Electronic Supplementary Information (ESI)

Transferring mixtures of chemicals from sediment to a bioassay using silicone-based passive sampling and dosing

Lukas Mustajärvi[a], Ann-Kristin Eriksson-Wiklund[a], Elena Gorokhova[a], Annika Jahnke[b], and Anna Sobek[a]

[a]*Department of Environmental Science and Analytical Chemistry (ACES), Stockholm University, Sweden*

[b]*Department of Cell Toxicology, Helmholtz Centre for Environmental Research - UFZ, Germany*

* Corresponding author.

E-mail addresses:

Lukas.Mustajarvi@aces.su.se (L. Mustajärvi)*
Ann-Kristin. Eriksson-Wiklund@aces.su.se (A.-K. Eriksson-Wiklund)
Elena.Gorokhova@aces.su.se (E. Gorokhova)
Annika.Jahnke@ufz.de (A. Jahnke)
Anna.Sobek@aces.su.se (A. Sobek)

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Table ESI 1. Spiking solutions with respective chemicals and concentrations [ng µL^{-1}]

Spiking solution	Chemical	ng µL-1
1	PCB 52	24
2	PCB 28, PCB 52, benzo(a)pyrene	68, 118, 84
3	PCB 28, benzo(a)pyrene	134, 173
4	anthracene, pyrene	1.9, 4.9
5	anthracene, pyrene	3.6, 9.8

Table ESI 2 .Chemicals used to investigate changes in the chemical composition after repeated usage of passive dosing vials and their respective log K_{ow}.

	log K_{ow}	Ref.
Monochlorphenol (4-)	2.39	1
Dicholorphenol (2,6-)	2.75	1
Dicholorphenol (3,5-)	3.62	1
γ -Hexachlorocyclohexane (γ-HCH)	3.72	1
Acenaphthene	3.97	2
Triclosan	4.76	3, 4
Pyrene	5.06	2
PCB 28	5.92	5
PCB 153	7.31	5

Table ESI 3. ANOVA test statistics output, testing differences in the percentage of dead algal cells in controls between experimental runs. SS=Sum of squares, DF= degree of freedom, MS=mean of squares, F= test statistics, p= value; significance level p<0.05. The data were Box-Cox transformed to approach normal distribution.

	SS	DF	MS	F	p
Intercept	0.000074	1	0.000074	10.21	0.01
experiment	0.000024	3	0.000008	1.09	0.41
Error	0.000058	8	0.000007		

Table ESI 4. Output of the Generalized Linear Model (GLM) with normal error structure and log-link output that was applied to test the effect of the exposure level on the percentage of dead cells in the population (A) (degree of freedom = 22) and the corresponding normal probability plot of residuals (B); significance level p<0.05. The data were Box-Cox transformed to approach normal distribution.

A)

	Estimate	Standard	Wald	p value
Intercept	-13.1576	2.5384	26.86	< 0.0001
exposure level	4.2238	1.1882	12.63	0.0004
Scale	0.0034	0.0005	48.00	< 0.0001

B)

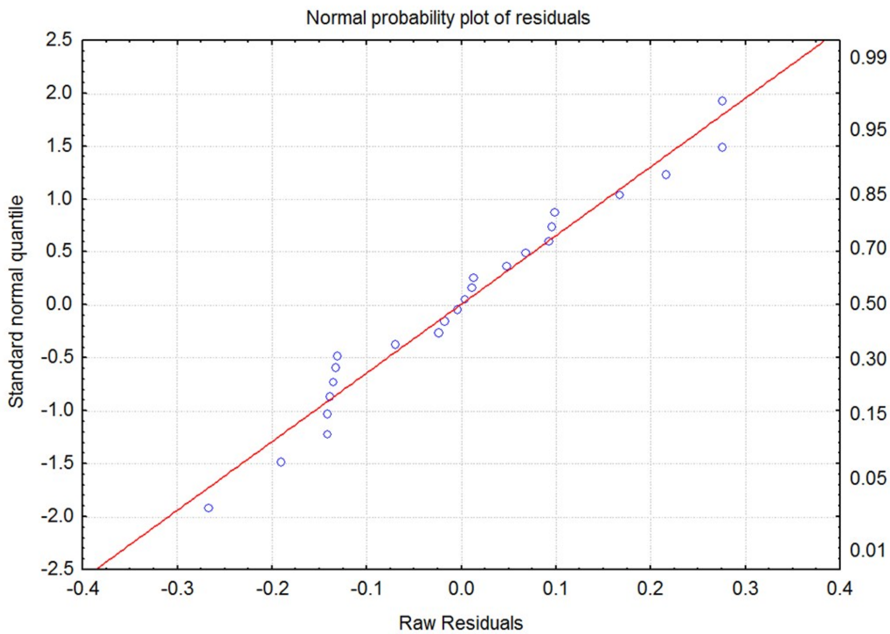

Table ESI 5. Unpaired t-test output, testing the difference in between the percentage of dead algal cells between the ambient exposure level (1:1) and the corresponding control. t-value= test statistics, DF= degree of freedom, p= value; significance level p<0.05.

