APPENDIX D. PASSIVE SAMPLER STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE (SOP) LB-16

EX SITU DETERMINATION OF POREWATER PCBS USING POLYETHYLENE PASSIVE SAMPLERS

Note: This SOP is based upon guidance for the use of passive sampling in the evaluation of contaminated sediments (EPA et al., 2017).

METHOD DESCRIPTION

This SOP provides instruction on the use of polyethylene (PE) passives samplers in the laboratory for the estimation of polychlorinated biphenyl (PCB) porewater concentrations in sediments. PE samplers will be equilibrated with sediments for 1 month using an orbital shaker, ensuring that there is sufficient mass of PE sampler to achieve the lowest possible PCB detection limits, and utilizing sufficient mass of sediment in each exposure so the addition of the PE sampler does not deplete PCBs from the sediment significantly (i.e., < 1% of the mass of the PCBs in the sediment should be absorbed by the sampler).

Pre-cleaned and performance reference compound (PRC) impregnated PE sheets will be prepared by Axys Analytical Services Ltd. (Axys) using procedures described in Gschwend et al. 2012 (Attachment D1). The PE sheets will then be transported to Analytical Resources, Inc. (ARI) on ice as detailed in the accompanying Surface Sediment Quality Assurance Project Plan (QAPP), and stored in a freezer (<-4 °C) until the aqueous equilibrium exposures are started.

Sediment samples will be collected, transported and stored at ARI as detailed in the accompanying QAPP. Before starting the aqueous equilibrium exposures, a small sub-sample of each stored sediment sample will be taken to determine moisture content. This information will be used to determine the volume of sodium azide (biocide) solution to be added to the exposures to achieve a well-formed slurry (80% water content).

PCB analysis of the samplers will be conducted by Axys following methods described in the accompanying QAPP. PCB concentrations in the samplers will be used to estimate porewater concentration as described in the accompanying QAPP, using the physical and chemical properties presented in Table D-1.

EQUIPMENT

- Drying Oven: Capable of maintaining a temperature of 105 ±2°C
- Aluminum Weighing Boats
- Top loading balance (readability = 0.01 g, capacity = 4000 g)
- Desiccator
- Laboratory grade deionized water
- Sodium azide
- Top loading balance (readability = 0.0001 g, capacity = 200 g)
- Pre-cleaned, PRC impregnated PE sheets
- Orbital shaker (capacity = > 20 kg)
- Stainless steel scissors
- Methanol

PERSONAL PROTECTIVE EQUIPMENT (PPE)

The analyst will be attired in the following PPE:

- Safety glasses
- Lab coat
- Nitrile gloves
- Long pants
- Closed-toe shoes

ATTACHMENTS

- Attachment D1 (Gschwend et al., 2012)

MOISTURE CONTENT

Calibration

1. Verify the balance was calibrated by the laboratory prior to use.

Sample Preparation

1. Initiate a Percent Moisture Benchsheet and complete the required information.
2. Record the Analyst, Oven ID, Thermometer ID, and Balance ID on the Percent Moisture Benchsheet.
3. Label an aluminum dish with a unique numerical ID (1, 2, 3…) for each sample.
4. Weigh the aluminum dish using a calibrated analytical balance and record the weight measurement on the benchsheet to the nearest 0.01g in the "Tare" column.
5. Thoroughly mix the sample then measure 5-10 g of sample into the pre-weighed, pre-labeled aluminum dish. Weigh to the nearest 0.01 g and record the weight measurement onto the benchsheet in the "Wet Wt." column.
6. Place the dish in the drying oven maintained at a temperature of 105 ±2°C. Record the date and the time the samples were placed in the oven.
7. Dry the sample for a minimum of 12 hours but do not exceed 24 hours. After this time has elapsed, remove the samples from the oven and allow them to cool in a desiccator before weighing.
8. Reweigh the samples and record the weight measurements in the "Tare+Dry Wt." column of the benchsheet.
9. Record the oven temperature and time out of the oven on the benchsheet.
10. Calculate sediment moisture content using formula below and record on the benchsheet.

