, analytical methods, data analysis, interpretation of results, and conclusions will be prepared by OSU and submitted approximately 45 days after completion of fieldwork.

8.3 Data Sharing

Water concentration data will be available to the FSES program at Oregon State for inclusion into scientific publication and dissertations. Data values will be compared to those obtained from additional technologies currently co-deployed at Mosier, and will be used with appropriate acknowledgement of primary data ownership by Union Pacific.
Cleaning of Low Density Polyethylene for Fabrication of PSD

Food Safety and Environmental Stewardship Program
Oregon State University Standard Operating Procedure

Scope
This document describes the procedure for fabricating PSD from low density polyethylene (LDPE) tubing received from Brentwood Plastic Inc. Cleaning of the LDPE removes chromatographic interferences and other contaminants and must be performed prior to preparation of PSD for any experimental uses. Cleaning includes solvent washes, vacuum drying and storage of PSD.

Contributors: S.E. Allan

Responsible
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

Status
This document is considered current standard operating procedure of the Food Safety and Environmental Stewardship Laboratory when management approval is documented by signature below. This Standard Operating Procedure is effective on the date of approval signature and supersedes all previous versions.

Approved
Kim A. Anderson, Ph.D.

Director
Title
Date

Historical File
Signature/Initials
Date

I. Equipment and Apparatus
A. Additive-free low-density polyethylene lay-flat tubing, approximately 75-95 µm thick x 2.54 cm wide – Barefoot® from Brentwood Plastics Inc.
B. Large, wide mouth amber glass jars (1000 mL) with lid
C. Shaker table in warm room or floor incubator with shaker
D. Continuous Temperature Logger (optional)
E. Measuring tool to 1.08 meter
F. Two flex frames with aluminum rods
G. Two clamps
H. Scissors

II. Reagents
A. Hexanes – Fisher Scientific Optima Grade or better
B. Acetone – Fisher Scientific Optima Grade or better
III. Procedure
A. LDPE pre-cleaning in preparation to fabricate PSD
1. Assign a sequential lot number for the PSD batch and mark on the bench sheet. A log book for PSD pre-cleaning is maintained and can be referenced for lot numbers.
2. Clean working surface by cleaning thoroughly with detergent and water then acetone and hexanes.
3. Wear nitrile gloves at all times while handling tubing. Minimize exposure to environment.
5. Place 8 pre-cut PSDs, or < 9 m of tubing, in a 1 L clean amber jar.
6. Add warm hexanes approximately 100 mL of hexanes for 1 m of tubing.
   a) Warm hexanes to approximately 26°C in a water bath prior to use
   b) Assure the tubing moves freely and hexanes contact all surfaces.
7. Place jar(s) on shaker table in warm room or floor incubator.
   a) The temperature during pre-cleaning should be maintained at 26 ± 4°C. Set shaker table at 60 ± 15 RPM
   b) Record the temperature of the warm room or incubator immediately prior to each exchange. OPTIONAL: use a temperature logger or other recorder.
8. Three hexanes extractions occur over a total of approximately 72 hour extraction time. Each exchange occurs approximately every 24 hrs. Note: All exchanges should occur in a hood.
   a) Pour used hexanes into a solvent waste container while maintaining PSD in the jar.
   b) Rinse tubing and inside of jar with a small amount of hexanes and discard hexane waste.
   c) Refill jar with warm hexanes as before.

B. PSD Drying and Storage
1. Prepare workspace in fume hood as described in A.2-3 above
2. Remove hexanes from tubing by squeezing towards open end.
3. Dry PSDs according to SOP 2131 Drying of PSDs.
4. After drying, store tubing in clean jar or can. Label with PE PSD batch lot number.

IV. Quality Control
A. In recognition of advances that are occurring in separations, the analyst is permitted certain options to improve the separations or lower the cost. Each time such a modification is made to the method, the analyst is required to document any alterations to the procedure on bench sheets during the analysis. For permanent changes, the standard operating procedures should be formally updated with new validations.
B. Place a minimum of 2 PSDs from each lot in a clean container and archived in the freezer. Individual projects will often have specific data quality objectives (DQOs).

C. Extract a minimum of 1 blank PSD from each lot using at least one of the PE PSD methods, (e.g. SOP 406) to check for background. Analysis and results will be documented on the PSD preparation bench sheet.

D. For instance, the Superfund PAH project DQO is a 500 pg D12-Perylene spike on the cleaned PSD requires a 2:1 (+/- 20%) peak to background noise.

V. Documentation Requirements
A. Check [http://fses.oregonstate.edu/sop-toc](http://fses.oregonstate.edu/sop-toc) for current version of all required documents.

B. A bench sheet is required for PE PSD pre-cleaning
   a) [2120-LDPE-pre-cleaning-for-PSD-Fabrication.xls](2120-LDPE-pre-cleaning-for-PSD-Fabrication.xls)

VI. Safety and Health
A. Please consult Material Safety Data Sheet (MSDS) information on reagents. Personnel performing this method will observe all appropriate Oregon State University laboratory safety procedures. All procedures involving hexanes should be carried out under a chemical hood. Always use personal protective equipment. Use protective gloves (nitrile) while handling all PE PSDs. Dispose of solvents into solvent waste containers for disposal by Environmental Health and Safety Department, OSU campus.

VII. Validation
A. Not applicable
# SOP 2120 Cleaning of LDPE for Fabrication of PSD

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<th>Approx. vol. hexanes per jar (mL)</th>
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<th>1st solvent exchange start time</th>
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<th>Roll PSD- remove hexanes</th>
<th>Transfer PSD to drying keg</th>
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**Initials**

**Date**

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**Initials**

**Date**

**Temperature Logger:** YES / NO

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**QC Check - Analysis of Blank PSD**

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**Chemist Review**

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**FSES Director Review**

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Drying of Pre-cleaned Organic Passive Sampling Devices (PSDs)

Scope
This document describes the procedure for drying pre-cleaned organic passive sampling devices (PSDs). This document is applicable to LFT (made of LDPE) and silicone polymer based PSDs.

Contributors: S.E. Allan, K.A. Hobbie, K. Kamerud

Responsibilities
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

Status
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Approved
Kim A. Anderson, Ph.D.

Director
Title

Date

Historical File
Signature/Initials

Date

I. Equipment and Apparatus
A. Nitrile gloves
B. Two (2) polyurethane foam plugs (PUFs) – cleaned using SOP 2130.
C. A large cylindrical glass column/tube such as a Soxhlet extractor with fittings to accommodate PUFs with a tight seal, or a large metal chamber such as a Cornelius keg.
D. Flow meter capable of measuring 1 to 10 L/min at room temp
E. Tygon vacuum tubing, applicable fittings for drying chamber and vacuum supply
F. Appropriate clean PSD storage container (Examples: glass jar, paint can, PTFE bag)

II. Reagents
A. Hexanes – Rinse Grade or better
B. Acetone – Rinse Grade or better
C. Ethyl Acetate – Rinse Grade or better
D. Methanol – Rinse Grade or better
III. Procedure: Either a glass or metal chamber can be used in this drying method, use appropriate steps below based on apparatus. Use protective gloves (nitrile) while handling all PSDs.

A. Glass Chamber PSD Drying Method
   NOTE – Glass Chamber should be cleaned according to SOP 2110
   1. Place PSDs into the large cylindrical column or Soxhlet extractor tube.
   2. Stack two clean PUFs in the top of the tube or funnel and attach the male fitting to the condenser end of the column or tube (See Figure 1). Note: multiple tapered fittings may be needed for a proper fit.
   3. Block the flask end (bottom) of the column or tube with a stopper modified to fit the vacuum tubing. Attach the other end of the Tygon tubing to the flow meter. Attach the exit end of the flow meter to the house vacuum line.
   4. Turn on vacuum and adjust flow to approximately 3.6 to 5.3 cc/min meters (approx 1 L/min, or 2.5,6 on Brooks E/C meter SN 7004-39338/4).
   5. Leave apparatus under vacuum until organic solvent is removed from PSDs, ~48 to 72 hours at room temperature, depending on the amount of tubing.
   6. After drying, store PSDs in a clean container. Label with PSD batch lot number.

B. Metal Chamber Pre-cleaning Method
   1. For LFT use:
      a) Rinse inside of keg with acetone, including metal tubing. Repeat two more times with acetone, and three times with hexane.
      b) Attach keg to vacuum with tygon tubing.
      c) Rinse the inside of the metal tubing by creating a small pool of acetone in the bottom of the keg. Recap the keg, and turn on the vacuum until all solvent is removed. Repeat two more times with acetone and three times with hexane.
      d) Remove the o-ring from the keg lid.
      e) Rinse the keg lid three times with acetone and hexane.
      f) Using a Kimwipe clean o-ring with acetone and hexane.
      g) Replace o-ring in keg lid.
      h) Before use clean the outside of the keg using a Kimwipe with acetone and hexane.
   2. For Silicone use:
      a) See Metal Chamber pre-cleaning for LFT (III.B.1)
      b) Use ethyl acetate in place of acetone and methanol in place of hexane.