Mean control	Mean 1:1 exposure	t-value	DF	p
0.001990	0.003621	-1.63187	4	0.178044

Table ESI 6. Pore water concentration of PAH and PCBs in sediment (0-20 cm) sampled in Ålöfjärden, Baltic Sea, measured with coated jars, given with standard deviations (n = 3). $K_{pdms\text{-}w}$, from the regression in ref.[6], K_{ow} for PAHs from ref.[2] and for PCBs from ref.[5]

	Pore water concentration [pg/L]
Naphthalene	< LOD
Acenaphthene	12000 ± 2800
Fluorene	9200 ± 970
Phenanthrene	13000 ± 1700
Anthracene	15000 ± 1400
Fluoranthene	110000 ± 7900
Pyrene	77000 ± 4800
Benzo(a)anthracene	5900 ± 350
Chrysene	7100 ± 540
Benzo(b)fluoranthene	5600 ± 350
Benzo(k)fluoranthene	1600 ± 110
Benzo(a)pyrene	1800 ± 110
Indeno(1.2.3-cd)pyrene	410 ± 34
Dibenz(a,h)anthracene	79 ± 15
Benzo(g,h,i)perylene	630 ± 100
PCB 28	48 ± 4.1
PCB 52	45 ± 2.6
PCB 101	8.5 ± 0.57
PCB 118	1.1 ± 0.098
PCB 153	2.0 ± 0.24

PCB 138	2.6 ± 0.25
PCB 180	0.55 ± 0.064

< LOD, below the limit of detection.

Table ESI 7 Polymer-water partition coefficients ($K_{p\text{-}w}$) compiled in ref.[7] and the references therein, and from other research studies. A ± indicates reported measure of variance. SD = Standard deviation. RSD = Relative standard deviation. SE = Standard error. CI = Confidence interval. Min = Minimum. Max =Maximum. -- = no data. Type of sampler, coating thickness and manufacturer.

Anthracene

log $K_{p\text{-}w}$	±		**ref**
3.20	± 10-15 % RSD	Fiber 7 µm PDMS Supelco	[8]
3.46	± 10-15 % RSD	Fiber 100 µm PDMS Supelco	[8]
4.38	--	Fiber 7 µm PDMS Supelco	[9]
4.31	--	Fiber 100 µm PDMS Supelco	[9]
3.82	± 0.03 SD	Fiber 28.5 µm PDMS Poly Micro Industries	[10]
4.12	--	Fiber 100 µm PDMS Supleco	[11]
4.19	± 0.08 SD	Fiber 10 µm PDMS Fiber guide	[12]
3.93	3.88, 3.98 (min, max)	Sheet 1 mm PDMS Specialty Silicone Products	[13]
3.98	3.88, 4.06 (min, max)	Sheet 1 mm PDMS Specialty Silicone Products	[13]
4.17	± 9-30 % RSD	Fiber 7 µm PDMS Supelco	[14]
4.08	± 0.05 95% CI	Sheet 0.4 mm silicon rubber Silastic A Dow Corning	[15]
3.92	± 0.05 95% CI	Sheet 0.5 mm silicon rubber SR Batch 0 Vizo Zeewolde	[15]
4.18	± 0.03 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	[15]
3.91	± 0.04 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	[15]
4.21	± 0.03 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	[15]

4.20	± 0.03 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
4.21	± 0.04 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
4.09	± 0.12 SE	Casted vial PDMS MDX4-4210 Dow corning	16

