Polybromodiphenyl Ethers and Decabromodiphenyl Ethane in Aquatic Sediments from Southern and Eastern Arkansas, United States

Hua Wei,[†] Azivy Che Aziz-Schwanbeck,[‡] Yonghong Zou,[§] Margaret B. Corcoran,[‖]
Armen Poghosyan,[‖] An Li,[†,*] Karl J. Rockne,[‡] Erik R. Christensen,[§] and Neil C. Sturchio[‖]

† School of Public Health, University of Illinois-Chicago
‡ Department of Civil and Materials Engineering, University of Illinois-Chicago
§ Department of Civil Engineering and Mechanics, University of Wisconsin-Milwaukee
‖ Department of Earth and Environmental Sciences, University of Illinois-Chicago

* Corresponding author's email: anli@uic.edu

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A1. Description of sampling locations

Sampling Location Description	Sampling Spot
Lake Calion (ACL) is an impoundment located only miles northeast of the town of El Dorado. It is a lowland lake with high chloride concentration in water. It has been used for water supply, angling (public fishing), and recreation.	
Lake Jack Lee (AJL), also called Lake Felsenthal, is part of the Ouachita River system which covers much of southern Arkansas. It is a reservoir backed by the Felsenthal National Wildlife Refuge wetlands. It is about 40 miles east of El Dorado.	
West Lake of El Dorado (AED) was constructed between 1931 and 1960 although the exact date is unknown. A spring-fed muddy upper pond to the southwest feeds into West Lake. West Lake has completely emptied and dried twice since 1970 (Woods, 2011). AED is within miles from Chemtura manufacturing facilities.	
AMW is a man-made L-shaped 30 acre pond. It was built in 1952, and had received treated effluent from the Magnolia Wastewater Treatment System facility until 1989. It is within a few miles from Albemarle facility although has not received the wastewater directly from that plant (Russell, 2009).	

Lake Old Town (<u>AOT</u>) is one of many natural oxbow lakes near the Mississippi River in central east Arkansas. It has very high nutrient, chlorophyll a, and suspended solids contents in its water. It is used for recreation (USGS, 2006).	
Lake Frierson (<u>AFR</u>), located in the northeast Arkansas, is an upland lake. It has low chlorophyll-a concentrations but unusually high clay turbidity thus very low light penetration (USGS, 2006). It is used for fishing and other recreations (USGS, 2006). AFR is close to the PBDE "source center" identified by Zhu and Hites (2006).	

A2. Results of on-site water quality measurements

ID	SA [a] (km^2)	CA [a] (km^2)	Water depth (m) [b]	Temp. (°C) [c]	DO [c] (mg/L)	DO [c] (%)	Cond. [d] (µS/cm)	pH	Core length (cm)	Number of sections
AED	0.11	1.33	3.7	28	5.21	66.4	112	7.2	25	20
AMW	0.09	NA [e]	1.5	27.7	10.02	127.2	474	7.2	58	29
ACL	2.06	17	2	28.7	4.25	53.2	98.2	7.3	45	24
AJL	56.66	28107	2	27.3	4.97	56.4	74.7	7.15	35	22
AOT	3.64	60	1.6	26.8	8.42	104.8	112	NA [e]	75	33
AFR	1.36	19	2	24.9	5.88	72	NA [e]	NA [e]	30	21

a. SA = water surface area, CA = catchment area. From USGS (2006)
b. Water depth was measured onsite with a marked anchor rope.
c. Temp = temperature; DO = dissolved oxygen. Temp, DO and pH were analyzed with an YSI-550A handheld meter.
d. Cond. = conductivity. It was analyzed using an Orion Model 1230 in the laboratory of Arkansas Department of Environmental Quality (ADEQ).
e. NA = not available or not measured.