Calculations

$$\text{Percent moisture} = 100 - \left(\frac{[(Tare+Dry\ Wt.)-(Tare)]}{(Wet\ Wt.)}\ x\ 100\right)$$

AQUEOUS EQUILIBRIUM EXPOSURES

Passive Sampler Preparation

1. Prior to starting the equilibrium exposures, remove PE sheets from the freezer.

2. Cut PE sheets into 0.1 g PE strips using methanol wiped stainless steel scissors. Always handle the PE sheets and strips wearing nitrile gloves.

3. Wrap all PE strips in aluminum foil and store at 4ºC ± 2°C until all sediment slurries are ready as described below.

Exposure Preparation

1. Initiate an Exposure Setup Benchsheet and complete the required information.
2. Label 1 L wide mouth glass jars, with Teflon-lined caps, with unique sediment sample IDs.
3. Thoroughly homogenize the sediment samples with a stainless steel spoon in a stainless steel bowl, and place approximately 1 kg wet weight of each sample into its corresponding labeled jar.
4. Record the mass of sediment in each jar in the Exposure Setup Benchsheet to the nearest 0.1 g.
5. Accounting for the moisture content of each sediment sample, calculate the volume of 2 g/L sodium azide solution to be added to each glass jar to achieve a sediment slurry of 80% moisture content (see formula below). Add the corresponding volume of solution to each jar, and record the volume added in the Exposure Setup Benchsheet to the nearest ml.

$$\text{Volume (ml)} = (80\% - \text{percent moisture}) \times \text{sediment mass (g)}$$

6. Add a pre-weighed 0.1 g PE strip to each jar. Record the weight of each strip to 4 decimal points in the Exposure Setup Benchsheet.
7. Tightly seal the lid of each jar, and securely load all jars onto the orbital shaker. Jars should be positioned securely, ensuring they will be able to safely withstand 1 month of agitation without breakage.
8. Turn on the orbital shaker and gradually increase the shaking speed to around 100 rpm.
9. Monitor jars for the first hour to ensure jars remain securely in place during shaking, and make any packing adjustments if necessary.
10. Initiate a Daily Conditions Benchsheet and complete the required information.
11. Monitor temperature, shakers, and jars daily for 1 month, and record observations on the Daily Conditions Benchsheet. Jars should be maintained at room temperature (20-25°C) for the duration of the exposure.

Exposure Termination

1. After 1 month, stop the orbital shaker and remove all glass jars.
2. Gradually empty the contents of each jar into a beaker to recover each PE strip.
3. Rinse each PE strip with laboratory grade deionized water to remove all attached sediment.
4. Wipe each PE strip with clean laboratory wipes to remove any remaining sediment and water.
5. Wrap each PE strip in a clean labeled aluminum foil envelope. Insert each envelope in a resealable plastic bag with corresponding sample ID label.
6. Insert all resealable plastic bags into a larger resealable plastic bag, and place the large bag on ice in a cooler for transport to Axys for analysis. Sample packing, transport information, and sample custody procedures are described in accompanying QAPP.

QUALITY ASSURANCE AND QUALITY CONTROL SAMPLES

The following quality assurance and quality control samples will be included to support data validation and usability determination:

- Passive sampler exposure blank (5 percent frequency, or 1 per maximum 20 samples). For the laboratory blank, 900 ml laboratory grade deionized water and 100 ml of 2 g/L sodium azide solution will be added to a wide mouth glass jar. A pre-weighed 0.1 g PRC impregnated PE strip will be added to this jar, and the jar will be treated in the same way as jars containing sediment samples.

- Duplicate samples (5 percent frequency, or 1 per maximum 20 samples). Prepare duplicate aqueous equilibrium exposures for select samples.