C. Metal Chamber PSD Drying Method
   1. Place PSDs into drying keg and close lid.
   2. Stack two large or four small clean PUFs into the filter cartridge.
   3. Attach the filter cartridge to the keg input using Tygon tubing.
   4. Attach the flow meter to the keg output using Tygon tubing. Attach the exit end of the flow meter to the house vacuum line.
5. Turn on vacuum and adjust flow to approximately 3.6 to 5.3 cc/min (or approx 1 L/min, or 2.5,6 on Brooks E/C meter SN 7004-39338/4).
6. Leave apparatus under vacuum until all organic solvent is removed from PSDs, ~48 to 72 hours at room temperature, depending on the amount of tubing.
7. After drying, store PSDs in clean container. Label with PSD batch lot number.

IV. Quality Control
   A. See appropriate PSD pre-cleaning SOP for applicable Quality Control requirements

V. Documentation Requirements
   A. Check http://fses.oregonstate.edu/sop-toc for current version of all required documents.
      1. See PSD pre-cleaning method SOP for required documentation

VI. Safety and Health
   A. Please consult Material Safety Data Sheet (MSDS) information on reagents. Personnel performing this method will observe all appropriate Oregon State University laboratory safety procedures. All procedures involving hexanes should be carried out under a chemical hood. Always use personal protective equipment. Use protective gloves (nitrile) while handling all LFTs. Dispose of solvents into solvent waste containers for disposal by Environmental Health and Safety Department, OSU campus.

VII. Validation
   A. Not applicable
**Figure 1. Example Glass Drying chamber - LFT drying.** Soxhlet extractor tube attached to PUF cylinder (top) and flow meter (bottom). Note: two PUFs in male fitting connecting to female Soxhlet piece, and stopper connected to male Soxhlet piece and tygon tubing.

**Figure 2. Example Metal Drying Chamber**
Title  Preparation of PE PSDs for Use in Environmental Sampling Equipment

Scope
This document describes the procedure for preparation of polyethylene (PE) passive sampling devices PSDs for use on spiders and other environmental sampling equipment. Fortification of PE PSDs with Performance Reference Compounds (PRCs) is also described. PRC Fortification is necessary for determination of site specific analyte uptake rates in field deployed PSDs.

Contributors: S.E Allan, G. Sower

Responsibilities
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

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Approved______________________________  Director______________________________
Kim A. Anderson, Ph.D.  Title  Date

Historical File______________________________
Signature/Initials  Date

I.  Equipment and Apparatus
Note: Clean all glassware and containers for use in organic chemistries (see SOP 2110- Laboratory Container Cleaning procedure).
A.  Heat sealer, TEW Electric Impulse Sealer TISH-100 – or equivalent
B.  Appropriate storage container, examples: wide mouth amber glass jars or PTFE clip and seal bags
C.  100 µL pipetman – or equivalent
D.  Measuring tool to 1 meter or equivalent (for un-cut PSD)
E.  Two flex frames with aluminum rods – or equivalent (for un-cut PSD)
F.  Two c-clamps – or equivalent (for un-cut PSD)
G.  Scissors (for un-cut PSD)
H.  Pre-cleaned PSD (See SOP 2120)

II.  Reagents
A.  Hexanes – Fisher Scientific Optima Grade – or better
B.  Acetone – Fisher Scientific Optima Grade – or better
III. Standards
   A. Performance Reference Compound standard fortification solution. See SOP 2240 for standard preparation, labeling, storage and expiration.

IV. Procedure
   A. Preparation of work station
      1. Table surface, sealer, flex-frames, clamps and scissors must be solvent cleaned (two rinses of acetone, followed by two rinses of hexanes) before use.
      2. Set up the flex-frame with aluminum pegs and c-clamps 99 cm apart (for un-cut PSD)
   B. Preparation of PSD for assembly on spiders
      1. Clean nitrile gloves should be worn at all times during PSD preparation. Minimize exposure to environment and light.
      2. Heat-seal a 4 cm circumference loop on one end of the tubing using flex frames.
      3. If PSD was not pre-cut, measure and cut the PSD before proceeding to the next step.
      4. At this stage, include PRC fortification if needed. See Section IV.C below.
      5. Force air out of the tube by running the tube from the sealed to open end through fingers.
      6. Heat-seal a 4 cm circumference loop on the other end of the tubing.
      7. The total length of the PSD is approximately 99 to 100 cm after sealing both ends.
      8. Store completed PSD and label container with number and fortification yes/no. Note: For cage deployment in the field, five or ten PSD are commonly stored together.
   C. Fortification of PSDs
      1. PSD PRC fortification solution must be prepared by a chemist in accordance with the needs of the project.
         a) The chemist will indicate the volume of solution that should be added to each tube.
      2. Carry out fortification just before step #5 in section IV.B.
         a) Open the unsealed end of the PSD and open the tube by running fingers down the tube seam toward end.
         b) Insert the pipette tip into the tube opening and add the fortification to the tube.
      3. Complete the steps 5-8 in section IV.B.

V. Quality Control
   A. Each preparation batch will include a single lab preparation blank, which will remain open to the environment for the time-frame of the PSD batch construction.
   B. Record the PSD lot number and the fortification solution information on the construction bench sheet.

VI. Documentation Requirements
   A. Check http://fses.oregonstate.edu/sop-toc for current version of all required documents.
      1. Bench sheet for PSD construction.
VII. Safety and Health
   A. Please consult Material Safety Data Sheet (MSDS) information on reagents. Personnel performing this method will adhere to Oregon State University Chemical Hygiene Plan and applicable FSES safety SOPs.
   B. Always use personal protective equipment.
   C. Use protective gloves (nitrile) while handling PSDs.
   D. Dispose of solvents into solvent waste containers for disposal by Environmental Health and Safety Department, OSU campus.

VIII. Validation
   A. Not applicable
SOP 2121 Benchsheet  
PE Passive Sampling Device Construction

Project: ________________________________

Date: ________________________________

Participants: __________________________

Check For Completion

1. Retrieve SOP 2121 for reference
2. Verify quantity of PSD tubing needed:
   - _______ Total PSDs Required
   - _______ PRC Fortified
   - _______ Unfortified
   PSD used for construction LOT #: _______

3. Bring spiking solution to room temperature

4. Plug in heat sealer

5. Verify pipette:
   - Pipette volume: _______
   - Pipette serial number: _______

6. Clean prep table and all relevant equipment with acetone and hexanes

7. Test the length of the first PSD constructed on a deployment spider frame

8. Construct lab preparation blank and hang in construction area

9. Construct all remaining PSDs

Start Time: ____________________________  End Time: ____________________________

PRC Solution

<table>
<thead>
<tr>
<th>FSES ID</th>
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<th>Expiration Date</th>
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Notes:

Chemist Review
   Signature  Date

FSES Director Review
   Signature  Date
Aquatic Field Sampling Methods for Passive Sampling Devices

Food Safety and Environmental Stewardship Program
Oregon State University Standard Operating Procedure

Scope
This method describes the techniques for deploying and retrieving organic and inorganic passive sampling devices (PSD) in water and sediment pore water. Deployment gear options for oceanic, riverine and lentic systems are explained. Deployment gear and hardware cleaning is described.

Contributors: S.E. Allan, G. Sower, L.B. Paulik

Responsibilities
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

Status
This document is considered current standard operating procedure of the Food Safety and Environmental Stewardship Laboratory when management approval is documented by signature below. This Standard Operating Procedure is effective on the date of approval signature and supersedes all previous versions.

Approved ___________________  Director ___________________  Date _____________
Kim A. Anderson, Ph.D.  Title  Date

Historical File ___________________  Date _____________
Signature/Initials

I. Equipment and Apparatus
   A. Passive Sampling Devices (PSD) such as polyethylene, silicone, or DGT prepared for deployment (as per applicable SOP 2120, 208)
   B. Amber jars or sealable Teflon bags
   C. Top buoys, support buoys
   D. Steel cables
   E. Adjustable rigging (lock-down bolts, U-bolts)
   F. Ropes
   G. Anchors
   H. Deployment cages or probes, with mounting and closure hardware

II. Equipment and Apparatus Gear Clean-up at End of Field Season
   A. Pressure washer
   B. Steel brushes
   C. Scrub pad
   D. Oven capable of holding the cages and maintaining ~350 °C.
II. Reagents
A. HCl, reagent grade for 2 N HCl Bath
B. 18 M Ω•cm water

III. Field Deployment (Figure 1)
Specific projects may have unique data quality objectives and field methods that criteria may be used, and will take precedent for those samples. During deployment and retrieval, take precautions to minimize PSD exposure to the air and prevent contact with unclean surfaces. Always wear gloves when handling PSDs.