B1. Physical properties of sediment by site

	Wet Density (g/cm³)	Dry Density (g/cm³)	Particle Density (g/cm³)	Organic Carbon (mg/g)	Black Carbon (mg/g)	Organic Matter (mg/g)	Water (%)	Solid (%)	Porosity (%)
ACL									(N = 24)
Ave	1.27	0.40	2.53	39.88	2.70	69.43	64.23	35.77	84.40
Median	1.19	0.38	2.53	40.95	2.54	68.89	68.03	31.97	84.87
Min	1.09	0.17	2.42	21.52	1.57	25.51	33.29	15.70	72.04
Max	1.86	0.70	2.60	66.63	4.00	130.95	84.30	66.71	93.10
AMW									(N = 28)
Ave	1.05	0.10	2.12	211.85	2.94	355.38	93.20	6.80	95.67
Median	1.03	0.06	2.10	204.92	2.85	371.16	96.51	3.49	97.16
Min	0.96	0.02	1.98	8.11	2.00	17.93	41.61	1.49	62.50
Max	1.68	0.98	2.62	311.53	4.67	473.58	98.51	58.39	99.22
AED									(N = 20)
Ave	1.20	0.42	2.55	33.15	2.31	53.92	53.01	46.99	83.69
Median	1.14	0.25	2.53	43.42	2.06	69.08	77.50	22.50	90.23
Min	1.03	0.13	2.49	8.38	1.03	14.49	1.23	6.16	67.15
Max	1.59	0.86	2.62	51.83	4.40	88.04	93.84	98.77	94.95
AJL									(N = 22)
Ave	1.24	0.65	2.52	39.89	1.66	74.83	36.41	63.59	74.45
Median	1.16	0.68	2.56	25.15	1.48	46.81	28.72	71.28	73.42
Min	0.98	0.15	2.38	15.35	1.15	27.22	7.45	13.76	58.78
Max	1.58	1.06	2.60	93.25	2.63	157.82	86.24	92.55	93.96
AOT									(N = 30)
Ave	1.08	0.20	2.56	31.45	5.07	52.19	67.29	32.71	92.28
Median	1.08	0.18	2.56	28.64	4.42	47.06	83.49	16.51	92.99
Min	0.47	0.06	2.51	18.40	0.98	24.71	5.84	5.46	86.52
Max	1.37	0.35	2.60	51.56	10.67	79.66	94.54	94.16	97.78
AFR									(N = 21)
Ave	0.70	0.52	2.60	15.61	1.50	25.60	15.13	84.87	80.20
Median	0.48	0.37	2.60	15.38	1.52	26.09	11.88	88.12	85.67
Min	0.29	0.27	2.59	12.64	1.00	20.88	3.65	57.35	51.25
Max	1.72	1.27	2.61	18.84	1.89	31.34	42.65	96.35	89.69
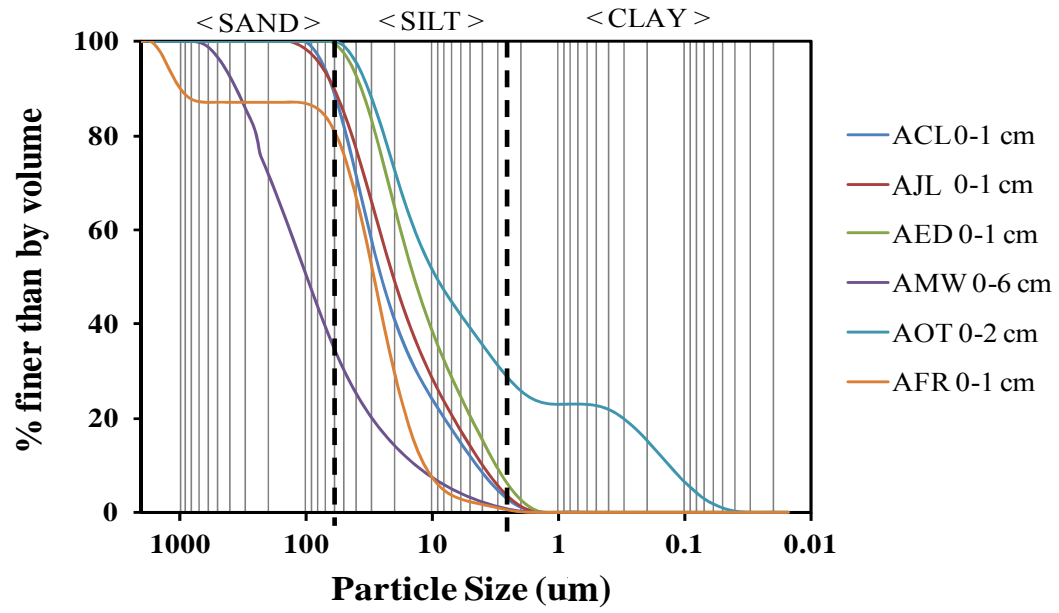
B2. Particle size distribution of surface sediments

C1. Methods of radionuclide activity measurement and sedimentation rate estimation

Dry sediment samples (0.70 to 8.07 g) were weighed into thin aluminum counting cans and sealed. Gamma activities were measured for ^{210}Pb at 46.5 keV, ^{226}Ra at 186.2 keV, and ^{137}Cs at 661.6 keV, with a Canberra model GR3020 reverse-electrode intrinsic Ge detector system (34% efficiency) interfaced with a DSA-2000 digital spectrum analyzer. Detector efficiency was calibrated in the same geometry using certified standards DL-1a (CANMET, U-Th ore) and SRM-4357 (NIST, ocean sediment). Uncertainties in sample activities (1σ) were calculated using counting statistics, including uncertainties in background subtraction.

The Constant Initial Concentration (CIC) Model and the Constant Rate of Supply (CRS) Model as described by Appleby (2001) were applied to the ^{210}Pb activity data, with a single slope through all sediment layers or using two slopes in the cases there could be possible changes in sedimentation rate over the core depth. Mass sedimentation rates (MSR) were calculated as slope $\times \lambda$, where $\lambda = 0.0311$ is the decay constant for ^{210}Pb. The year of sediment deposition was then estimated for each sediment layer using the MSRs with the cumulative dry mass. MSRs were also estimated based on linear extrapolation of the sediment cumulative dry mass between 2009 (the top layer) and 1963 which is at the peak of ^{137}Cs activity.