DATA MANAGEMENT

All data will be recorded immediately, legibly and in ink on the appropriate benchsheets. Any recording mistakes will be struck out with a single line and initialed and dated by the analyst. Hard copies of completed forms will be compiled and stored. Forms will also be scanned electronically, and these electronic copies will be submitted to EPA as part of the Data Report.

WASTE DISPOSAL

The analyst will perform this analysis with the intent of obtaining the best quality analytical results while generating the minimum amount of waste.

Waste generated during this analysis will include the sediment from all aqueous equilibrium exposures. This waste will be composited and disposed appropriately per laboratory procedures.

REFERENCES

EPA, SERDP, ESTCP. 2017. Laboratory, field, and analytical procedures for using passive sampling in the evaluation of contaminated sediments: user's manual. EPA/600/R- 16/357. February 2017 final web version (1.0). US Environmental Protection Agency, US Department of Defense, Strategic Environmental Research and Development Program, and Environmental Security Technology Certification Program.

Gschwend P, MacFarlane J, Palaia K, Reichenbacher S, Gouveia D. 2012. Passive PE sampling in support of in situ remediation of contaminated sediments (standard operating procedure for the preparation of polyethylene devices). ESTCP Project ER-200915. SERDP/ESTCP

Table D1. Physicochemical Properties of PCBs

Constituent of Interest	CAS No.	log K_{ow}[a]	log K_{PEW} (L_W/kg_{PE})[a]	log D_{PE} (cm^2/s)[a]
PCB Congeners				
PCB 1	2051-60-7	4.46	4.17	-8.46
PCB 2	2051-61-8	4.69	4.40	-8.46
PCB 3	2051-62-9	4.69	4.40	-8.46
PCB 4	13029-08-8	4.65	4.36	-8.64
PCB 5	16605-91-7	4.97	4.68	-8.64
PCB 6	25569-80-6	5.06	4.77	-8.64
PCB 7	33284-50-3	5.07	4.78	-8.64
PCB 8	34883-43-7	5.07	4.78	-8.64
PCB 9	34883-39-1	5.06	4.77	-8.64
PCB 10	33146-45-1	4.84	4.55	-8.64
PCB 11	2050-67-1	5.28	4.99	-8.64
PCB 12	2974-92-7	5.22	4.93	-8.64
PCB 13	2974-90-5	5.29	5.00	-8.64
PCB 14	34883-41-5	5.28	4.99	-8.64
PCB 15	2050-68-2	5.3	5.01	-8.64
PCB 16	38444-78-9	5.16	4.87	-8.81
PCB 17	37680-66-3	5.25	4.96	-8.81
PCB 18	37680-65-2	5.24	4.95	-8.81
PCB 19	38444-73-4	5.02	4.73	-8.81
PCB 20	38444-84-7	5.57	5.28	-8.81
PCB 21	55702-46-0	5.51	5.22	-8.81
PCB 22	38444-85-8	5.58	5.29	-8.81
PCB 23	55720-44-0	5.57	5.28	-8.81
PCB 24	55702-45-9	5.35	5.06	-8.81
PCB 25	55712-37-3	5.67	5.38	-8.81
PCB 26	38444-81-4	5.6	5.31	-8.81
PCB 27	38444-76-7	5.44	5.15	-8.81
PCB 28	7012-37-5	5.67	5.38	-8.81
PCB 29	15862-07-4	5.6	5.31	-8.81
PCB 30	35693-92-6	5.44	5.15	-8.81
PCB 31	16606-02-3	5.67	5.38	-8.81
PCB 32	38444-77-8	5.44	5.15	-8.81
PCB 33	38444-86-9	5.6	5.31	-8.81
PCB 34	37680-68-5	5.66	5.37	-8.81
PCB 35	37680-69-6	5.82	5.53	-8.81
PCB 36	38444-87-0	5.88	5.59	-8.81
PCB 37	38444-90-5	5.83	5.54	-8.81
PCB 38	53555-66-1	5.76	5.47	-8.81
PCB 39	38444-88-1	5.89	5.60	-8.81
PCB 40	38444-93-8	5.66	5.37	-8.98
PCB 41	52663-59-9	5.69	5.40	-8.98
PCB 42	36559-22-5	5.76	5.47	-8.98
PCB 43	70362-46-8	5.75	5.46	-8.98
PCB 44	41464-39-5	5.75	5.46	-8.98
PCB 45	70362-45-7	5.53	5.24	-8.98
PCB 46	41464-47-5	5.53	5.24	-8.98
PCB 47	2437-79-8	5.85	5.56	-8.98
PCB 48	70362-47-9	5.78	5.49	-8.98
PCB 49	41464-40-8	5.85	5.56	-8.98
PCB 50	62796-65-0	5.63	5.34	-8.98
PCB 51	68194-04-7	5.63	5.34	-8.98
PCB 52	35693-99-3	5.84	5.55	-8.98
PCB 53	41464-41-9	5.62	5.33	-8.98
PCB 54	15968-05-5	5.21	4.92	-8.98

