

GUIDANCE DOCUMENT

Passive PE Sampling in Support of In Situ Remediation of Contaminated Sediments: Standard Operating Procedure for PED Preparation

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Standard Operating Procedure for the Preparation of Polyethylene (PE) and Polyethylene Devices (PEDs) Used for Passive Sampling

1.0 SCOPE AND APPLICATION

- 1.1 This method describes a procedure for preparing and handling polyethylene (PE) films that will be cut into strips and used in polyethylene devices (PEDs) to passively sample hydrophobic organic compounds (HOCs) in environmental media.
- 1.2 This method generates PE that can be deployed within PEDs for passive sampling of HOCs in atmospheric, aqueous, or sediment-porewater systems.
- 1.3 PE that is prepared by this method is suitable for laboratory or *in situ* field deployment.

2.0 SUMMARY OF METHOD

- 2.1 A known mass of low density polyethylene (LDPE) sheet, usually gram quantities, is cleaned by sequentially extracting with methylene chloride, methanol, and ultrapure water in a closed glass vessel.
- 2.2 Clean PE is equilibrated with performance reference compounds (PRCs) dissolved in water or methanol-water (see Appendix 1 for possible PRCs).
- 2.3 Prepared PE is stored in contaminant-free, sealed, glass vessels.
- 2.4 Shortly before deployment, the PE is cut into strips and either placed in aluminum mesh bags for water sampling water or aluminum frames for sediment sampling. PEDs are transported to the field wrapped in clean aluminum foil.
- 2.5 In the field, the PE is exposed to the environmental medium of concern. HOCs in the medium diffuse into the PE, while PRCs diffuse out.

3.0 INTERFERENCES

- 3.1 PE is susceptible to contamination from atmospheric vapors and contact with surfaces (e.g., worker hands), so it must remain in clean sealed vessels until deployment.

4.0 APPARATUS AND MATERIALS

- 4.1 Extraction vessels: 1-L glass bottles or screw capped jars (foil-lined lids).
- 4.2 Storage vessels: bottles with glass stoppers or amber jars (foil-lined lids).
- 4.3 Bottle/jar tumbler, shaker table, bottle roller, or equivalent.
- 4.4 Low density polyethylene (LDPE): commercial grade, large sheet at 25 μ m (1 mil) or 51 μ m (2 mil) thickness. The thickness is chosen to be strong enough to withstand stresses during deployment (e.g., insertion into sediment), but thin enough to exchange a significant fraction (e.g., >20%) of its PRCs during the deployment time to be used.
- 4.5 Food grade aluminum foil (solvent cleaned and/or combusted to remove any organic residue from foil production)

- 4.6 Stainless steel forceps
- 4.7 Teflon (or similar non-contaminating material) cutting board

5.0 REAGENTS

- 5.1 Methylene chloride, CH₂Cl₂, pesticide grade or equivalent
- 5.2 Methanol, CH₃OH, pesticide grade or equivalent
- 5.3 Organic-free reagent water (as defined in SW-846 Chapter 1)
- 5.4 Research grade PRCs certified >98+% pure.

Note: Specific standard materials, concentrations, solvents, and solvent purity requirements will be determined based upon that target HOCs of concern for the particular application

6.0 PRESERVATION AND HANDLING

- 6.1 Clean PE should be stored in clean sealed glass vessels.
- 6.2 Until deployment, prepared PE (PE loaded with PRCs) is stored in sealed glass containers with a few mL of organic-free reagent water added to maintain 100% relative humidity within the storage vessels (minimizing sorptive losses of PRCs to glass vessel walls).
- 6.3 Laboratory and field personnel should wear nitrile or latex gloves whenever handling clean PE.
- 6.4 Methylene chloride-rinsed, stainless steel forceps and scissors are used when manipulation of clean PE is required.
- 6.5 Methylene chloride-rinsed, aluminum foil is used to cover any surface that clean PE may encounter.