C2. Results of sediment core dating

The activities of ^{210}Pb, ^{137}Cs, and ^{226}Ra are plotted against sediment depth in Figure C-1. Activities shown were corrected for the decay between the sample collection date and the detection date. The CIC and CRS plots are shown in Figure C-2. All estimated MSRs are summarized in Table C-1. Reported in Table 1 of the main text are the MSRs selected based on the discussion below for individual sites. They were used to estimate the years used for the discussions in the main text as well as in SI D3 and SI D5.

In <u>ACL</u>, the ^{210}Pb activity reached a steady value and the activity ratio of ^{210}Pb/^{226}Ra approached 1 in the bottom three sections. Therefore, the supported ^{210}Pb was calculated by averaging the ^{210}Pb activities of the bottom three sections. The ^{210}Pb activity declines from top of core to bottom, and there are clear onset and peak of ^{137}Cs. The average MSRs (0.124 g/cm^2-y) obtained from CIC and CRS models with single slopes produced the deposition years that best fit to the known site history and better match with the years of ^{137}Cs onset and peak.

In <u>AED</u>, there was no clear indication that supported ^{210}Pb was reached. The ^{210}Pb activity increases to depth 8.5 cm then declines. Severe disturbance of the sediments had occurred in the past, as suggested by the known history of the site (SI A1) and shown in the CIC plot in Figure C-2. There are two peaks but no clear onset of ^{137}Cs. Severe mixing at this site made the use of both CIC and CRS models difficult. A careful comparison among all estimated MSRs indicated that the MSR (0.196 g/cm^2-y) based on the higher peak of ^{137}Cs produced the deposition years that best fit to the known site history as well as the PBDE production history.

In <u>AMW</u>, there was no clear indication that supported ^{210}Pb was reached. The clayey sections below 50 cm were excluded from the analysis. The lowest ^{210}Pb activity was found in the top section, which was therefore excluded from the calculations of the slope in the CIC model. The CIC plot indicates the severe disturbance of the sediments in the past. The ^{210}Pb activity declines steadily from sections 2 through the bottom. The average MSR (0.0611 g/cm^2-y) obtained from CIC and CRS models with single slopes produced the deposition years that best fit to the known site history.
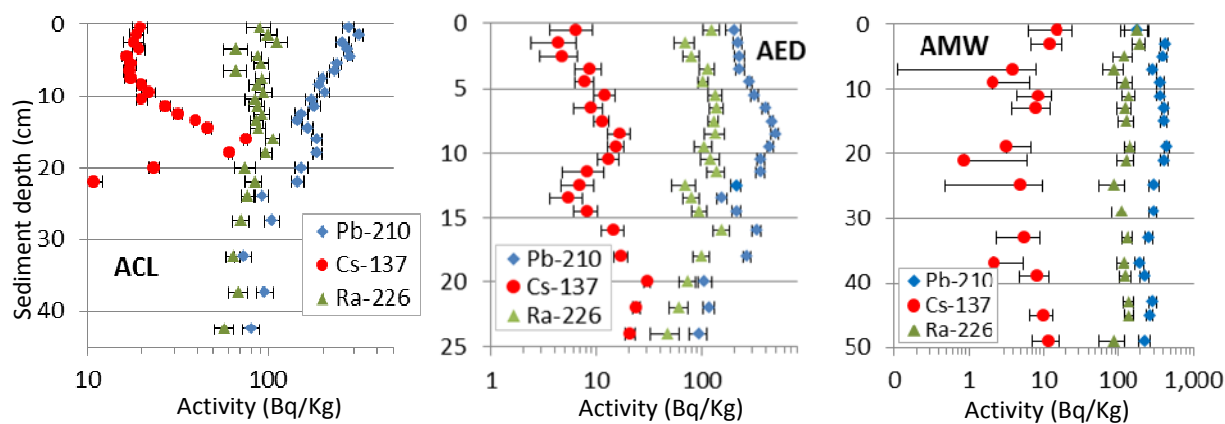
Figure C-1: Activities (Bq/Kg) for ^{210}Pb, ^{137}Cs, and ^{226}Ra for the three cores.
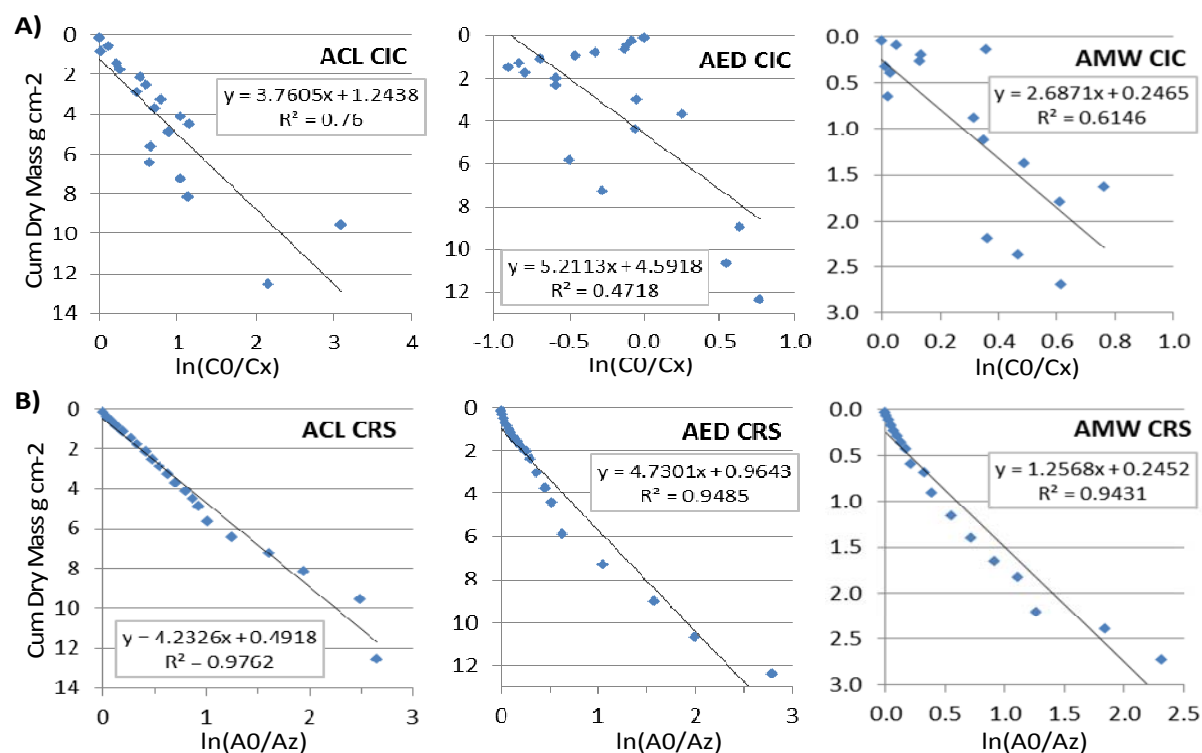
Figure C-2: (A) CIC plot – cumulative dry mass (g/cm^2) vs. ln(C$_0$/C$_X$) where C$_0$ is unsupported ^{210}Pb activity in the top section and C$_X$ is unsupported ^{210}Pb activity of section x. (B) CRS plot – cumulative dry mass (g/cm^2) vs. ln(A$_0$/A$_Z$) where A$_0$ is ^{210}Pb inventory for the entire core and A$_Z$ is the ^{210}Pb inventory up to depth z.