Table D1. Physicochemical Properties of PCBs

Constituent of Interest	CAS No.	log K_{ow}[a]	log K_{PEW} (L_W/kg_{PE})[a]	log D_{PE} (cm^2/s)[a]
PCB 55	74338-24-2	6.11	5.82	-8.98
PCB 56	41464-43-1	6.11	5.82	-8.98
PCB 57	74472-33-6	6.17	5.88	-8.98
PCB 58	41464-49-7	6.17	5.88	-8.98
PCB 59	74472-33-6	5.95	5.66	-8.98
PCB 60	33025-41-1	6.11	5.82	-8.98
PCB 61	33284-53-6	6.04	5.75	-8.98
PCB 62	54230-22-7	5.89	5.60	-8.98
PCB 63	74472-34-7	6.17	5.88	-8.98
PCB 64	52663-58-8	5.95	5.66	-8.98
PCB 65	33284-54-7	5.86	5.57	-8.98
PCB 66	32598-10-0	6.2	5.91	-8.98
PCB 67	73575-53-8	6.2	5.91	-8.98
PCB 68	73575-52-7	6.26	5.97	-8.98
PCB 69	60233-24-1	6.04	5.75	-8.98
PCB 70	32598-11-1	6.2	5.91	-8.98
PCB 71	41464-46-4	5.98	5.69	-8.98
PCB 72	41464-42-0	6.26	5.97	-8.98
PCB 73	74338-23-1	6.04	5.75	-8.98
PCB 74	32690-93-0	6.2	5.91	-8.98
PCB 75	32598-12-2	6.05	5.76	-8.98
PCB 76	70362-48-0	6.13	5.84	-8.98
PCB 77 - WHO 12	32598-13-3	6.36	6.07	-8.98
PCB 78	70362-49-1	6.35	6.06	-8.98
PCB 79	41464-48-6	6.42	6.13	-8.98
PCB 80	33284-52-5	6.48	6.19	-8.98
PCB 81 - WHO 12	70362-50-4	6.36	6.07	-8.98
PCB 82	52663-62-4	6.2	5.91	-9.16
PCB 83	60145-20-2	6.26	5.97	-9.16
PCB 84	52663-60-2	6.04	5.75	-9.16
PCB 85	65510-45-4	6.3	6.01	-9.16
PCB 86	55312-69-1	6.23	5.94	-9.16
PCB 87	38380-02-8	6.29	6.00	-9.16
PCB 88	55215-17-3	6.07	5.78	-9.16
PCB 89	73575-57-2	6.07	5.78	-9.16
PCB 90	68194-07-0	6.36	6.07	-9.16
PCB 91	68194-05-8	6.13	5.84	-9.16
PCB 92	52663-61-3	6.35	6.06	-9.16
PCB 93	73575-56-1	6.04	5.75	-9.16
PCB 94	73575-55-0	6.13	5.84	-9.16
PCB 95	38379-99-6	6.13	5.84	-9.16
PCB 96	73575-54-9	5.71	5.42	-9.16
PCB 97	41464-51-1	6.29	6.00	-9.16
PCB 98	60233-25-2	6.13	5.84	-9.16
PCB 99	38380-01-7	6.39	6.10	-9.16
PCB 100	39485-83-1	6.23	5.94	-9.16
PCB 101	37680-73-2	6.38	6.09	-9.16
PCB 102	68194-06-9	6.16	5.87	-9.16
PCB 103	60145-21-3	6.22	5.93	-9.16
PCB 104	56558-16-8	5.81	5.52	-9.16
PCB 105 - WHO 12	32598-14-4	6.65	6.36	-9.16
PCB 106	70424-69-0	6.64	6.35	-9.16
PCB 107	70424-68-9	6.71	6.42	-9.16
PCB 108	70362-41-3	6.71	6.42	-9.16
PCB 109	74472-35-8	6.48	6.19	-9.16