7.0 PROCEDURE

- 7.1 Polyethylene Cleaning Procedure: LDPE is purchased from hardware/painting stores in large sheets ('dropcloth or plastic tarp' material) with thickness of 25 μ m (1 mil) or 51 μ m (2 mil), depending on the user's need for strength (choose thicker) and desire to use short deployment times (used thinner). The sheet is cut into strips sized for environment and frames to be used. An organic solvent cleaning sequence is then used to prepare the PE. This process ensures that extractable oligomers, plasticizers, and contaminating organic chemicals are removed from the PE prior to use. All extractions are performed sequentially in the same container.
 - 7.1.1 Methylene chloride is placed into the extraction vessel, and the PE strips are immersed in the container for 24 hours to enable time for diffusive transfers out of the PE. The initial methylene chloride extract is discarded and a second methylene chloride extraction is performed for 24 hours. The second methylene chloride extract is discarded and replaced by methanol in order to remove methylene chloride from the PE. Methanol immersion is also done for 24 hours. The initial methanol extract is discarded and followed by a second methanol soak for 24 hours. Finally, the second methanol extract is discarded and the PE undergoes three 24-hour soaks with organic-free reagent water (within the same

extraction vessel) to remove residual methanol from the PE.

7.1.2 The cleaned PE is stored in organic-free reagent water in the extraction vessel until further processing.

7.2 Polyethylene Preparation with Performance Recovery Compounds (PRCs): PRCs are loaded into the clean PE, prior to its field deployment, by utilizing either aqueous (Fernandez et al. 2009) or 80:20 methanol:water equilibrations (Booij et al., 2002). Depending on the hydrophobic organic compounds of interest, PRCs should be chosen which mimic mass transfer phenomena governing exchanges during field deployments. It is important to avoid adding PRCs that the analytical laboratory already uses as surrogate or injection standards. PRC loading is performed by placing the PE in pre-cleaned glass vessels containing known PRC solutions made up in organic-free reagent water with or without pesticide-grade methanol. The PE user should estimate the expected accumulation of target compounds in the passive sampler and seek to load with similar levels of PRCs to facilitate the eventual chemical analyses. Sufficient PRC equilibration time during this PE preparation step is necessary to ensure uniform PE loading across the entire PE thickness; hence thicker PE sheet is more robust for field use, but takes longer to load with PRCs.

7.2.1 Isotopically labeled compounds are useful internal standards when Gas Chromatography-Mass Spectrometry (GCMS) is the method of separation and detection. For example, deuterated polycyclic aromatic hydrocarbons (PAHs) and C13-labeled PCBs are effective methodological standards for PE passive sampling. One subset of compounds, distributed across the range of PAHs to be assessed (e.g., d10-phenanthrene, d10-pyrene, and d12-chrysene), should be used as PRCs, while another set (e.g., d10-anthracene, d10-fluoranthene, and d12-benz(a)anthracene) is used as surrogate (recovery) compounds during later analysis of field-deployed PE. Finally, compounds such as d10-acenaphthene, d14-*m*-terphenyl, and d12-perylene can be used as injection standards. Similar sets of labeled compounds should be used for other compound classes (see Appendix 1). Note: if PE samples are eventually to be analyzed at a contract laboratory, PRC choices must be made so as not to conflict with recovery and injection standards used by that laboratory.

7.2.2 As subsequent analysis (e.g., GCMS) is best achieved with both PRCs and target HOCs present at like concentrations in the PE extracts, the optimal concentration level of the PRC loaded into the PE is dependent on the environment in which the PE is to be deployed. For example, if a target HOC is expected to occur in the water or pore water near 1 ng/L levels, one can use that compound's LDPE-water partition coefficient (e.g., Fernandez et al., 2009; Lohmann, 2012) to estimate the expected levels in the PE after deployment:

$$\text{Concentration in PE (ng/kg)} \sim K_{LDPE-water} * \text{concentration in (pore)water (ng/L)}$$

So if the $K_{LDPE-water}$ for the target HOC of interest is 10^5 (L/kg), then the concentration of the target HOC in the PE will approach 100 ug/kg. Based on this estimate, the PRCs are loaded into the PE at similar concentrations. Appendix 2 shows a typical calculation used to design a PRC-containing MeOH:H₂O solution of PCBs suited for causing an 0.82 g strip of PE to acquire about 100 ug of each PRC per kg of PE (equivalent to 100 ng/g PE).

7.2.3 Aqueous PRC Loading: A solvent-cleaned and dried glass container is filled with ultrapure water that has been spiked with known concentrations of PRCs (e.g., using calculations like those shown in Appendix 2). A known mass of pre-cleaned PE is then added and weighted to insure complete PE submersion. The vessel is agitated to remove any air pockets adhering to the submerged PE. Equilibration times vary for different PRC/PE thickness combinations and the PE-water phase ratio. For PAHs and PCBs, use at least 30 days to insure homogeneous distributions of the PRCs throughout the entire thickness of the PE film unless faster equilibration has been confirmed. Confirmation can be done by time course measures of PRC concentrations in the PE or by showing that concentrations of PRCs are the same for films of different thicknesses, but the same masses. Generally, PE is stored in the PRC solution until it is to be deployed.