Table C-1. Summary of estimated mass sedimentation rates by different models (g/cm^2-y)

Site	^{137}Cs	CRS	CIC	CRS, CIC Average	CRS (2 slopes)			
					Slope-1	Depth, cm	Slope-2	Depth, cm
ACL	0.1223	0.1318	0.1171	0.1244	0.1559	0 - 15	0.1118	15 - 30
AMW	0.0593	0.0386	0.0837	0.0611	0.0589	0 - 34	0.0224	34 - 50
AED	0.1960	0.1473	0.1623	0.1548	0.2552	0 - 15	0.0956	15 - 25

D1. Method of Chemical Analysis

A standard mixture of 39 PBDEs (BDEs 1, 2, 3, 7, 8, 10, 11, 12, 13, 15, 17, 25, 28, 30, 32, 33, 35, 37, 47, 49, 66, 71, 75, 77, 85, 99, 100, 116, 118, 119, 126, 138, 153, 154, 155, 166, 181, 183, 190), DBDPE, decabromobiphneyl (BB209) was purchased from AccuStandard (New Haven, CT). $^{13}C_{12}$-labeled decachlorobiphneyl (PCB209L) and BDE209 was purchased from Cambridge Isotope Laboratories (Andover, MA). Other PBDE standards (BDEs 30, 190, 194, 195, 196, 197, 198, 201, 202, 203, 204, 205, 206, 207, 208) were provided by Accustandard. All the solvents used were Optima® or HPLC-GC/MS grade and purchased from Fisher Scientific (Pittsburgh, PA). Silica gel (100–200 mesh, Davisil grade 644) and anhydrous sodium sulfate (Na_2SO_4) were purchased from Fisher, and dried at 140°C. Bio-beads S-X3 (200 – 400 mesh) were purchased from Bio-Rad Laboratories (Richmond, CA).