Table D1. Physicochemical Properties of PCBs

Constituent of Interest	CAS No.	log K_{ow} [a]	log K_{PEW} (L_W/kg_{PE}) [a]	log D_{PE} (cm^2/s) [a]
PCB 110	38380-03-9	6.48	6.19	-9.16
PCB 111	39635-32-0	6.76	6.47	-9.16
PCB 112	74472-36-9	6.45	6.16	-9.16
PCB 113	68194-10-5	6.54	6.25	-9.16
PCB 114 - WHO 12	74472-37-0	6.65	6.36	-9.16
PCB 115	74472-38-1	6.49	6.20	-9.16
PCB 116	18259-05-7	6.33	6.04	-9.16
PCB 117	68194-11-6	6.46	6.17	-9.16
PCB 118	31508-00-6	6.74	6.45	-9.16
PCB 119	56558-17-9	6.58	6.29	-9.16
PCB 120	68194-12-7	6.79	6.50	-9.16
PCB 121	56558-18-0	6.64	6.35	-9.16
PCB 122	76842-07-4	6.64	6.35	-9.16
PCB 123 - WHO 12	65510-44-3	6.74	6.45	-9.16
PCB 124	70424-70-3	6.73	6.44	-9.16
PCB 125	74472-39-2	6.51	6.22	-9.16
PCB 126 - WHO 12	57465-28-8	6.89	6.60	-9.16
PCB 127	39635-33-1	6.95	6.66	-9.16
PCB 128	38380-07-3	6.74	6.45	-9.33
PCB 129	55215-18-4	6.73	6.44	-9.33
PCB 130	52663-66-8	6.8	6.51	-9.33
PCB 131	61798-70-7	6.58	6.29	-9.33
PCB 132	38380-05-1	6.58	6.29	-9.33
PCB 133	35694-04-3	6.86	6.57	-9.33
PCB 134	52704-70-8	6.55	6.26	-9.33
PCB 135	52744-13-5	6.64	6.35	-9.33
PCB 136	38411-22-2	6.22	5.93	-9.33
PCB 137	35694-06-5	6.83	6.54	-9.33
PCB 138	35065-28-2	6.83	6.54	-9.33
PCB 139	56030-56-9	6.67	6.38	-9.33
PCB 140	59291-64-4	6.67	6.38	-9.33
PCB 141	52712-04-6	6.82	6.53	-9.33
PCB 142	41411-61-4	6.51	6.22	-9.33
PCB 143	68194-15-0	6.6	6.31	-9.33
PCB 144	68194-14-9	6.67	6.38	-9.33
PCB 145	74472-40-5	6.25	5.96	-9.33
PCB 146	51908-16-8	6.89	6.60	-9.33
PCB 147	68194-13-8	6.64	6.35	-9.33
PCB 148	74472-41-6	6.73	6.44	-9.33
PCB 149	38380-04-0	6.67	6.38	-9.33
PCB 150	68194-08-1	6.33	6.04	-9.33
PCB 151	52663-63-5	6.64	6.35	-9.33
PCB 152	68194-09-2	6.22	5.93	-9.33
PCB 153	35065-27-1	6.92	6.63	-9.33
PCB 154	60145-22-4	6.76	6.47	-9.33
PCB 155	33979-03-2	6.41	6.12	-9.33
PCB 156 - WHO 12	38380-08-4	7.18	6.89	-9.33
PCB 157 - WHO 12	69782-90-7	7.18	6.89	-9.33
PCB 158	74472-42-7	7.02	6.73	-9.33