A. Water column deployment
1. Cable gear can be used in any system where suspension of the PSDs in the water column is desired and water movement will not displace the rigging. Gear should be adjusted for appropriate water depths and cage placement depending on the project
   a) The weight of the anchor and buoyancy of the support buoy should be appropriate for the deployment conditions.
   b) A top buoy, may be used to serve as a visual marker, can be attached to the support buoy. Allow plenty of slack line to account for changes in water depth.
2. Alternatively, cages can be suspended from or attached to permanent structures.
3. Deployment of PSDs in water column
   a) Place PSD on deployment hardware.
   b) Place deployment hardware in the deployment cage and secure.
   c) Attach cage(s) to deployment gear.
   d) Submerge entire apparatus into water and verify that deployment is correct before releasing the top buoy or rope.

B. Sediment porewater deployment
1. When selecting deployment locations, keep in mind that you must be able to retrieve the probe at the end of the deployment. Changing tides, etc., necessitate careful selection of deployment locations.
2. It is suggested to attach the the probe to something immobile onshore, using metal cable or equivalent or to the water/air PSD gear. This reduces the risk of loss or theft.
3. Deployment of PSDs in porewater. (See Figure 1 & 2.)
   a) Place PSD on deployment hardware (probe insert).
   b) Place deployment hardware in the deployment probe and secure.
   c) Submerge probe into sediment, with only the cylindrical top in the water column. For soil deployments or in tidal flats the top portion of the probe may be in the air column. Attach probe(s) to shore for added security if needed.

IV. Field Retrieval
A. Locate sampling gear and remove from water/sediment.
B. Remove PSDs from the protective cage/probe and deployment hardware.
   1. Organic PSDs can be cleaned with water from the sampling site to remove sediment or biofouling if necessary.
   2. DGTs can be rinsed with 18 M Ω•cm water to remove biofouling in field if necessary.
C. Store PSDs in appropriate labeled container for transport.
V. Field Re-deployment
   A. Clean deployment gear to remove biofouling and sediment
   B. Inspect deployment gear and hardware for damage or excessive ware and replace or repair as needed.
   C. Deploy PSDs as described in section III.C.

VI. Cleaning Deployment Gear at End of Field Season
   A. Cleaning buoys and ropes
      1. Spray with the pressure washer
      2. Air-dry before storage
      3. Inspect gear for damage or ware and repair or replace as needed
   B. Cleaning deployment hardware when necessary if the pressure washer is not sufficient.
      1. Soak deployment hardware in 2 N HCl for at least 5 minutes.
      2. Remove hardware from acid bath and wash with the pressure washer.
      3. Scrub hardware until no debris remains
      4. Rinse in DI water.
      5. Reassemble deployment hardware, air-dry, then bake at ~350 °C for 12 hours.
      6. Store the hardware in bags or clean containers. Note the date and clean status.
   C. All other deployment gear and hardware (clips, springs etc.) can be cleaned according to SOP 2110 and stored in bags or clean containers.

VII. Quality Control
   A. Each field batch should include a trip blank and field blank.

VIII. Documentation Requirements
   A. Check http://fses.oregonstate.edu/sop-toc for current version of all required documents.
      1. A completed field collection sheet is required for all field deployments
      2. A completed field collection sheet is required for all field retrievals

IX. Safety and Health
   A. Please consult Material Safety Data Sheet (MSDS) information on reagents. Personnel performing this method will adhere to Oregon State University Chemical Hygiene Plan and applicable FSES safety SOPs. Closed toed shoes should be worn on boats, and when using the pressure washer. Life jackets should always be worn on boats. Request and adhere to boat operator’s Health and Safety Plan. Other safety gear may be applicable when in the field.

X. References

DOCUMENT UNCONTROLLED IF PRINTED - See http://fses.oregonstate.edu/sop-toc
for latest version
XI. Validation
A. Not applicable

Figure 1: Example aquatic system PSD deployment set-up includes top buoy attached by rope to support buoy, steel cable, adjusting rigging, anchor, PSD cage deployed at 1 to 10 ft from sediment bottom, and sediment probes attached to anchor.
Figure 2: Sediment probe insert/PSD configuration for porewater PSD deployment.
# SOP 100 PSD Deployment Benchsheet

**Deployment Date:**

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<th>Project Number</th>
<th>Project Name</th>
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<th>Collector Name (print and sign)</th>
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<th>Environmental Media (Circle one)</th>
<th>PSD Fortification Type (Circle one)</th>
<th>GLOVES ON Load PSD into Sampler (check)</th>
<th>Deployment Start Time</th>
<th>Initials &amp; Date</th>
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<tr>
<th>Chemist Review</th>
<th>Signature</th>
<th>Date</th>
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</thead>
<tbody>
<tr>
<td>Director Review</td>
<td>Signature</td>
<td>Date</td>
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**DOCUMENT UNCONTROLLED IF PRINTED - See http://fses.oregonstate.edu/sop-toc for latest version**
SOP 100 PSD Retrieval Benchsheet

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<td><strong>Site Location</strong></td>
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<td><strong>Site Description (optional):</strong></td>
<td><strong>Longitude:</strong></td>
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<table>
<thead>
<tr>
<th>Sample Name (ID)</th>
<th>PSD type (Circle one)</th>
<th>Environmental Media (Circle one)</th>
<th>PSD Fortification Type (Circle one)</th>
<th>GLOVES ON - Retrieve Samplers, Remove PSDs and store in labeled container (check)</th>
<th>Deploy. End Time</th>
<th>Initials &amp; Date</th>
<th>Notes:</th>
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<td>Bioassay / PRC Fortified / NA</td>
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<td><strong>QC Type</strong></td>
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<td><strong>Water Depth</strong></td>
<td>Field Blank</td>
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<tr>
<td><strong>Other:</strong></td>
<td>Trip Blank</td>
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</tbody>
</table>

Chemist Review | Signature | Date |
Director Review | Signature | Date |
Title  Chain of Custody for QAPP

Scope
To ensure proper documentation of samples received with chain-of-custody documentation as designated for the FSES Laboratory Quality Assurance Program Plan.

Contributors:  K.A. Anderson

Responsibilities
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

Status
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Approved

Kim A. Anderson, Ph.D.  Director

Title  Date

Historical File

Signature/Initials  Date

This SOP was created to meet the FSES Laboratory Quality Assurance Program Plan (QAPP) project needs. SOP 2020, filed under GLP Hold, should be used in cases when adherence to strict Good Laboratory Practice (GLP) is required.

I.  Documentation practices at FSES Laboratory are guided by a QAPP. Proper chain of custody for a QAPP refers to documentation of sample possession from collection, sample receipt, sample preparation, analysis and reporting, and disposition. This sample chronological history provides integrity to sample collection, identification, shipment, analysis, and reporting of results. Chain of custody procedures require the following:

A.  Sample receipt is documented with signatures of the person relinquishing the sample and the person receiving the samples.

B.  Storage of the sample is documented with initials of the person placing the sample in storage.
C. A secured storage area is used with limited access to only defined staff.
D. Sample archival and disposal is documented.

II. When FSES staff are not involved in the collection of samples, the starting point for active sample accountability and traceability is typically sample receipt which includes the hand-off (shipping documentation or signature) from courier to FSES. Samples may be delivered by U.S. mail, FEDEX, UPS, bus, and/or freezer truck. Sample conditions vary (i.e., frozen, cooled, ambient temperature, sealed, unsealed, intact, broken, leaking, decaying). Sample documentation may not be complete by sample collector (e.g., documentation describing sampling methods, date sampled, sample identification, description of sample, sample size, shipment information, desired analysis, storage conditions, client contact information, and reporting parameters). All such circumstances (when provided) should be documented upon sample receipt as they may have significant bearing on the reliability and quality of the analytical result. All sample submissions and receipt information are logged into the FSES Sample Log-in Book or LIMS and a case file is generated which serves as a systematic and dependable receptacle for documentation. Signature of person(s) relinquishing samples may not always be possible when samples are not hand delivered or are dropped off. Indicate NA (not available) and date/initial. See attached example FSES chain of custody.

III. Intra-laboratory chain of custody for regulated studies is documented with sample processing and analysis bench sheets. These documents record sample handling, subsampling, methodology, date and personnel performing extraction steps, lot number, manufacturer, and grade of extraction reagents, serial number and manufacturer of pipettes and analytical balances, sample extract storage (location, conditions) and date of instrumental analysis. When a method or procedure has been developed for specific sampling processing and analysis, bench sheets and forms are available under the respective FSES standard operating procedures (SOPs).
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<th>GC-MS oxy-PAHs</th>
<th>GC-MS - Flame Retardants</th>
<th>GC-MS - PCBs</th>
<th>AMDIS multi-organic contaminant screen</th>
<th>GC-MS - Pesticides</th>
<th>LC-MS - oxy-PAHs</th>
<th>TID</th>
<th>Preservation Used</th>
<th>Solvent (if Applicable)</th>
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**TESTS REQUESTED**

Would you like your samples returned?  