The frozen samples were thawed, air-dried, and mixed with Na_2SO_4. The mixtures were spiked with the surrogates BDE30 and BDE190, and Soxhlet extracted with 150 mL hexane-DCM (1:1, v/v) for 20 h. Granular copper was added to remove elemental sulfur. The extracts were then concentrated to about 2 mL using a rotary evaporator. The extracts were then cleaned up by solvent elution through a glass columns (40cm length × 11mm i.d.) packed with 1g neutral, 2 g basic, 1 g neutral, 4 g acidic and 1 g neutral silica gel from the bottom to the top. For AMW samples, additional cleanup procedures including gel permeation chromatography (GPC) and concentrated sulfuric acid wash were used before the silica gel cleanup. The elution solvents were 80 mL hexane for silica gel cleanup and 140 mL hexane/DCM (1:1, v/v) for GPC. The volume of the eluate was reduced to 2 mL with the rotary evaporator. The internal standards of PCB209L and BB209 were added before instrumental analysis.

A total of 49 PBDE congeners and DBDPE were analyzed on an Agilent 6890 gas chromatography (GC) coupled with 5973N mass spectrometer (MS) with electron capture negative ionization (ECNI). GC/MS parameters were optimized in our previous studies.[16,17] For each run, 60 μL (20 μL × 3) was introduced to a programmable temperature vaporization (PTV) injection port in the solvent vent mode. The temperature program for the PTV inlet was from 40 °C (holding for 1.5 min) to 300 at 600 °C/min. The vent flow was 100 mL/min until 1.4 min. The purge flow was 50 mL/min from 2.75 min until 10 min. An Rtx-1614 capillary column (30 m length × 0.25 mm i.d. × 0.10 μm film thickness) was used for separation with helium carrier gas at constant flow of 1.5 mL/min. The initial oven temperature was 90 °C for 3 min, and then increased to 200 °C at 10 °C/min and further to 300 °C at 2 °C/min. The final temperature was kept for 15 min. The MS ion source, quadrupole and interface temperatures were 200, 106, and 280 °C, respectively. Methane was used as moderating gas. The MS was operated in the selected ion monitoring (SIM) mode with m/z 486.6 and 488.6 for BDE209, 79 and 81 for other PBDEs, DBDPE and BB209, and 510 and 512 for PCB209L.

D2. Concentrations and recoveries of PBDE and DBDPE in matrix spikes and SRM1944 [a]

	Sodium sulfate spiked with standards [b]					SRM 1944				
	Spike	S1	S2	R1%	R2%	A	B	Ave	Ref [c]	R%
BDE28/33	3.65	3.55	3.67	97.3	100.5	0.28	0.30	0.29	0.26	111.5
BDE47	3.64	3.65	3.81	100.3	104.7	1.46	1.42	1.44	1.63	88.3
BDE66	3.57	3.52	3.68	98.6	103.1	0.14	0.14	0.14	0.13	107.7
BDE100	3.6	3.51	3.67	97.5	101.9	0.34	0.36	0.35	0.46	76.1
BDE99	3.51	3.36	3.61	95.7	102.8	1.56	1.70	1.63	1.8	90.6
BDE85	3.61	3.42	3.69	94.7	102.2	0.04	0.06	0.05	NA	-
BDE154	3.74	3.62	3.91	96.8	104.5	0.72	0.76	0.74	1.24	59.7
BDE153	3.59	3.46	3.78	96.4	105.3	4.92	4.76	4.84	6.53	74.1
BDE183	3.15	3.02	3.32	95.9	105.4	23.4	19.8	21.6	32.2	67.1
BDE209	46.1	46.5	49.2	100.9	106.8	190.9	196.9	193.9	128	151.5
DBDPE	60.7	55.2	62.6	90.9	103.1	ND	ND	ND	NA	-

a. All concentrations are in ng/g dw.

b. Spike: spiked standard concentrations; S1 and S2: Concentrations measured in duplicate using the same laboratory procedure.

c. Geometric mean. From (i) Stapleton et al., Determination of polybrominated diphenyl ethers in environmental standard reference materials. *Analytical and Bioanalytical Chemistry* **2007,** *387*, (7), 2365-2379; and (ii) Schantz et al., NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment. NISTIR 7501. 2008. http://www.nist.gov/mml/analytical/upload/07report.pdf (accessed May 12, 2012)

D3. Regression Statistics against Distance from Source (Log Y = *m* log Distance + *b*) *