Table D1. Physicochemical Properties of PCBs

Constituent of Interest	CAS No.	log K_{ow} [a]	log K_{PEW} (L_W/kg_{PE}) [a]	log D_{PE} (cm^2/s) [a]
PCB 159	39635-35-3	7.24	6.95	-9.33
PCB 160	41411-62-5	6.93	6.64	-9.33
PCB 161	74472-43-8	7.08	6.79	-9.33
PCB 162	39635-34-2	7.24	6.95	-9.33
PCB 163	74472-44-9	6.99	6.70	-9.33
PCB 164	74472-45-0	7.02	6.73	-9.33
PCB 165	74472-46-1	7.05	6.76	-9.33
PCB 166	41411-63-6	6.93	6.64	-9.33
PCB 167 - WHO 12	52663-72-6	7.27	6.98	-9.33
PCB 168	59291-65-5	7.11	6.82	-9.33
PCB 169	32774-16-6	7.42	7.13	-9.33
PCB 170	35065-30-6	7.27	6.98	-9.4
PCB 171	52663-71-5	7.11	6.82	-9.4
PCB 172	52663-74-8	7.33	7.04	-9.4
PCB 173	68194-16-1	7.02	6.73	-9.4
PCB 174	38411-25-5	7.11	6.82	-9.4
PCB 175	40186-70-7	7.17	6.88	-9.4
PCB 176	52663-65-7	6.76	6.47	-9.4
PCB 177	52663-70-4	7.08	6.79	-9.4
PCB 178	52663-67-9	7.14	6.85	-9.4
PCB 179	52663-64-6	6.73	6.44	-9.4
PCB 180	35065-29-3	7.36	7.07	-9.4
PCB 181	74472-47-2	7.11	6.82	-9.4
PCB 182	60145-23-5	7.2	6.91	-9.4
PCB 183	52663-69-1	7.2	6.91	-9.4
PCB 184	74472-48-3	6.85	6.56	-9.4
PCB 185	52712-05-7	7.11	6.82	-9.4
PCB 186	74472-49-4	6.69	6.40	-9.4
PCB 187	52663-68-0	7.17	6.88	-9.4
PCB 188	74487-85-7	6.82	6.53	-9.4
PCB 189 - WHO 12	39635-31-9	7.71	7.42	-9.4
PCB 190	41411-64-7	7.46	7.17	-9.4
PCB 191	74472-50-7	7.55	7.26	-9.4
PCB 192	74472-51-8	7.52	7.23	-9.4
PCB 193	69782-91-8	7.52	7.23	-9.4
PCB 194	35694-08-7	7.8	7.51	-9.66
PCB 195	52663-78-2	7.56	7.27	-9.66
PCB 196	42740-50-1	7.65	7.36	-9.66
PCB 197	33091-17-7	7.3	7.01	-9.66
PCB 198	68194-17-2	7.62	7.33	-9.66
PCB 199	52663-75-9	7.2	6.91	-9.66
PCB 200	52663-73-7	7.27	6.98	-9.66
PCB 201	40186-71-8	7.62	7.33	-9.66
PCB 202	2136-99-4	7.24	6.95	-9.66
PCB 203	52663-76-0	7.65	7.36	-9.66
PCB 204	74472-52-9	7.3	7.01	-9.66
PCB 205	74472-53-0	8	7.71	-9.66