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<th>NO</th>
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**OTHER INFORMATION**

**Reporting Requirements**

- **Key**
  - _e-mail Results_
  - _Verbal Results_
  - _FAX Results_

**Storage Requirements:**

- **Storage Location:**
  - Location
  - Signature

**CHAIN OF CUSTODY:**

- **Signature**
  - Time/Date
  - Relinquished By:
  - Received By:

**OSU Oregon State University**

DOCUMENT UNCONTROLLED IF PRINTED - See http://fses.oregonstate.edu/sop-toc for latest version
Title: Cleaning Field Deployed Polyethylene Passive Sampling Devices (PSDs)

Scope
This method describes the method for cleaning polyethylene passive sampling devices that have been deployed in the environment. Cleaning is intended to remove potential analytic interferences prior to extraction of organic compounds from the LFT. Procedures for cleaning air and water deployed LFT are described.

Contributors: S.E. Allan

Responsibilities
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

Status
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Approved

Kim A. Anderson, Ph.D.

Director

Title

Date

Historical File

Signature/Initials

Date

I. Equipment and Apparatus
Note: All jars and equipment are cleaned for use in organic chemistries (see SOP 2110-Laboratory Container Cleaning procedure)

A. Large, wide-mouth glass jars (for cleaning)
B. Amber, wide-mouth glass jars (for storage and later dialysis)
C. Scissors

II. Reagents
A. Hexanes, EMD OmniSolv grade, or equivalent
B. Acetone, EMD OmniSolv grade, or equivalent
C. Isopropanol, EMD reagent grade, or equivalent
D. 1 N HCL solution, Fisher, reagent grade, or equivalent
E. 18 MΩ•cm water, EASYpure UV, Barnstead, or equivalent
III. Water Deployed polyethylene PSD cleaning

Note: All cleaning should be carried out in a solvent hood. Care should be taken to minimize PSD contamination including exposure to air and gloves should be worn at all times when handling PSDs.

A. Polyethylene PSDs are cleaned individually to remove debris, biofouling, water and other potential analytical interferences.

B. Cleaning solutions should be prepared in large glass jars and replaced as needed or between every sample. PSDs are sequentially cleaned in three different solvents described below:
   1. If PSD are excessively dirty, perform an initial cleaning with 18 MΩ•cm water.
   2. Cut the tubing lengthwise prior to cleaning.
   3. Submerge PSD in 1N HCL
      a) Clean tubing thoroughly using fingers or cleaning tools as necessary to remove debris and biofouling, remove then,
   4. Submerge PSD in 18 MΩ•cm water
      a) Remove as much water as possible by passing tubing through fingers remove then,
   5. Quickly submerge PSD sequentially in two isopropanol baths
      a) Remove excess solvent by passing tubing through fingers
   6. Dispose of isopropanol, rinse glassware and replace isopropanol baths between samples to prevent carry over.

II. Air Deployed polyethylene PSD Cleaning

A. PSDs are cleaned individually to remove particulate matter and moisture.

   1. Quickly submerge each PSD in isopropanol then remove excess solvent by passing tubing through fingers.

III. PSD Cutting and Storage

A. Cleaned PSDs are cut into pieces using clean scissors

   1. Rinse scissors with acetone prior to use and between samples.
   2. Cut sealed loops and PSD into sections to facilitate dialysis

C. Cleaned, cut PSDs are stored in amber jars until dialysis or further processing.

V. Quality Control

A. A post deployment cleaning blank should be included with each batch.

VI. Documentation Requirements

1. A completed benchsheet is required. Check http://fses.oregonstate.edu/sop-toc for current version of all required documents.

2. SOP 108 Cleaning Field Deployed Polyethylene Passive Sampling Devices (PSDs) Bench Sheet

VII. Safety and Health

A. Please consult Material Safety Data Sheet (MSDS) information on reagents. Personnel performing this method will observe all appropriate Oregon State University laboratory safety procedures.
VIII. References
   Not applicable

IX. Validation
   A. Not applicable
### Cleaning Field Deployed Polyethylene Passive Sampling Devices (PSDs) Bench Sheet

**Cleaning Date:**
- **Deployment Date:**
- **Retrieval Date:**
- **Project:**
- **Chemists:**

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<th>Sample Name</th>
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<th>Wash in 18 MΩ Water</th>
<th>Submerge in First Isopropanol Bath</th>
<th>Submerge in Second Isopropanol Bath</th>
<th>Cut PSD into sections and cut loops</th>
<th>Store in organic-clean amber jar</th>
<th>Dispose, rinse, and refill isopropanol bathes</th>
<th>Date</th>
<th>Initial</th>
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</table>

**Company and Grade**
- **Lot Number**

**Notes:**

**LFT**
- **Storage Location**

**Chemist Review**
- **Signature**
- **Date**

**FSES Director Review**
- **Signature**
- **Date**
Title: Extraction of Organic Compounds from Polyethylene (PE) Passive Sampling Devices (PSD)

Scope
This method describes the extraction of organic compounds by hexanes from polyethylene passive sampling devices. Extracts are concentrated in an appropriate solvent to a final volume for instrumental analysis.

Contributors: S.E. Allan, G.J. Sower, R.P. Scott

Responsibilities
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

Status
This document is considered current standard operating procedure of the Food Safety and Environmental Stewardship Laboratory when management approval is documented by signature below. This Standard Operating Procedure is effective on the date of approval signature and supersedes all previous versions.

Approved

Kim A. Anderson, Ph.D.
Title
Date

Director

Historical File

Signature/Initials
Date

I. Equipment and Apparatus
Note: Clean all glassware and containers for use in organic chemistries (see SOP 2110-Laboratory Container Cleaning procedure).

A. Amber glass jars; wide-mouth, screw-cap (50 mL and 500 mL)
B. Adjustable pipette
C. TurboVap™ 500 Closed Cell evaporator (or equivalent)
D. TurboVap™, Zymark LV evaporator (or equivalent)
E. TurboVap 500 mL Tubes with 1 mL sensor mark
F. Borosilicate centrifuge tubes (sized for TurboVap™)
G. Glass pipettes with 2 mL rubber bulbs
H. Large glass funnel
I. Glass stir rod
J. Chromatography vials, 2 mL, amber, Agilent (no substitutes)
K. Chromatography vial caps, Agilent (no substitutes)
L. Aluminum foil
II. Reagents
A. Hexanes, Fisher, GC-Resolve Grade, for dialysis (no substitutes)
B. Hexanes, Fisher, Optima Grade, for glassware rinses
C. Acetone, Fisher, Optima, HPLC or Pesticide Grade

III. Standards
A. Primary Standards – See SOP 2240 Analytical Standard Receipt, Storage and Documentation.
B. Standard Solutions – See the compound specific analysis SOP for the analyte composite and SOP 2250 for standard preparation, storage and expiration.

IV. Sample Preparation
Prior to dialysis PSDs should be cleaned (if necessary), cut into pieces and stored in the wide mouth amber jars used for dialysis (see SOP 108).

Note: Wear clean nitrile gloves at all times when handling tubing and minimize environmental exposure.

Note: If photosensitive compounds are to be determined minimize exposure to light during dialysis and volume reduction steps.

Note: If required, surrogate standards can be added immediately prior to dialysis to account for losses during the process.

A. PE PSD Dialysis
1. 1st Dialysis
   a) Add ~40 mL of hexanes (GC-resolve) for each PSD or enough to completely cover the PSDs.
   b) Let stand for at least 4 hours or overnight at room temperature.
   c) Transfer first dialysate to TurboVap tube.
      (1) Place the funnel on top of the tube and decant hexane into tube. A stir rod or other tool can be used to maintain PSD in the jar.
      (2) Rinse the following items with n-hexane and capture all rinsate in the tube:
         (a) PSD and inside of jar (3 times on 2nd dialysis)
         (b) Inside of the funnel and exterior bottom quarter of the funnel.
         (c) Glass rod or other tool that was in contact with dialysate.
      (3) Rinse the funnel and other glassware with acetone and hexanes between samples

2. 2nd Dialysis
   a) Add the same amount of hexanes as used in section IV.A.1.a to the original amber jar that still contains the PSD.
   b) Let stand for 2 hours.
   c) Transfer to TurboVap tube using the same procedure described in IV.A.1.c.
      (1) Both dialysates are combined into one tube.
B. Extract Concentration
1. Reduce solvent to approximately 1 mL using the TurboVap closed cell evaporator.
2. Quantitatively transfer extracts to centrifuge tubes using glass pipettes.
   a) Prior to transfer mark 1 mL volume, or other desired final volume, on the centrifuge tubes
3. Concentrate extract to final volume in appropriate solvent.
   a) Vortex in centrifuge tube then transfer extract to labeled chromatography vial.