7.2.4 Methanol-Aided PRC Loading: A solvent-cleaned and dried glass container is filled with an 80:20 mixture of pesticide grade methanol and ultrapure water that has been spiked with known concentrations of PRCs (e.g., see calculations in Appendix 2). A known mass of pre-cleaned PE is then added and weighted to insure complete submersion. The vessel should be agitated to remove any air pockets adhering to the submerged PE. Equilibration times vary for different PRC/PE thickness combinations and the PE-solvent phase ratio, but typically this step is completed within 7 days since methanol swells the PE and thereby speeds PRC diffusion into the polymer sheet (Booij et al., 2002). Generally, the PE is stored in the PRC solution until shortly before it is to be deployed. Before deployment, the PRC-loaded PE is rinsed with ultrapure water, and then it is soaked in ultrapure water for 24 h to remove methanol from the PE. This methanol leaching step is repeated twice to insure complete methanol removal.

7.3 PED Assembly

7.3.1 PEDs can be pre-assembled with prepared PE strips up to a few days prior to deployment depending on the target compounds of interest.

7.3.2 FOR WATER SAMPLING WITH PE IN A STAINLESS STEEL MESH BAG. Since PE that is openly exposed in the water column has been observed to be eaten by aquatic organisms, the PE must be protected by deploying it in a mesh bag.

7.3.2.1 Cut rectangles from the mesh that are larger than the piece of PE to be deployed. Clean the mesh with methylene chloride, methanol, and water.

7.3.2.2 Wearing nitrile gloves, and using solvent-rinsed stainless steel forceps, lay a piece of the mesh on a clean surface such as an aluminum-foil covered lab bench. Remove the PE strip from its container and lay it on top of a stainless steel mesh. Place the second mesh on top. The two meshes are sealed together by folding the edges over on one another, and then sewing them together with nylon fishing line. Grommets can be added to the upper corners to facilitate mesh labeling and attachments in the field.

7.3.3 FOR SEDIMENT BED SAMPLING WITH PE IN AN ALUMINUM SHEET METAL FRAME. In order to insert the PE strips into a sediment bed, the PE must be carried by an aluminum frame (Figure 1).

7.3.3.1. Aluminum sheet metal is cut into two complementary pieces that can be bolted together such that a piece of PE sheet is held in place. After cutting, these pieces of aluminum must be washed with organic solvents (e.g., methylene chloride and methanol) and then rinsed with water.

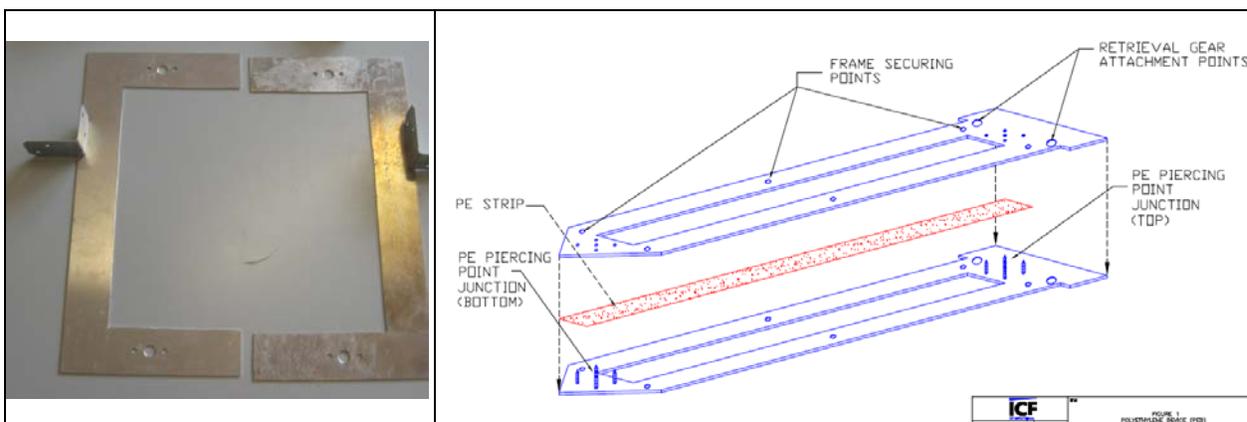


Figure 1.
(left panel) Aluminum sheet cut into two "C-shaped" pieces allowing the investigator to mount and hold ~25 cm strips of PE an open window when the two pieces are overlapped and bolted together.
(right panel) Drawing of two aluminum sheet pieces cut so as to sandwich a strip of PE and expose about 50 cm of length.