|	BDE28	BDE47	BDE99	BDE153	BDE183	BDE209	$\Sigma_{1\text{-}7}$ BDEs	$\Sigma_{8\text{-}9}$ BDEs	ΣPBDEs	DBDPE
				Y = Surface Concentration, ng g^{-1} dw						
N	4	5	5	5	5	5	5	5	5	6
m	-0.40	-0.56	-0.62	-0.97	-1.85	-2.339	-1.20	-2.23	-2.31	-2.407
b	-0.75	0.30	0.40	1.21	3.09	6.574	2.66	5.06	6.56	6.327
R^2	0.98	0.78	0.79	0.84	0.97	0.998	1.00	1.00	1.00	0.914
p	0.010	0.048	0.044	0.027	0.002	0.00003	0.000	0.000	0.000	0.003
				Y = Surface Concentration, ng g^{-1} OC						
N	4	5	5	5	5	5	5	5	5	6
m	-0.22	-0.38	-0.44	-0.79	-1.67	-2.16	-1.02	-2.05	-2.13	-1.87
b	0.32	1.32	1.42	2.23	4.11	7.59	3.67	6.08	7.58	6.51
R^2	0.42	0.88	0.76	0.62	0.99	0.99	0.93	0.99	0.99	0.69
p	0.350	0.019	0.053	0.113	0.001	0.001	0.008	0.001	0.001	0.040
				Y = Peak Concentration, ng g^{-1} dw						
N	4	5	5	5	5	5	5	5	5	6
m	-0.83	-0.62	-0.94	-1.28	-1.77	-2.22	-1.41	-2.19	-2.22	-2.72
b	0.27	0.76	1.20	2.07	3.30	6.59	3.19	5.17	6.60	7.06
R^2	0.85	0.76	0.96	0.72	0.98	1.00	0.98	0.99	1.00	0.91
p	0.078	0.055	0.003	0.069	0.001	0.000	0.002	0.000	0.000	0.003
N	4	5	5	5	5	5	5	5	5	6
m	-0.90	-0.70	-0.94	-1.28	-1.75	-2.25	-1.41	-2.20	-2.24	-2.19
b	2.02	2.46	2.74	3.58	4.79	8.16	4.68	6.76	8.17	7.31
R^2	0.61	0.55	0.77	0.55	0.89	0.96	0.81	0.94	0.95	0.85
p	0.216	0.151	0.052	0.154	0.017	0.004	0.037	0.006	0.004	0.009
				Y = Inventory, ng cm^{-2}						
N	5	6	6	6	6	6	6	6	6	6
m	-1.61	-1.72	-1.96	-2.13	-3.11	-2.87	-2.31	-2.77	-2.86	-2.38
b	2.89	4.03	4.32	4.75	6.81	8.93	5.24	6.64	8.94	6.37
R^2	0.66	0.76	0.80	0.74	0.89	0.82	0.86	0.79	0.82	0.82
p	0.093	0.023	0.017	0.027	0.005	0.013	0.008	0.018	0.012	0.013