V. Quality Control
A. Each batch of extracts should consist of field samples and appropriate field and/or laboratory QC samples to ensure batch extraction quality.

VI. Documentation Requirements
A. A completed bench/QC summary sheet is required for all analyses. Check http://fses.oregonstate.edu/sop-toc for current version of all required documents.
   1. SOP 406 Extraction of Organic Compounds from PE PSD Benchsheet

VII. Safety and Health
A. Please consult Material Safety Data Sheet (MSDS) information on reagents. Personnel performing this method will adhere to Oregon State University Chemical Hygiene Plan and applicable FSES safety SOPs. All procedures involving hexanes should be carried out under a chemical hood. Always use personal protective equipment. Dispose of solvents into solvent waste containers for disposal by Environmental Health and Safety Department, OSU campus.

VIII. References
# SOP 406.02 Extraction of Organic Compounds from PE PSD Benchsheet

**Dialysis Start Date:**

**PSD Deployment Date:**

**PSD Retrieval Date:**

**PSD Cleaning Date:**

**Chemists:**

<table>
<thead>
<tr>
<th>FSES Sample Number</th>
<th>Sample Name</th>
<th>First Dialysis - 4 h or overnight (40 mL n-Hexane/1m PSD) Start Time</th>
<th>Transfer dialysate to TurboVap tube - Rinse Funnel 3 times</th>
<th>Second Dialysis 2 h (40 mL n-Hexane/1m PSD) Start Time</th>
<th>Combine dialysates Rinse PSD, Jar and Funnel 3 times</th>
<th>Reduce sample to ~1 mL using TurboVap Closed Cell Concentrator</th>
<th>Quantitative transfer of sample to marked (1 mL) centrifuge tube</th>
<th>Reduce to 1 mL or less in TurboVap Evaporator- adjust volume to 1 mL</th>
<th>Transfer samples to marked amber chrom vials for storage</th>
<th>Completed Date</th>
<th>Completed Initial</th>
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**Dialysis Quality Control**

<table>
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<tr>
<th>Reagent Blank</th>
<th>Reagent Company and Grade</th>
<th>Lot Number</th>
<th>Hexanes</th>
<th>Fisher, GC Resolve</th>
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**Equipment**

<table>
<thead>
<tr>
<th>TurboVap® 500 Closed Cell Concentrator</th>
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<tr>
<th>TurboVap® LV Evaporator</th>
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**Samples**

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<th>Storage Location</th>
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**Surrogate**

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<tr>
<th>FSES Number</th>
<th>Concentration</th>
<th>Amount Spiked</th>
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</table>

**Notes:**

**Chemist Review**

<table>
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<tr>
<th>Signature</th>
<th>Date</th>
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**FSES Director Review**

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Determination of Parent and Methyl Substituted PAHs using GC-MS/MS

Food Safety and Environmental Stewardship Program
Oregon State University Standard Operating Procedure

Scope
This procedure applies to the analysis of parent and alkyl substituted PAH compounds by GC-MS/MS. Instrument detection limits are approximately 0.33 to 6.44 pg/uL. Sample and standard solutions should be stored with limited exposure to UV light.

Contributors: G. R. Wilson

Responsibilities
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

Status
This document is considered current standard operating procedure of the Food Safety and Environmental Stewardship Laboratory when management approval is documented by signature below. This Standard Operating Procedure is effective on the date of approval signature and supersedes all previous versions.

Approved

Kim A. Anderson, Ph.D. Title Date

Historical File

Signature/Initials Date

I. Equipment and Apparatus
A. Agilent 7890/7000C GC/MS/MS
B. Splitless single taper line, Agilent, 4 mm ID, Part# 5190-2292 (or equivalent)
C. Deactivated glass wool
D. Septa, Restek, Thermolite 11mm Part# 27142 (or equivalent)
E. Column, J&W Scientific, Select PAH, Part# CP7462, 30m, ID 0.25mm, Film 0.15 um
F. Chromatography vials, 2 mL, amber, screw cap, Agilent (or equivalent)
G. Chromatography vial inserts, Agilent (or equivalent)

II. Reagents
A. Hexanes, Fisher GC Resolv™ (or equivalent)
B. Ethyl Acetate, EMD Omnisolve (or equivalent)
C. Acetone, EMD Omnisolve (or equivalent)
D. Isooctane, Fisher, Optima (or equivalent)

III. Standards
A. See Table 1 for a list of analytes, surrogates and internal standards.
B. Primary Standards are neat standards or solutions ordered from several sources
C. Standard Solutions
   1. Analytical standards shall be prepared in isooctane or hexane at levels that are comparable to estimated sample levels. The suggested range is 10 to 1000 ng/mL.
   2. Expiration of solutions occurs five years after preparation of that solution. Shorter expiration periods may be used and should be used as appropriate, consult with the senior chemist or director if you have any questions. Expired standards must be recertified and documented before use.
   3. Fortification solutions are prepared such that the concentration of spiked compounds in the final extract will be 500 pg/μL. This concentration will be verified prior to use in extractions. Expiration of spike solutions (and subsequent re-verification) occurs one (5 years from the date of preparation, or as noted per chemist discretion.
   4. All stock, standard, and spike solutions are stored at \( \leq 4^\circ C \) and allowed to warm to room temperature prior to use. Storage of neat standards and solutions will be based on the suppliers’ recommendations.

IV. Instrument Operating Parameters
A. Refer to Table 2 for operating conditions
   1. Carrier Gas: Helium (99.99% purity)
   2. Pack a small amount of deactivated glass wool into injection port liner
   3. Use acetone and hexane wash vials for syringe cleaning between injections

V. Sample Preparation
A. See appropriate SOP for sample preparation procedures.

VI. Sample Analysis
A. Sample extracts are analyzed on the GC/MS/MS by method I-PAH-XXX-###_MRM.m ("\( I \" = instrument, "\( X \" = matrix type, "\#" = current method version increasing with each calibration or tune) using the appropriate standards and quality control samples. Qualitative identification of analytes in the extract is performed using retention time and mass spectral information. Quantitative analysis is performed by quantifying the relative abundance of characteristic masses (MRM) see Table 1.
B. Calibration
   1. Standard calibration mixes, containing all target analytes in the method, or target analytes of specific interest, will be run at the beginning of a method or when the verification standard fails DQOs. The calibration standard series will be run, the data edited, and the calibration curve updated prior to running samples.
   2. The calibration standard samples should be calibrated according to the procedures outlined in the MS MassHunter User’s Guide. Analytical standards are used to quantify concentrations using a calibration curve specific to each analyte. Curves can be constructed using an average
response factor (ARF), linear or quadratic curves with or without weighing depending on their individual instrumental response.

3. Before running a batch of samples a continuing calibration verification (CCV) sample will be analyzed. See Table 3 for DQOs and corrective actions.

C. Analysis

1. Use GC/MS/MS method name: I-PAH-XXX-###_MRM. Analytes, surrogate compounds and the internal standard are identified and quantified as pg/µL by the MassHunter software. At least one other MRM is used as a qualifier in the mass spectral display for identification (see Table 1). Analytes are corrected for surrogate compound recovery by the MassHunter software. Surrogate compound responses are a ratio of relative response factors to the internal standard. Surrogate compounds and internal standards can change with different matrices and extraction methods. Please refer to the matrix specific SOP to determine applicable surrogate compounds and internal standards. Analyte instrumental responses are expressed as a ratio of relative response factors to their specified surrogate compound. If the response for a peak exceeds the calibration range by more than nominally 20%, dilute the extract, and adjust the internal standard concentration to 500 pg/µL and reanalyze.

VII. Calculations

A. Fortification levels:

1. \[ \frac{[\text{std vol (µL)} \times \text{std conc (pg/µL)}]}{\text{final volume of sample (µL)}} = \text{fortification level (pg/µL)} \]

VIII. Quality Control

A. The calibration curve should have an \( r^2 \geq 0.99 \) and consist of no fewer than five (5) standards. A continuing calibration verification should be run at the beginning of each day. If the percentage recovery is +/- 30% of the true values sample analysis may begin. If the recoveries are outside the DQOs then the instrument should be re-calibrated and/or troubleshoot as applicable. At least one calibration check should be run each day, for large batches, a calibration check should be run every 10-15 samples. Specific projects may have unique data quality objectives and that criteria may be used, and will take precedent to assess method performance as applicable. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the specific extraction method used.

B. In recognition of advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to document any alterations to the procedure on bench sheets during the analysis. For permanent changes, the standard operating procedures should be formally updated with new validations.

C. A solvent blank should be run with each sequence, the concentration of analytes detected in the laboratory reagent blank should be zero.

D. The recovery of analytes in laboratory spikes should be between 70% and 130% of the true value.
IX. Documentation Requirements
A. Check http://fses.oregonstate.edu/sop-toc for current version of all required documents.
B. A completed bench sheet, QC summary and a copy of the calibration verification results are required for all analyses.