Pyrene

log $K_{p\text{-}w}$	±		**ref**
3.72	± 10-15 % RSD	Fiber 7 µm PDMS Supelco	8
3.79	± 10-15 % RSD	Fiber 100 µm PDMS Supelco	8
4.68	--	Fiber 7 µm PDMS Supelco	9
4.49	--	Fiber 100 µm PDMS Supelco	9
4.25	± 0.01 SD	Fiber 28.5 µm PDMS Poly Micro Industries	10
4.4	--	Fiber 100 µm PDMS Supleco	11
4.73	± 0.06 SD	Fiber 10 µm PDMS Fiberguide	12
4.27	4.24, 4.29 (min, max)	Sheet 1 mm PDMS Specialty Silicone Products	13
4.36	4.26, 4.45 (min, max)	Sheet 1 mm PDMS Specialty Silicone Products	13
4.63	± 9-30 % RSD	Fiber 7 µm PDMS Supelco	14
4.36	± 0.031	Fiber 28.5 µm PDMS (Poly Micro Industries ?)	17
4.22	± 0.05 SD	Fiber 28.5 µm PDMS Poly Micro Industries	18
4.56	± 0.07 95% CI	Sheet 0.4 mm silicon rubber Silastic A Dow Corning	15
4.38	± 0.08 95% CI	Sheet 0.5 mm silicon rubber SR Batch 0 Vizo Zeewolde	15
4.64	± 0.03 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	15
4.38	± 0.04 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	15
4.69	± 0.06 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
4.67	± 0.04 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
4.67	± 0.04 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
4.77	± 0.06 SE	Casted vial PDMS MDX4-4210 Dow corning	16

Benzo(a)pyrene

log $K_{p\text{-}w}$	±		**ref**
4.66	± 10-15 % RSD	Fiber 7 µm PDMS Supelco	8

5.27	--	Fiber 7 µm PDMS Supelco	9
4.99	--	Fiber 100 µm PDMS Supelco	9
5.18	± 0.02 SD	Fiber 28.5 µm PDMS Poly Micro Industries	10
5.22	± 0.15 SD	Fiber 10 µm PDMS Fiberguide	12
5.36	5.26, 5.44 (min, max)	Sheet 1 mm PDMS Specialty Silicone Products	13
5.09	4.98, 5.18 (min, max)	Sheet 1 mm PDMS Specialty Silicone Products	13
5.19	± 9-30 % RSD	Fiber 7 µm PDMS Supelco	14
4.59	± 0.05 SD	Fiber 28.5 µm PDMS Poly Micro Industries	18
6.06	± 0.11 SD	Fiber 7 µm PDMS Supelco	19
6.06	± 0.15 SD	Fiber 30 µm PDMS Supelco	19
5.55	± 0.11 95% CI	Sheet 0.4 mm silicon rubber Silastic A Dow Corning	15
5.22	± 0.06 95% CI	Sheet 0.5 mm silicon rubber SR Batch 0 Vizo Zeewolde	15
5.65	± 0.05 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	15
5.22	± 0.04 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	15
5.71	± 0.05 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
5.7	± 0.03 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
5.67	± 0.05 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
5.66	± 0.15 SE	Casted vial PDMS MDX4-4210 Dow corning	16

PCB 28

log $K_{p\text{-}w}$	±		ref
4.65	± 10-15 % RSD	Fiber 7 µm PDMS Supelco	8
5.27	(0.06) = SD	Fiber 100 µm PDMS Poly Micro Industries	20
5.34	± 0.07 SD	Fiber 30 µm PDMS Poly Micro Industries	20
5.24	± 0.04 SD	Fiber 7 µm PDMS Poly Micro Industries	20
5.44	(0.05) SD	Sheet 500 µm PDMS Altecweb	20
5.47	(0.21) SD	Fiber 7µm PDMS Supelco	21
5.18	(0.11) SD	Fiber 100 µm PDMS Supelco	21
4.59	--	PDMS trap Restek	22
4.67	--	PDMS trap Restek	22
5.36	± 0.05 SE	Fiber 28.5 µm PDMS Poly Micro	23

			Industries	
	5.42	± 0.04 95% CI	Sheet 0.4 mm silicon rubber Silastic A Dow Corning	15
	5.23	± 0.07 95% CI	Sheet 0.5 mm silicon rubber SR Batch 0 Vizo Zeewolde	15
	5.5	± 0.06 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	15
	5.23	± 0.06 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	15
	5.54	± 0.06 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
	5.53	± 0.04 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
	5.52	± 0.05 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
	5.17	--	Fibers 16.5 µm Prime Optical Fiber Co	24