7.3.3.2 Wearing nitrile gloves, lay a piece of the aluminum frame containing the PE piercing points (sheet metal screws, see Figure 1), sharp side up, on a sheet of solvent-rinsed aluminum foil.

7.3.3.3 Using solvent-rinsed stainless steel forceps, remove the PE strip from its container and lay the strip lengthwise across both sets of PE piercing point junctions. PE strips should have been sized to fit the frame with a little extra length, allowing the investigator to cut a small strip of PE from one end to serve as sample for PRC concentration measures before the sampler is deployed. At one end of the PED frame, gently push the remainder of the PE strip onto the PE piercing points so all points penetrate the PE strip. Gently pull the other end of the PE strip over the adjacent PE piercing points, keeping the PE strip taut, and push that end of the PE strip into the PE piercing points. The tautness of the PE strip should have as minimal deflection as possible between the two PE piercing point junctions, but not too tight so that movement of the PE causes it to rip or tear. Place the other PED frame over the PED frame containing the PE strip so that each of the PE piercing point junctions meet and both PED frames are flush against each other. Secure the two frames together using the appropriate hardware (stainless steel machine screws, locking washers, and cap nuts).

7.3.3.4 Wrap the entire PED frame in solvent-rinsed aluminum foil to prevent exposure during transport and field preparation activities.

7.4 PE and PED Storage and Shipment:

7.4.1 Prepared PEDs in their foil envelopes may be stored a few days at ambient temperature prior to deployment. Freezing or excessive heat should be avoided to minimize the likelihood of changing the polymer crystallinity. It is recommended that PEDs be hand carried or shipped in a timely fashion (Overnight or Next Day if possible) to minimize chances sampler contamination or damage.

7.4.2 If PE is to be shipped to another location for PED assembly, it is recommended that the PE strips are individually sealed in pre-cleaned glass vials that contain a little water. Freeze shipping should be avoided, but cold (refrigeration temperature) packing may be necessary depending on time of season and individual laboratory handling/quality control procedures.

8.0 QUALITY CONTROL

8.1 PRC Loading Validation: At least six representative samples of prepared PE should be collected (e.g., 6 x 10 mg pieces), extracted, and analyzed prior to field deployment to validate that the PRC concentrations are consistent with their intended loadings and these standards have uniform concentrations in a batch of PE.

8.2 Target HOC Blanks: Subsamples of prepared PE, commensurate in size with the planned environmental PE samples (e.g., 10 cm wide by 5 cm long by 25 um thick and therefore weighing about 120 mg), should be collected, extracted, and analyzed prior to field deployment to demonstrate that other substances have not contaminated the PE which would contribute to interfering background for the target HOCs.

9.0 METHOD PERFORMANCE

9.1 PRC data, obtained from PE samples collected from >six parts of the prepared PE, should be consistent within about 10% (i.e., 100 x standard deviation / mean).

9.2 Target HOC concentrations should be undetectable in the prepared PE (e.g., < 1 ng/g PE assuming 100 mg PE subsamples).

10.0 REFERENCES

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- Booij, K, Smedes, F., van Weerlee, E.M., Chemosphere 2002, 46, 1157-1161.
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Appendix 1 Suggested Performance Reference Compounds (PRCs), Surrogate Compounds (Recovery Standards), and Injection Standards.

A. PRCs, suitable for polycyclic aromatic hydrocarbon (PAH) determinations when Gas Chromatography-Mass Spectrometry (GCMS) is the preferred method of detection, include, but are not restricted to, deuterated PAH compounds. One subset should be used as PRCs, while reserving others for use as surrogate (recovery) compounds. Still other compounds such as terphenyl can be used as injection standards.