X. Safety and Health
A. Please consult Material Safety Data Sheet (MSDS) for information on PAHs and reagents. The toxicity and carcinogenicity of some chemicals used in this method have not been precisely defined; each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Consult instrument manual for information on safe operation of the GC/MS/MS. Personnel performing this method will observe all appropriate state, federal, and Oregon State University laboratory safety procedures. Further consult SOP4110 for instrumental operation. Precautions should be taken when handling solid chemicals per Oregon State Environmental Health and Safety office for handling carcinogens. Note: handling solids may require specialized trained personnel. More information can be found at http://oregonstate.edu/ehs/carcign/carcign.html

XI. References
A. Application 5989-4184EN: Synchronous SIM/Scan Low-Level PAH Analysis Using the Agilent Technologies 6890/5975 inert GC/MSD, Agilent Technologies, 2005.

XII. Validation
A. This method has been reviewed and validated, the validation packet is located in the fire proof cabinet or the document vault in Weniger.
Table 1: Polycyclic aromatic hydrocarbon compounds, surrogates and internal standards detected by GC/MS. *Surrogate compounds and internal standards can change with different matrices and extraction methods. Please refer to the matrix specific extraction SOP to determine applicable surrogate compounds and internal standards. Listed in Order of Retention on a 30m Select PAH Column:

<table>
<thead>
<tr>
<th>Compound Name</th>
<th>CAS #</th>
<th>Type</th>
<th>Exp_RT</th>
<th>LOD (pg/μL)</th>
<th>LOQ (pg/μL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Naphthalene-D8 SS</td>
<td>1146-65-2</td>
<td>Surrogate</td>
<td>4.11</td>
<td>0.33</td>
<td>1.00</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>91-20-3</td>
<td>Target</td>
<td>4.12</td>
<td>1.04</td>
<td>5.20</td>
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<td>2-Methylnaphthalene</td>
<td>91-57-6</td>
<td>Target</td>
<td>4.57</td>
<td>0.70</td>
<td>3.50</td>
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<td>1-Methylnaphthalene</td>
<td>90-12-0</td>
<td>Target</td>
<td>4.69</td>
<td>0.28</td>
<td>1.39</td>
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<tr>
<td>2-Ethynaphthalene</td>
<td>939-27-5</td>
<td>Target</td>
<td>5.00</td>
<td>0.97</td>
<td>4.84</td>
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<tr>
<td>2,6-Dimethylnaphthalene</td>
<td>28804-88-8</td>
<td>Target</td>
<td>5.07</td>
<td>0.89</td>
<td>4.43</td>
</tr>
<tr>
<td>1,6-Dimethylnaphthalene</td>
<td>575-43-9</td>
<td>Target</td>
<td>5.22</td>
<td>0.81</td>
<td>4.05</td>
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<td>1,4-Dimethylnaphthalene</td>
<td>571-58-4</td>
<td>Target</td>
<td>5.37</td>
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<td>5.86</td>
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<td>2,6-Diethynaphthalene</td>
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<td>Target</td>
<td>6.22</td>
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<td>4.06</td>
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<td>Fluorene D10-PRC</td>
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<td>Target</td>
<td>6.67</td>
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<td>6.73</td>
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<td>3.97</td>
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<td>Dibenzothiophene</td>
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<td>9.24</td>
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<td>9.60</td>
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<td>Phenanthrene</td>
<td>85-01-8</td>
<td>Target</td>
<td>9.71</td>
<td>0.46</td>
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<tr>
<td>Anthracene</td>
<td>120-12-7</td>
<td>Target</td>
<td>9.86</td>
<td>1.05</td>
<td>5.23</td>
</tr>
<tr>
<td>2-Methylphenanthrene</td>
<td>2531-84-2</td>
<td>Target</td>
<td>11.61</td>
<td>0.39</td>
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</tr>
<tr>
<td>2-Methylanthracene</td>
<td>613-12-7</td>
<td>Target</td>
<td>11.75</td>
<td>0.47</td>
<td>2.36</td>
</tr>
<tr>
<td>1-Methylphenanthrene</td>
<td>832-69-9</td>
<td>Target</td>
<td>12.23</td>
<td>1.06</td>
<td>5.32</td>
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<tr>
<td>9-Methylanthracene</td>
<td>779-02-2</td>
<td>Target</td>
<td>13.24</td>
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<td>3,6-Dimethylphenanthrene</td>
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<td>Target</td>
<td>13.24</td>
<td>0.42</td>
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<td>Target</td>
<td>15.14</td>
<td>0.34</td>
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<tr>
<td>Fluoranthen D10 SS</td>
<td>93951-69-0</td>
<td>Surrogate</td>
<td>15.53</td>
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<td>Fluoranthen</td>
<td>206-44-0</td>
<td>Target</td>
<td>15.66</td>
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<tr>
<td>p,p’ DDE D8-PRC</td>
<td>93952-19-3</td>
<td>Target</td>
<td>16.26</td>
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<td>5.00</td>
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<tr>
<td>9,10-Dimethylanthracene</td>
<td>781-43-1</td>
<td>Target</td>
<td>17.20</td>
<td>0.85</td>
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<td>Pyrene D10-PRC</td>
<td>1718-52-1</td>
<td>Target</td>
<td>17.20</td>
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<td>Pyrene</td>
<td>129-00-0</td>
<td>Target</td>
<td>17.20</td>
<td>0.42</td>
<td>2.09</td>
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<table>
<thead>
<tr>
<th>Compound</th>
<th>Retene</th>
<th>Target</th>
<th>Molar Extinction Coefficient</th>
<th>Target Molar Extinction Coefficient</th>
<th>FT-MS Peak Assignment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Retene</td>
<td>483-65-8</td>
<td>17.38</td>
<td>0.84</td>
<td>4.19</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Benzo(a)fluorene</td>
<td>238-84-6</td>
<td>19.35</td>
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<td>5.00</td>
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<tr>
<td>Benzo(b)fluorene</td>
<td>243-17-4</td>
<td>19.73</td>
<td>1.67</td>
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<td>FT-MS Peak Assignment</td>
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<tr>
<td>Benzo(c)fluorene</td>
<td>205-12-9</td>
<td>19.83</td>
<td>0.30</td>
<td>1.50</td>
<td>FT-MS Peak Assignment</td>
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<tr>
<td>1-Methylpyrene</td>
<td>2381-21-7</td>
<td>20.89</td>
<td>0.38</td>
<td>1.90</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Benzo(a)anthracene</td>
<td>56-55-3</td>
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<td>0.75</td>
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</tr>
<tr>
<td>Cyclopenta(c,d)pyrene</td>
<td>27208-37-3</td>
<td>25.95</td>
<td>0.53</td>
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<td>Chrysene-D12 SS</td>
<td>1719-03-5</td>
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<td>Triphenylene</td>
<td>217-59-4</td>
<td>26.04</td>
<td>0.41</td>
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<td>Chrysene</td>
<td>218-01-9</td>
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<td>6-Methylchrysene</td>
<td>1705-85-7</td>
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<td>0.89</td>
<td>4.44</td>
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<td>5-Methylchrysene</td>
<td>3697-24-3</td>
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<td>1.67</td>
<td>5.00</td>
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<tr>
<td>Benzo(b)fluoranthene D12-PRC</td>
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<td>1.67</td>
<td>5.00</td>
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<td>Benzo(b)fluoranthene</td>
<td>205-99-2</td>
<td>30.35</td>
<td>0.37</td>
<td>1.85</td>
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</tr>
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<td>7,12-Dimethylbenz(a)anthracene</td>
<td>57-97-6</td>
<td>30.43</td>
<td>0.94</td>
<td>4.71</td>
<td>FT-MS Peak Assignment</td>
</tr>
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<td>Benzo(k)fluoranthene</td>
<td>207-08-9</td>
<td>30.48</td>
<td>0.53</td>
<td>2.63</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Benzo(j)fluoranthene</td>
<td>205-82-3</td>
<td>30.56</td>
<td>0.56</td>
<td>2.79</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Benz[j]+[e]aceanthrylene</td>
<td>202-33-5 &amp; 199-55-2</td>
<td>31.25</td>
<td>1.67</td>
<td>5.00</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Benzo(e)pyrene</td>
<td>192-97-2</td>
<td>32.25</td>
<td>0.71</td>
<td>3.53</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Benzo(a)pyrene-D12 SS</td>
<td>63466-71-7</td>
<td>32.41</td>
<td>1.67</td>
<td>5.00</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Benzo(a)pyrene</td>
<td>50-32-8</td>
<td>32.58</td>
<td>1.18</td>
<td>5.90</td>
<td>FT-MS Peak Assignment</td>
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<tr>
<td>Perylene-D12</td>
<td>1520-96-3</td>
<td>33.14</td>
<td>4.71</td>
<td>2.04</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Indeno(1,2,3-c,d) pyrene</td>
<td>193-39-5</td>
<td>40.34</td>
<td>0.26</td>
<td>1.32</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Dibenz(a,h)anthracene</td>
<td>53-70-3</td>
<td>40.41</td>
<td>1.02</td>
<td>5.11</td>
<td>FT-MS Peak Assignment</td>
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<td>Picene</td>
<td>213-46-7</td>
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<td>93951-66-7</td>
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<td>Benzo(ghi)perylene</td>
<td>191-24-2</td>
<td>41.71</td>
<td>0.34</td>
<td>1.71</td>
<td>FT-MS Peak Assignment</td>
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<tr>
<td>Anthanthrene</td>
<td>191-26-4</td>
<td>42.20</td>
<td>0.33</td>
<td>1.65</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Naptho[1,2-b]fluoranthene</td>
<td>5385-22-8</td>
<td>44.20</td>
<td>1.67</td>
<td>5.00</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Naptho[2,3-j]fluoranthene</td>
<td>205-83-4</td>
<td>44.28</td>
<td>1.67</td>
<td>5.00</td>
<td>FT-MS Peak Assignment</td>
</tr>
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<td>Dibenz[a,e]fluoroanthene</td>
<td>5385-75-1</td>
<td>44.43</td>
<td>0.47</td>
<td>2.36</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Dibenz[a,l]pyrene</td>
<td>191-30-0</td>
<td>44.59</td>
<td>0.48</td>
<td>2.41</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Naptho[2,3-k]fluoranthene</td>
<td>207-18-1</td>
<td>44.84</td>
<td>1.67</td>
<td>5.00</td>
<td>FT-MS Peak Assignment</td>
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<tr>
<td>Naptho[2,3-e]pyrene</td>
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<td>5.00</td>
<td>FT-MS Peak Assignment</td>
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<td>45.50</td>
<td>6.44</td>
<td>32.22</td>
<td>FT-MS Peak Assignment</td>
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<td>Coronene</td>
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<td>FT-MS Peak Assignment</td>
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<tr>
<td>Dibenz[e,l]pyrene</td>
<td>192-51-8</td>
<td>45.72</td>
<td>1.67</td>
<td>5.00</td>
<td>FT-MS Peak Assignment</td>
</tr>
<tr>
<td>Naptho[2,3-a]pyrene</td>
<td>196-42-9</td>
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<td>1.67</td>
<td>5.00</td>
<td>FT-MS Peak Assignment</td>
</tr>
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<td>Benzo(b)perylene</td>
<td>197-70-6</td>
<td>45.93</td>
<td>1.67</td>
<td>5.00</td>
<td>FT-MS Peak Assignment</td>
</tr>
</tbody>
</table>
Table 2: Instrument Control Parameters (Copied from MassHunter acqmeth.txt)