* Distance calculations: For PBDEs, the distance is weighted by the relative releases reported on U.S. EPA TRI (http://iaspub.epa.gov/triexplorer/tri_release.geography). The weighing factors, Albemarle: Chemtura, are 6:94 (air release in 2009) for the surface and peak concentrations, and 83:17 (total on site releases, 1988-2009) for the inventory. No release data are available for years before 1988. Site AMW is excluded for concentrations of PBDEs. For DBDPE, the average distances to the two facilities were used, and all sites are included.
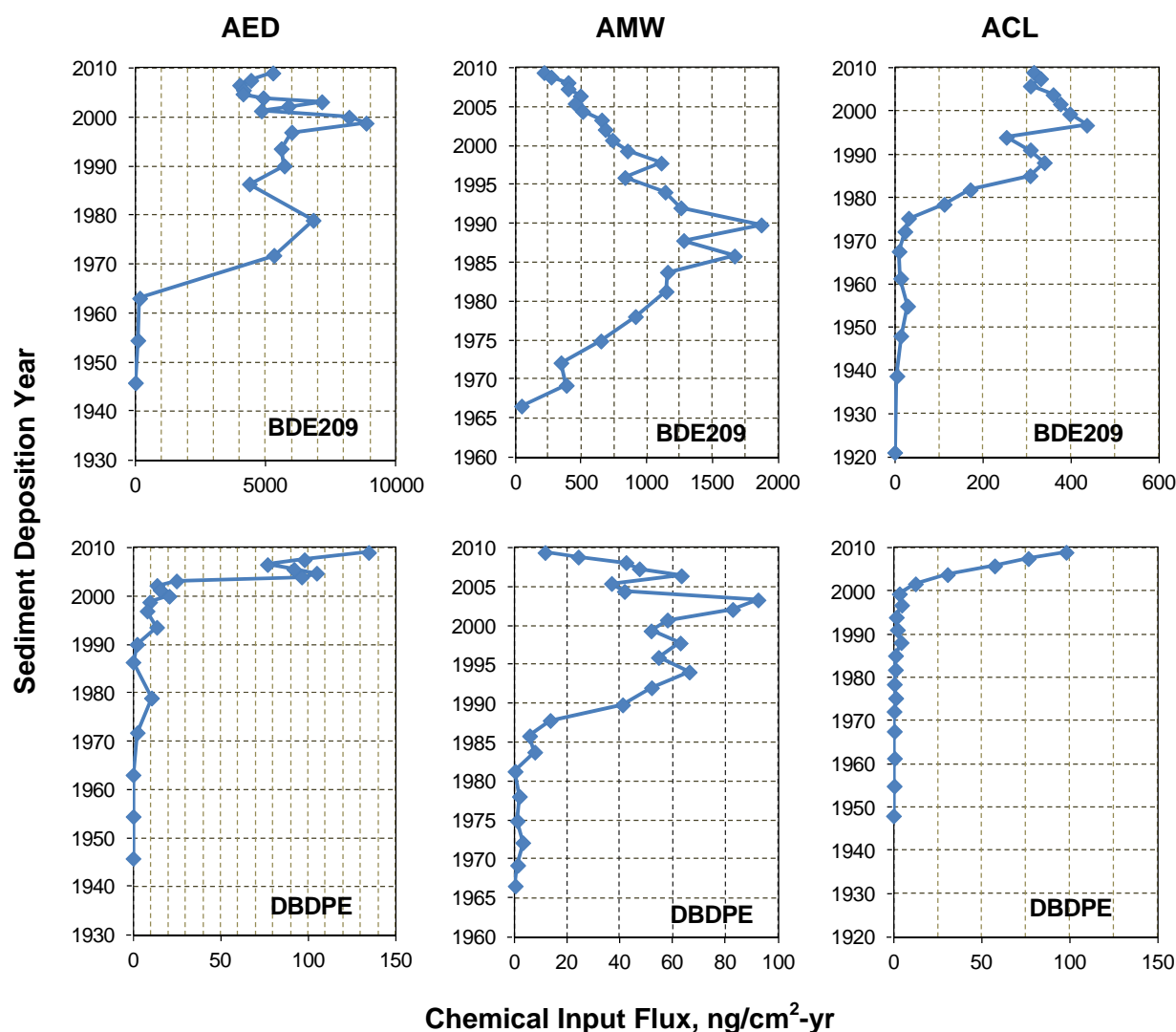
D4. Input chronology of BDE209 and DBDPE to the sediment at three locations. The input
flux of chemicals into the sediment segment i was calculated as $\text{Flux}_i = C_i \times \text{MSR} / \text{FF}$,
where C_i is the chemical concentration in ng/g dw, MSR is the mass sedimentation rate in
g/cm²-yr (Table 1), and FF is the focusing factor which is not available and was set to 1
assuming sediment movement after deposition is minimal. Due to this uncertainty, this
graph should be used with caution.
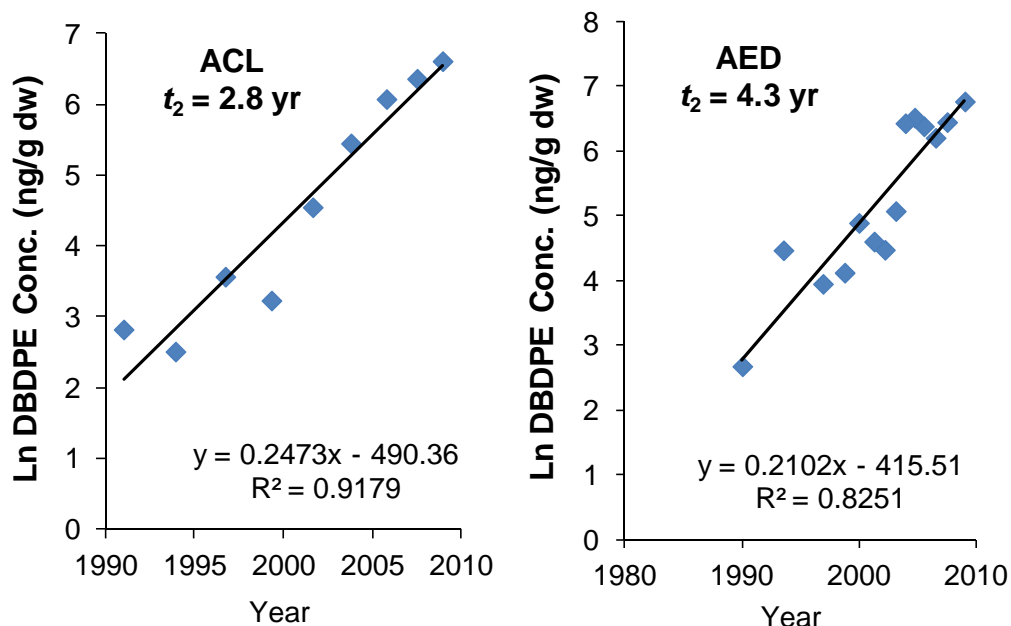
SUMMARY OUTPUT   **ACL** (time range: 1990-2009)

Regression Statistics			ANOVA					
Multiple R	0.9581		ANOVA					
R Square	0.9179			df	SS	MS	F	Sig. F
Adjusted R Square	0.9062		Regression	1	18.851	18.851	78.277	5E-05
Standard Error	0.4907		Residual	7	1.6857	0.2408		
Observations	9		Total	8	20.536			