Targets: PAHs	Method: GCMS			Detection Limit ~ 100 pg / 100 mg PE		
PRCs	d10-phenanthrene	d10-pyrene	d12-chrysene			
Surrogates	d10-anthracene	d10-fluoranthene	d12-benz(a)anthracene			
Injection Standards	d10-acenaphthene	d14- <i>m</i> -terphenyl	d12-perylene			

B. PRCs and surrogate compounds suitable for polychlorinated biphenyl (PCB) determinations when GCMS is the preferred method of detection include, but are not restricted to, ¹³C-labeled or deuterated PCB congeners. One subset, for example including a tri-, tetra-, penta-, hexa-, and heptachloro-biphenyl, can be used as PRCs while reserving different tri-, tetra-, penta-, hexa-, and heptachloro-biphenyl congeners to serve as surrogate compounds. Still other compounds such as deuterated PAHs or rare PCBs (not contained in Aroclor/Clophan mixtures such as: PCB-39, PCB-55, PCB-104, PCB-150 and PCB-188) can be used as injection standards.

Targets: PCBs	Method: GCMS						Detection Limit ~ 100 pg / 100 mg PE					
PRCs	¹³ C PCB-28	¹³ C PCB-52	¹³ C PCB-101	¹³ C PCB-153	¹³ C PCB-180							
Surrogates	¹³ C PCB-19	d ₆ PCB-77	¹³ C PCB-105	¹³ C PCB-167	¹³ C PCB-170	¹³ C PCB-194						
Injection Standards	d17-39	d22-104	d34-55	d40-150	d52-188							

C. When analyzing for organochlorine pesticides such as DDT using GCMS, ¹³C labeled compounds can serve as PRCs and surrogate standards. Since DDT has been seen to degrade to form DDE or DDD in certain situations, one should use the 4,4'- isomer of DDT and the 2,4'-isomers of DDE and DDD as PRCs to allow appearance of ¹³C-labelled 4,4'-DDE or 4,4'-DDD to be interpreted as arising from reaction of the DDT PRC during the deployment. Deuterated or ¹³C labeled PCBs can be used as surrogate (recovery) and injection standards.

Targets: DDTs	Method: GCMS			Detection Limit ~ 200 pg / 100 mg PE		
PRCs	¹³ C 2,4'-DDE	¹³ C 2,4'-DDD	¹³ C 4,4'-DDT			
Surrogates	¹³ C-PCB111	¹³ C-PCB153	¹³ C 2,4'-DDT			
Injection Standards	d6 PCB 77	¹³ C PCB 105	¹³ C PCB 167			

Appendix 2. Example of spreadsheet used to design solution needed to impregnate PE with Performance Reference Compounds (PRCs) for PCB sampling. The leftmost section uses data from Booij et al. (2002) to establish a correlation between log K(polyethylene-80:20 MeOH:H₂O) values and log K_{ow} values from Hawker and Connell (1988). With this relationship, the second section shows is use to estimate the PE-MeOH:H₂O partition coefficients for PRCs of interest. Using these partition coefficients and a user-chosen mass of PE to prepare (here 0.82 g), the third section allows the user to find the PRC spiking solution concentration needed to obtain any desired initial PE concentration (here set to be 100 ng each PRC per g PE); for example, for congener 52, one needs to have 11 ng/mL of the initial 80:20 MeOH:H₂O solution to end up with about 100 ng/g PE. Finally, the right-most section uses the polyethylene-water partition coefficients (from Lohmann 2012) to check the importance of PRC losses from the PE when the MeOH is leached out in three successive steps after PRC loading. Successive calculations are described in the text below.

Example spreadsheet calculation for spiking PCBs into LDPE with 80:20 methanol-water solutions.