PCB 52

log K_{p-w}		±		**ref**
	4.98	± 10-15 % RSD	Fiber 7 µm PDMS	8
	5.60	± 0.05 SD	Fiber 100 µm PDMS Poly Micro Industries	20
	5.65	± 0.07 SD	Fiber 30 µm PDMS Poly Micro Industries	20
	5.58	± 0.05 SD	Fiber 7 µm PDMS Poly Micro Industries	20
	5.74	± 0.05 SD	Sheet 500 µm PDMS Altecweb	20
	5.11	--	PDMS trap Restek	22
	5.45	--	PDMS trap Restek	22
	5.37	± 0.05 SE	10 µm-thick PDMS coating (Fiberguide industry)	25
	5.38	--	Fiber 15 µm PDMS Fiberguide	26
	5.66	± 0.19 SD	Fiber 7 µm PDMS Supelco	19
	5.71	± 0.03 SD	Fiber 30 µm PDMS Supelco	19
	5.59	± 0.02 SE	Fiber 28.5 µm PDMS Poly Micro Industries	23
	5.72	± 0.05 95% CI	Sheet 0.4 mm silicon rubber Silastic A Dow Corning	15
	5.54	± 0.08 95% CI	Sheet 0.5 mm silicon rubber SR Batch 0 Vizo, Zeewolde,	15
	5.77	± 0.07 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	15
	5.54	± 0.06 95% CI	Sheet 0.5 mm silicon rubber SR-Red J-flex Industrial rubber products	15

5.82	± 0.07 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
5.81	± 0.06 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
5.79	± 0.07 95% CI	Sheet 0.5 mm silicon rubber AlteSil Altecweb	15
5.48	--	Fibers 16.5 µm Prime Optical Fiber Co.	24