Table D1. Physicochemical Properties of PCBs

Constituent of Interest	CAS No.	log K_{ow}[a]	log K_{PEW} (L_W/kg_{PE})[a]	log D_{PE} (cm^2/s)[a]
PCB 206	40186-72-9	8.09	7.80	-9.83
PCB 207	52663-79-3	7.74	7.45	-9.83
PCB 208	52663-77-1	7.71	7.42	-9.83
PCB 209	2051-24-3	8.18	7.89	-10

Notes:

PCB = polychlorinated biphenyl

K_{OW} = Octanol to Water Partition Constant

K_{PEW} = Polyethylene to Water Partition Constant

D_{PE} = Polyethylene Diffusivity

SDL = Sample Detection Limit

[a] Default values in PRC calculator: Gschwend, P.M., P. Tcaciuc, and J.N. Apell. 2014. Passive PE sampling in support of in situ remediation of contaminated sediments – Passive sampler PRC calculation software user’s guide. ESTCP Project ER-200915..

[b] Provided by AXYS

[c] Assuming 0.1 gram of PE and that full equilibrium is reached. Note that full equilibrium may not be achieved for all congeners.

PERCENT MOISTURE BENCHSHEET

Pan ID	Sample ID	Tare (g)	Wet Wt. (g)	Tare + Dry Wt. (g)	Dry Wt. (g)	% Moisture

Oven ID: ____________________ **Thermometer ID:** ______________

Date/Time In: ____________________ **Temp In:** ______________

Date/Time Out: ____________________ **Temp Out:** ______________

Analyst: ____________________ **Balance ID:** ______________ **Calibration Date** ______________

EXPOSURES SETUP BENCHSHEET

Sample ID	Sediment Mass (g ww)	Passive Sampler Mass (g)	Biocide Solution Volume (mL)

Start Date: ____________________

Stop Date: ____________________

Analyst: ____________________

DAILY CONDITIONS BENCHSHEET

Date	Room Temperature	Orbital Shaker Performance Acceptable (Y/N)	Jars and Lids Intact (Y/N)	Comments	Analyst

Thermometer ID:_____________

Orbital Shaker ID:_____________

Attachment D-1

GUIDANCE DOCUMENT

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

ESTCP Project ER-200915

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13. SUPPLEMENTARY NOTES

14. ABSTRACT

The purpose of this Standard Operating Procedure (SOP) is to provide a description of the methods used in the deployment and retrieval of polyethylene devices (PEDs) in sediment-pore water environments for the sampling of hydrophobic organic compounds (HOCs) such as polycyclic aromatic hydrocarbons (PAHs) and polychlorinated biphenyls (PCBs). The overall objective of PED sampling is to determine the horizontal and vertical distributions of HOCs in pore waters of bed sediments. This SOP does not discuss deployment of PEDs in atmospheric or aqueous systems. This SOP should be used in conjunction with companion SOPs for Preparation of Polyethylene (PE) Media and Extraction Procedures for PE Media.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			**17. LIMITATION OF ABSTRACT**	**18. NUMBER OF PAGES**	**19a. NAME OF RESPONSIBLE PERSON**
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Reset

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 25μm (1 mil) or 51μm (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, CH_2Cl_2, pesticide grade or equivalent

5.2 Methanol, CH_3OH, 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 25μm (1 mil) or 51μm (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 placed 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:

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

So if the $K_{LDPE\text{-}water}$ 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 $MeOH:H_2O$ 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.