<table>
<thead>
<tr>
<th>Instrument Control Parameters:</th>
<th>7000 QQQ</th>
</tr>
</thead>
</table>

D:\MassHunter\GCMS\1\methods\PAH-PSD-014_MRM.M
Thu Apr 03 12:54:04 2014

Control Information
-------

Sample Inlet : GC
Injection Source : GC ALS
Injection Location: Rear
Mass Spectrometer : Enabled

No Sample Prep method has been assigned to this method.

GC
Oven
Equilibration Time 0.5 min
Max Temperature 350 °C
Slow Fan Disabled
Oven Program On

Oven Program 60 °C for 1 min then 40 °C/min to 180 °C for 0 min
Oven#1 then 3 °C/min to 230 °C for 0 min
Oven#2 then 1.5 °C/min to 235 °C for 0 min
Oven#3 then 15 °C/min to 280 °C for 10 min
Oven#4 then 6 °C/min to 298 °C for 0 min
Oven#5 then 16 °C/min to 350 °C for 4 min
Oven#6
Run Time 47.25 min
Cryo Off

QQQ Collision Cell EPC
He Quench Gas On 2.25 mL/min
N2 Collision Gas On 1.5 mL/min

ALS
Back Injector
Syringe Size 10 μL
Injection Volume 1 μL
Solvent A Washes (PreInj) 2
Solvent A Washes (PostInj) 3
Solvent A Volume 8 μL
Solvent B Washes (PreInj) 2
Solvent B Washes (PostInj) 3
Solvent B Volume 8 μL
Sample Washes 0
Sample Wash Volume 8 μL
Sample Pumps 4
Dwell Time (PreInj) 0 min

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Dwell Time (PostInj)                         0 min
Solvent Wash Draw Speed                      300 μL/min
Solvent Wash Dispense Speed                  6000 μL/min
Sample Wash Draw Speed                      300 μL/min
Sample Wash Dispense Speed                   6000 μL/min
Injection Dispense Speed                    6000 μL/min
Viscosity Delay                             0 sec
Sample Depth                                Disabled
Injection Type                              Standard
L1 Airgap                                   0.2 μL

Tray                                         
Barcode heater                               Disabled
Barcode mixer                                Disabled

Sample Overlap                               
Mode                                         Sample overlap is not enabled

ALS Errors                                   
Pause for user interaction

Front MM Inlet He                            
***Excluded from Affecting GC’s                   

Readiness State***                           
Mode                                         Pulsed Splitless
Heater                                       Off
Pressure                                     Off
Total Flow                                   Off
Septum Purge Flow                            Off
Septum Purge Flow Mode                       Switched
Temperature Program                          Off
Temperature Program                          320 °C for 0 min
Run Time                                     47.25 min
Injection Pulse Pressure                     35 psi Until 0.3 min
Purge Flow to Split Vent                     50 mL/min at 0.7 min
Cryo                                         Off

Back SS Inlet He                             
Mode                                         Pulsed Splitless
Heater                                       On 320 °C
Pressure                                     On 17.677 psi
Total Flow                                   On 3 mL/min
Septum Purge Flow                            On 3 mL/min
Gas Saver                                    On 15 After 2 min mL/min
Injection Pulse Pressure                     35 psi Until 0.3 min
Purge Flow to Split Vent                     25 mL/min at 0.7 min

Thermal Aux 2 (MSD Transfer Line)            
Heater                                       On 320 °C

Column                                       
Column #1                                     
J&W CP7462                                   
Select PAH                                    
-60 °C–350 °C (350 °C): 30 m x 250 μm x 0.15 μm
In                                           Back SS Inlet He
Out                                          MSD
(Initial)                                    60 °C
Pressure                                     17.677 psi
Flow                                         2 mL/min
Average Velocity                             51.792 cm/sec

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<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
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<td>Holdup Time</td>
<td>0.96539 min</td>
</tr>
<tr>
<td>Flow Program</td>
<td>On</td>
</tr>
<tr>
<td>Flow Program (mL/min)</td>
<td>2 mL/min for 0 min</td>
</tr>
<tr>
<td>Run Time</td>
<td>47.25 min</td>
</tr>
<tr>
<td>APC</td>
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<tr>
<td>Aux EPC 4 H2</td>
<td>On</td>
</tr>
<tr>
<td>Pressure Program (psi)</td>
<td>50 psi for 0 min</td>
</tr>
<tr>
<td>Run Time</td>
<td>47.25 min</td>
</tr>
<tr>
<td>Aux EPC 5 He</td>
<td>Off</td>
</tr>
<tr>
<td>Pressure Program (psi)</td>
<td>5 psi for 0 min</td>
</tr>
<tr>
<td>Run Time</td>
<td>47.25 min</td>
</tr>
<tr>
<td>Aux EPC 6 H2</td>
<td>On</td>
</tr>
<tr>
<td>Pressure Program (psi)</td>
<td>2 psi for 0 min</td>
</tr>
<tr>
<td>Run Time</td>
<td>47.25 min</td>
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Table 3: Data Quality Objectives, Acceptance Criteria and Corrective Actions