Training data for estimation of K _{pe-meoH:H2O}			13C-labelled PRCs			use correlation to estimate			Solution concentration needed in ng/mL in order to get			fraction in PE after each water soak to remove MeOH				
PCB congener	meoh:water (ref 1)	log Kow (ref 2)	congener	log Kow (ref 2)	log Kpe-meoh:water(80:20)	for PE mass (g)	0.82042	100 ng/g PE	with VMeOH:water (mL)	125	ng/mL MeOH:H2O	congener	estim log Kpe-w log K _{pew} = 1.14*log Kow-1.14 (ref 3)	1st leach	2nd leach	3rd leach
4	0.20	4.65	52	5.84	0.97	0.058			11.29		52	5.52	0.9966	0.9932	0.9898	
29	1.05	5.6	101	6.38	1.26	0.107			6.15		101	6.13	0.9990	0.9980	0.9971	
155	1.29	6.41	153	6.92	1.55	0.188			3.49		153	6.75	0.9997	0.9994	0.9991	
204	1.67	7.3	180	7.36	1.78	0.284			2.31		180	7.25	0.9999	0.9998	0.9997	
			28	5.67	0.88	0.048			13.75		28	5.32	0.9950	0.9900	0.9850	
			47	5.85	0.98	0.059			11.16		47	5.53	0.9967	0.9934	0.9901	
			111	6.76	1.46	0.160			4.10		111	6.57	0.9996	0.9992	0.9988	
			153	6.92	1.55	0.188			3.49		153	6.75	0.9997	0.9994	0.9991	
			178	7.14	1.66	0.233			2.82		178	7.00	0.9998	0.9997	0.9995	
use to find following correlation: log K _{pe-mw(80:20)} = 0.532 (+/- 0.094) * log Kow(Hawker) - 2.133 (+/- 0.572) N = 4, R2 = 0.94, S.E. 0.18						PE mass										
references						number of strips	1									
1. Booij, K, Smedes, F., van Weerlee, E.M., Chemosphere 2002, 46, 1157-1161.						PE density (g/cm ³)	0.95									
2. Hawker DW and Connell DW. 1988. Environ. Sci. Technol. 22: 382-387.						PE thickness (cm)	0.00254	for 1 mil sheet								
3. Lohmann, R. Environ. Sci. & Technol.; 2012, 46, 606-618.						PE length (cm)	68									
						PE width (cm)	5									
						length*width*thickness *number of strips*density										
						mass of PE (g)	0.82									

Step 1: find/estimate PE-spiking solvent partition coefficients for PRCs in solvents of interest. Here 80:20 MeOH:H₂O values from Booi et al. (2002) are used to develop a LFER with K_{ow} values from the literature (Hawker and Connell, 1988); this relation is then used to estimate K_{pe-meoh:h2o} values for other PCB congeners.

Step 2: choose the size of PE needed for the sampling exercise (here a single 1 mil-thick strip of 5 cm width and 68 cm length) and solve for the PE mass (here 0.82 g). Also choose a vessel which is large enough in volume to fit the PE inside without extensive PE-PE surface contact, but small enough so that unacceptably expensive masses of the labeled PRCs are not used (here 125 mL ground glass stopped flask). For this PE mass and solution volume, use the PE-solution partition coefficients from step 1 to solve for the fractions of each PRC that will be in the PE at equilibrium using:

$$\text{fraction in PE} = 1 - (1 / (1 + \text{Mass}_{\text{pe}} * K_{\text{pe-solution}} / \text{Volume}_{\text{solution}}))$$

(e.g., 5.8% for congener #52)

Step 3. solve for spiking solution concentrations of PRCs that result in desired PRC loadings in the PE (here 100 ng/g_{PE}) using:

$$C_{\text{initial spiking solution}} = C_{\text{desired in PE}} * \text{Mass}_{\text{pe}} / \text{fraction in PE} / \text{Volume}_{\text{solution}}$$

(e.g., here find need about 11.3 ng congener #52 per mL to achieve 100 ng/g PE; this is concentration of the spiking solution that the investigator must make up to prepare PE for subsequent sampling at sites where it is expected that the (pore)water will cause the PE to accumulate about 10 to 100 ng of target PCBs/g_{PE}).

Step 4. PE is stored in the PRC loading solution until shortly before passive sampling use.

Step 5. if spiking solutions that contain organic cosolvents like MeOH were used, this MeOH must be leached out of the PE before it can be used for passive sampling. To insure that MeOH leaching will not substantially change PRC loading, calculate whether substantial fractions of the PRCs will be lost in subsequent steps required to leach the co-solvent from the PE. Since the leaching steps involve use of H₂O, use the PE-water partition coefficients; for PCBs, these are derived from a LFER found in the review by Lohmann (2012). With these values, we can solve for the fractional losses of individual PRCs to the leach water (assumes negligible MeOH builds up in the leach water) contained in 1000 mL ground glass stoppered flasks, using:

$$\text{fraction remaining in PE after a single leach step} = 1 - (1 / (1 + K_{\text{pe-H2O}} * \text{Mass}_{\text{pe}} / \text{Volume}_{\text{H2O}}))$$

(e.g., in this case for congener #52, one finds 99.66% of the PRC remains in the PE after the first leach. Two additional leaches lower this to 99.32% and 98.98%, respectively. More hydrophobic congeners are leached even less.)