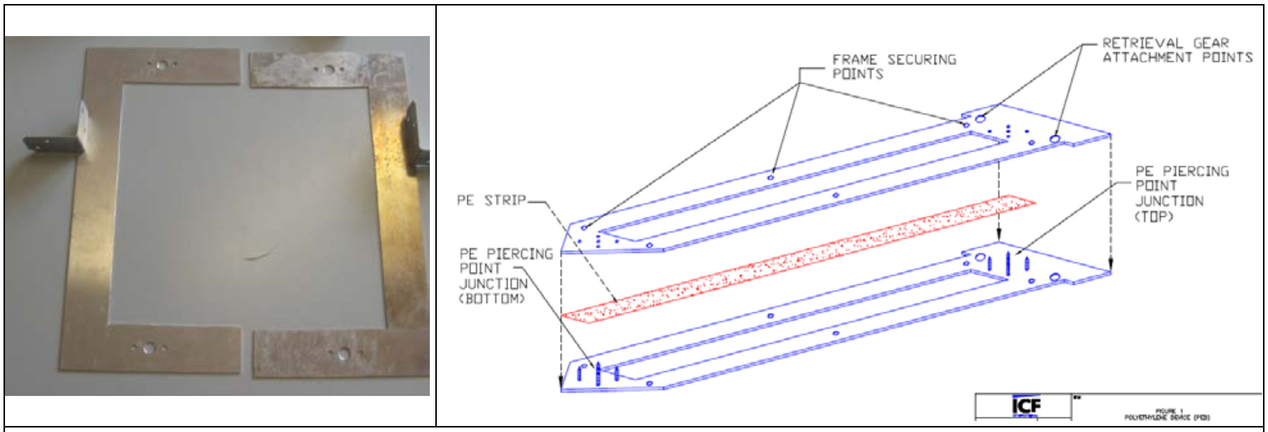

Figure 1.
(left panel) Aluminum sheet cut into two "C-shaped" pieces allowing the investigator to mount and hold ~25 cm strips of PE an open window when the two pieces are overlapped and bolted together.
(right panel) Drawing of two aluminum sheet pieces cut so as to sandwich a strip of PE and expose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

Adams, R.G., Lohmann, R., Fernandez L.A., MacFarlane, J.K., and Gschwend, P.M., Environ. Sci. & Technol. 2007, 41, 1317-1323.

Booij, K, Smedes, F., van Weerlee, E.M., Chemosphere 2002, 46, 1157-1161.

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Hawker DW and Connell DW. 1988. Environ. Sci. Technol. 22: 382-387.

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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-*m*-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, ^{13}C-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, ^{13}C 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 ^{13}C-labelled 4,4'-DDE of 4,4'-DDD to be interpreted as arising from reaction of the DDT PRC during the deployment. Deuterated or ^{13}C 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:H_2O 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 Kpe-meoh:H2O		**13C-labelled PRCs**		**use correlation to estimate**	**fraction in PE**		**Solution concentration needed in ng/mL in order to get**			**fraction in PE after each water soak to remove MeOH**		
						for PE mass (g)	**0.82042**	**100**	**ng/g PE**	**estim log Kpe-w**	**1st leach**	**2nd leach**	**3rd leach**
PCB congener	**log Kpe-meoh:water (ref 1)**	**log Kow (ref 2)**	**congener**	**log Kow (ref 2)**	**log Kpe-meoh:water(80:20)**	**with VMeOH:water (mL)**	**125**	**ng/mL MeOH:H2O**	**congener**	**log Kpew = 1.14*log Kow-1.14 (ref 3)**	**using**	**1000**	**mL water**
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 correlaltion:

log Kpe-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

number of strips	1	
PE density (g/cm3)	0.95	
PE thickness (cm)	0.00254	for 1 mil sheet
PE length (cm)	68	
PE width (cm)	5	
length*width*thickness *number of strips*density		
mass of PE (g)	**0.82**	

references

1. Booij, K, Smedes, F., van Weerlee, E.M., Chemosphere 2002, 46, 1157-1161.
2. Hawker DW and Connell DW. 1988. Environ. Sci. Technol. 22: 382-387.
3. Lohmann, R. Environ. Sci. & Technol.; 2012, 46, 606–618.

Step 1: find/estimate PE-spiking solvent partition coefficients for PRCs in solvents of interest. Here 80:20 MeOH:H_2O values from Booij 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 $K_{pe\text{-}meoh:h2o}$ 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:

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

(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:

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

(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 H_2O, 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:

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

(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.)