<table>
<thead>
<tr>
<th>QC Sample</th>
<th>Purpose</th>
<th>Frequency</th>
<th>Acceptance Criteria</th>
<th>Corrective Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial Calibration</td>
<td>Accuracy</td>
<td>Prior to project analyses and if post-</td>
<td>$R^2$ of $\geq 0.99$ if linear, $\geq 0.995$ if quadratic</td>
<td>Perform instrument maintenance, and reanalyze</td>
</tr>
<tr>
<td></td>
<td></td>
<td>maintenance calibration check fails</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Instrument Detection Limits</td>
<td>Detection Limit</td>
<td>Prior to project analyses</td>
<td>Signal to noise (peak to peak ratio is above 3)</td>
<td>Perform instrument maintenance, Reanalyze</td>
</tr>
<tr>
<td>Continuing Calibration Verification</td>
<td>Accuracy</td>
<td>One per 10-15 samples, or as necessary</td>
<td>$\pm 30%$ of true value for at least $80%$ of target analytes</td>
<td>Perform instrument maintenance and re-analyze samples</td>
</tr>
<tr>
<td></td>
<td>despite matrix or instrument</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>variability</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Instrument Blank</td>
<td>Detection Limit, contamination</td>
<td>Beginning of each batch</td>
<td>Below analyte Instrument Detection Limit</td>
<td>Re-analyze, perform instrument maintenance, and/or flag data</td>
</tr>
<tr>
<td>Sample Spike</td>
<td>Preparation and handling bias</td>
<td>One per 25 Field Samples</td>
<td>Analyte percent recovery $50%$ to $150%$</td>
<td>Flag Data as appropriate</td>
</tr>
<tr>
<td>Internal Standard Peak Area</td>
<td>Analytical Bias</td>
<td>Each sample and standard</td>
<td>Percent detected $50%$ to $100%$</td>
<td>Re-analyze if a standard, perform instrument maintenance, and/or flag sample data</td>
</tr>
<tr>
<td>Surrogate Recovery</td>
<td>Analytical Bias</td>
<td>Each sample</td>
<td>Percent Recovery $50%$ to $150%$</td>
<td>Re-analyze if it is a blank or standard, flag sample data</td>
</tr>
</tbody>
</table>
Table 4: Quantifier and Qualifier Ions and Collision Cell Voltages for each compound

<table>
<thead>
<tr>
<th>Name</th>
<th>CAS #</th>
<th>RT</th>
<th>Quantifier Precursor Ion</th>
<th>Quantifier Product Ion</th>
<th>Collision Energy (V)</th>
<th>Qualifier Precursor Ion</th>
<th>Qualifier Product Ion</th>
<th>Collision Energy (V)</th>
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<tbody>
<tr>
<td>Naphthalene-D8 SS</td>
<td>1146-65-2</td>
<td>4.11</td>
<td>136</td>
<td>108</td>
<td>20</td>
<td>136</td>
<td>84</td>
<td>25</td>
</tr>
<tr>
<td>Naphthalene</td>
<td>91-20-3</td>
<td>4.12</td>
<td>128</td>
<td>102</td>
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<td>128</td>
<td>78</td>
<td>20</td>
</tr>
<tr>
<td>2-Methylnaphthalene</td>
<td>91-57-6</td>
<td>4.57</td>
<td>142</td>
<td>141</td>
<td>15</td>
<td>142</td>
<td>115.1</td>
<td>20</td>
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<td>1-Methylnaphthalene</td>
<td>90-12-0</td>
<td>4.69</td>
<td>142</td>
<td>141</td>
<td>15</td>
<td>142</td>
<td>115.1</td>
<td>20</td>
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<td>2-Ethylnaphthalene</td>
<td>939-27-5</td>
<td>5.00</td>
<td>141</td>
<td>115</td>
<td>15</td>
<td>156</td>
<td>141</td>
<td>15</td>
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<tr>
<td>2,6-Dimethylnaphthalene</td>
<td>28804-88-8</td>
<td>5.07</td>
<td>156</td>
<td>141</td>
<td>15</td>
<td>141</td>
<td>115</td>
<td>15</td>
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<td>1,6-Dimethylnaphthalene</td>
<td>575-43-9</td>
<td>5.22</td>
<td>156</td>
<td>141</td>
<td>15</td>
<td>141</td>
<td>115</td>
<td>15</td>
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<td>1,4-dimethylnaphthalene</td>
<td>571-58-4</td>
<td>5.37</td>
<td>156</td>
<td>141</td>
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Title: Calculation of Freely Dissolved Water Concentrations of Organic Compounds Derived from Polyethylene Passive Sampling Devices (PSD)

Scope
This method describes the calculations required for determining surface water and sediment porewater concentrations of polycyclic aromatic hydrocarbons (PAH), oxygenated polycyclic aromatic hydrocarbons (OPAHs), and pesticides that have been collected using polyethylene (PE) passive sampling devices (PSD). Prior analysis by applicable standard analytical method is required to determine analyte concentrations in the sampler.

Contributors: G. Sower, L. Quarles, K.A. Anderson

Responsibilities
Staff members involved in performing this work are responsible for reading, understanding and complying with the requirements for this SOP. The Director is responsible for ensuring that the content of this SOP is complied with and that qualified staff performs these analyses. The senior chemists are responsible for informing staff members of the requirements and interpretation of this SOP and are responsible for enforcing this SOP.

Status
This document is considered current standard operating procedure of the Food Safety and Environmental Stewardship Laboratory when management approval is documented by signature below. This Standard Operating Procedure is effective on the date of approval signature and supersedes all previous versions.

Approved

Kim A. Anderson, Ph.D. Director
Title Date

Historical File
Signature/Initials Date

I. Laboratory quantitation of analytes in PSDs
A. Fortification levels for one PSD:
1. PRC fortification solution must be prepared by a chemist in accordance with the needs of the project.
2. \[\frac{\text{std. vol. (µL) } \times \text{ std. conc. (µg/µL)}}{\text{wt. of sample (g*)}} = \text{fortification level (µg/g)}\]

B. Quantitative analysis to determine unknown sample levels:
1. See the specific analyte SOP analytical procedure for quantitative measurement.
II. Calculations using performance reference compounds (PRCs) [4].

A. The following calculations are performed using XLIMS data to excel feature. This process will place detection/reporting limit flagged concentrations from the instrument into the attached excel spreadsheet and produce one file per sample.

B. The chemist should verify:
   1. Quantity of PE PSDs
   2. PRC fortification levels
   3. Duration of deployment in days

C. Final values are stored in XLIMS using the resulting excel file.

*Note the calculation described does not account for temperature differences and assumes that your sampling system temperature range is between 2 and 30 C. “The experimental evidence suggests that K_{sw} values are not temperature dependent in the 2 to 30 C temperature range” Huckins et al 2006 page 53. See Huckins et al 2006 (and references therein), page 53-56 for explanation and how to account for temperature if required.

Calculation of Sampling Rate ($R_s$) of PRC’s:

$$R_{s,PRC} = \left( V_s \times 10^{log K_{sw,PRC} \times k_e} \right) / 1000$$

where the sampler water partition coefficient ($K_{sw}$) is given by:

$$log K_{sw} = a_0 + 2.321 (log K_{ow}) - 0.1618(log K_{ow})^2$$

$$a_0 = -2.61 \text{ for PAHs, PCBs, 4,4′ - DDE}$$
$$a_0 = -3.2 \text{ for polar pesticides}$$

(From Huckins page 54 equation 3.28)

the volume ($V_s$) of sampler is:

$$V_s = (# \text{ of PE Strips}) \times (5.0 \text{ cm}^3)$$

the PRC release rate constant ($k_e$) is estimated using:

$$k_e = -\ln \left( \frac{t_E}{t_0} \right) / E$$

where:

$$t_0 = \text{ PRC amount in PE at time = 0}$$
$$t_E = \text{ PRC amount in PE at time = E}$$
$$E = \text{ Exposure duration in days}$$

Calculation of Water Concentration ($C_{w,analyte}$) for Target Compounds:

$$C_{w,analyte} = \frac{\text{analyte accumulation}}{V_s \times K_{sw}(1 - \exp \left( - \frac{R_{s,analyte} \times E}{V_s \times K_{sw,analyte}} \right))} \times 1000$$
where the sampling rate of the target compound \((R_{s,\text{analyte}})\) is:

\[
R_{s,\text{analyte}} = R_{s,\text{PRC}} \times \left(\frac{a_{\text{analyte}}}{a_{\text{PRC}}}\right)
\]

and:

\[
\log a = 0.0130\log K_{ow}^3 - 0.3173\log K_{ow}^2 + 2.244\log K_{ow}
\]

*(From Huckins page 60 equation 3.35)*

*Remember to use appropriate \(K_{ow}\) for \(K_{sw,\text{PRC}}\) and \(K_{sw,\text{analyte}}\) calculations*

### III. References


7. *Food Safety Environmental Stewardship Program SOP 2120: Preparation of Polyethylene Passive Sampling Devices for Environmental Sampling Equipment.*
Table 1. Target PAH compounds with suggested PRC compound and estimated method detection limit (MDL) based on three week deployment with 5 PSDs composited in surface water and 1 PSD in sediment pore water at a Portland Harbor Superfund Site with analysis by SOP 418. \(K_{ow}\) values are from: US EPA. [2014]. Estimation Programs Interface Suite™ for Microsoft® Windows, v 4.10. United States Environmental Protection Agency, Washington, DC, USA.

<table>
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<tr>
<th>Compound</th>
<th>CAS #</th>
<th>log Kow</th>
<th>PRC</th>
<th>MDL (ng/L)</th>
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Table 2. Target OPAH compounds from SOP 414 with suggested PRC compounds. $K_{ow}$ values are from: US EPA. [2014]. Estimation Programs Interface Suite™ for Microsoft® Windows, v 4.10. United States Environmental Protection Agency, Washington, DC, USA.

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<th>PRC</th>
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