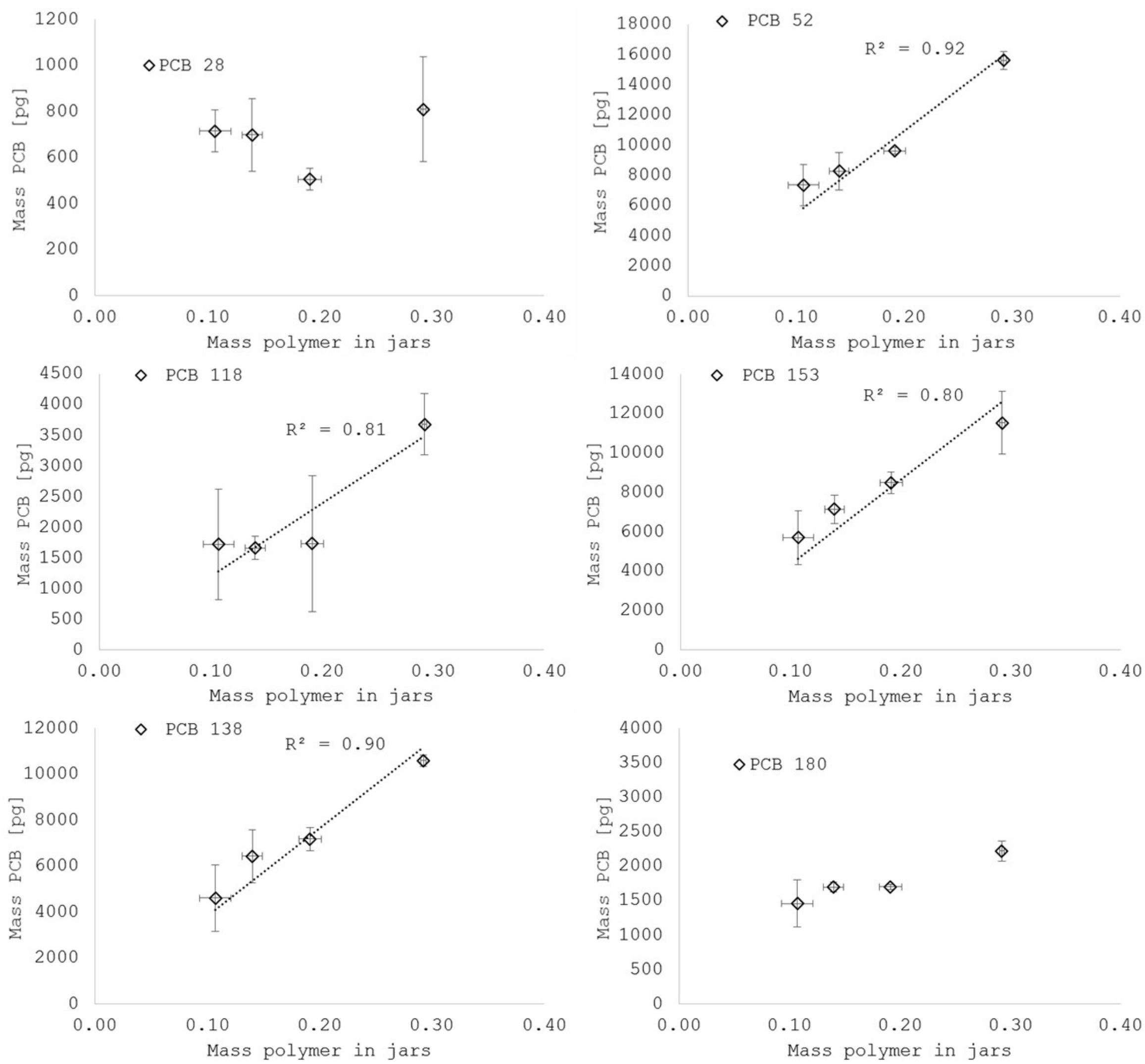

Figure ESI 1. Linear regression of the mass of PCBs quantified in extracts versus the mass of polymer in the coated jar, determined after 3 weeks of equilibration time. Trend lines are forced through origin. Error bars represent the standard deviations (n = 3). R^2 values were not included in the figure if they were negative.

Text ESI1. Chemicals and materials

Acetone (Merck, SE), dimethylformamide (VWR chemicals, SE), ethyl acetate (VWR chemicals, SE), methanol (Merck, SE), *n*-hexane (Merck, SE) and *n*-pentane (VWR chemicals, SE) were of HPLC grade. Silica (SiO_2) gel 60 (Merck, SE) and sodium azide were purchased from VWR chemicals, SE. Ethanol (99.7 %) was purchased from Solveco, SE. Stable isotope-labeled ($^{13}C_{12}$; IUPAC # 28, 53, 52, 101, 118, 138, 153 and 180) PCBs (Wellington Laboratories, Guelph, Canada) were purchased from Greyhound Chromatography and Allied Chemicals (Birkenhead, UK); PCB 53 and native PAHs (naphthalene, acenaphthene, acenaphthylene, fluorene, anthracene, phenanthrene, fluoranthene, pyrene, benz[a]anthracene, chrysene, benzo[b]fluoranthene, benzo[k]fluoranthene, benzo[a]pyrene, indeno[1,2,3-cd]pyrene, dibenz[a,h]anthracene, benzo[g,h,i]perylene) were purchased from Accustandard (New Haven, CT, USA). Deuterated (d_8-d_{12}) PAHs (same congeners as the native PAHs, except acenapthylene) and native PCBs (IUPAC # 28, 53, 52, 101, 118, 138, 153 and 180) were purchased from Larodan Fine Chemicals AB (Limhamn, SE). Triclosan (Irgasan DP300) was a gift from Ciba-Geigy (Novartis) and $^{13}C_6$-triclosan purchased from ALSACHIM. γ-HCH was purchased from Dr. Ehrenstorfer (Augsburg, DE). 4-monochlorophenol, 2,6-dichlorophenolic and 3,5-dichlorophenol were found stored at the Department of Environmental Science and Analytical Chemistry (ACES, Stockholm University). Their concentrations were checked before use. Dow Corning® 1-2577 conformal coating was used for the passive sampling jars. The 180 mL amber glass jars were purchased from Apodan Nordic Pharma Packing A/S, DK and lids from Nordic Pack, SE. The biomedical grade elastomer Dow Corning® Silastic ®

MDX4-4210 was cast in passive dosing vials used for testing of the loading efficiency, determination of the PDMS-water partition coefficients (K_{pdms-w}), and in the cell viability test. The 1.5 mL vials used in the cell viability test were purchased from Technolab, SE. TO-PRO-1 iodide was purchased from Thermo Fisher Scientific, SE. 1.5 mL Eppendorf tubes were from Sarstedt, DE.

Text ESI2. Study site and sediment sampling

Sediment (0-20 cm) for the transfer of an environmental mixture of chemicals in the bioassay was collected in the Ålöfjärden Bay, located ca 100 km south of Stockholm (the site is described in more detail in[27]), using a van Veen grab sampler. Concentrations of the legacy contaminants $\sum PAH_{15}$ and $\sum PCB_7$ in the sediment, analyzed as part of a separate study (Mustajärvi et al.) were 16 µg g^{-1} DW and 50 ng g^{-1} DW, respectively[27]. Pore water concentrations, determined with 17 µm thin polyoxymethylene passive samplers (POM-17), were 280 ng L^{-1} for $\sum PAH_{15}$ and 0.22 ng L^{-1} for $\sum PCB_7$ [27]. The water content in the sediment, determined in a pre-study, was 70 %. Sediment (0-20 cm) for method development was retrieved from a central area in Stockholm City, where high levels of $\sum PAH_{20}$ of ca 45 µg g^{-1} DW[28] and $\sum PCB_7$ of ca. 300 ng g^{-1} DW[29] had been measured in the sediment. The latter sediment was used in an initial test to validate the equilibrium partitioning between the sediment and the sampling polymer of different thicknesses.

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