	Coefficients	Std Error	t Stat	P-value	Lower 95%	Upper 95%	Lower 95.0%	Upper 95.0%
Intercept	-490.36	55.941	-8.7656	5E-05	-622.64	-358.08	-622.64	-358.08
X Variable 1	0.2473	0.028	8.8474	5E-05	0.1812	0.3135	0.1812	0.3135

SUMMARY OUTPUT   **AED** (time range: 1990-2009)

Regression Statistics			ANOVA					
Multiple R	0.9083		ANOVA					
R Square	0.8251			df	SS	MS	F	Sig. F
Adjusted R Square	0.8105		Regression	1	16.744	16.744	56.606	7E-06
Standard Error	0.5439		Residual	12	3.5496	0.2958		
Observations	14		Total	13	20.294			

	Coefficients	Std Error	t Stat	P-value	Lower 95%	Upper 95%	Lower 95.0%	Upper 95.0%
Intercept	-415.51	55.921	-7.4304	8E-06	-537.35	-293.67	-537.35	-293.67
X Variable 1	0.2102	0.0279	7.5237	7E-06	0.1493	0.2711	0.1493	0.2711

D5. Estimation of doubling time t_2 for DBDPE. In the Figures above, $t_2 = \ln(2)\,/\,$ slope
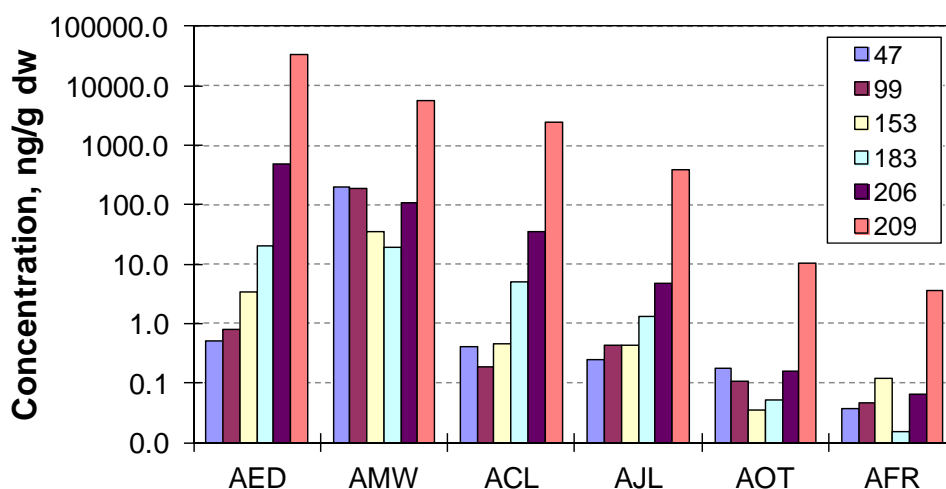
D6. Concentrations of major congeners in the surface sediments

D7. Major PBDE congener ratios

Ratio	17/28	28/47	47/99	99/153	153/183	183/196	196/206	206/209	183/209
					Tech Mix				
DE-71	0.280	0.007	0.786	8.934	54.400	–	–	–	–
Bromkal 70-5DE	0.500	0.002	0.955	8.421	16.121	–	–	–	–
DE-79	–	–	–	–	0.206	4.000	7.609	1.053	32.061
Bromkal 79-8DE	–	–	–	–	0.012	4.038	0.407	0.154	0.254
Saytex 102E	–	–	–	–	–	–	–	0.023	–
Bromkal 82-0DE	–	–	–	–	–	–	0.090	0.056	–
				Sludge (Average of 28 WWTPs in southern U.S.)					
TNSSS, 2009 *	–	0.015	0.975	10.598	5.774	–	–	–	0.0183
				AMW upper (0-30 cm)					
min	1.120	0.043	0.878	2.733	0.378	0.080	0.069	0.017	0.0016
max	3.789	0.101	1.959	5.564	1.857	0.195	0.169	0.040	0.0042
average	2.311	0.075	1.459	3.820	0.998	0.137	0.108	0.020	0.0027
median	2.176	0.075	1.446	3.686	0.957	0.146	0.101	0.019	0.0029
				AMW lower (30-49 cm)					
min	0.667	0.094	0.658	0.497	0.215	0.136	0.118	0.018	0.0029
max	1.455	0.158	0.952	2.704	0.500	1.255	0.354	0.029	0.0326
average	1.012	0.116	0.792	1.160	0.280	0.719	0.268	0.024	0.0174
median	1.042	0.113	0.788	1.071	0.249	0.700	0.282	0.023	0.0160

* Targeted National Sewage Sludge Survey Sampling and Analysis Technical Report, EPA-822-R-08-016.
  Office of Water, U.S. Environmental Protection Agency, Washington, DC, 2009.
  http://water.epa.gov/scitech/wastetech/biosolids/tnsss-overview.cfm

(a)   The total ion chromatogram (TIC) showing suspected nona-BDPEs in ACL surface (0-2 cm) sediment sample. Peaks 1 and 2 are also in AMW, AED, and AJL.
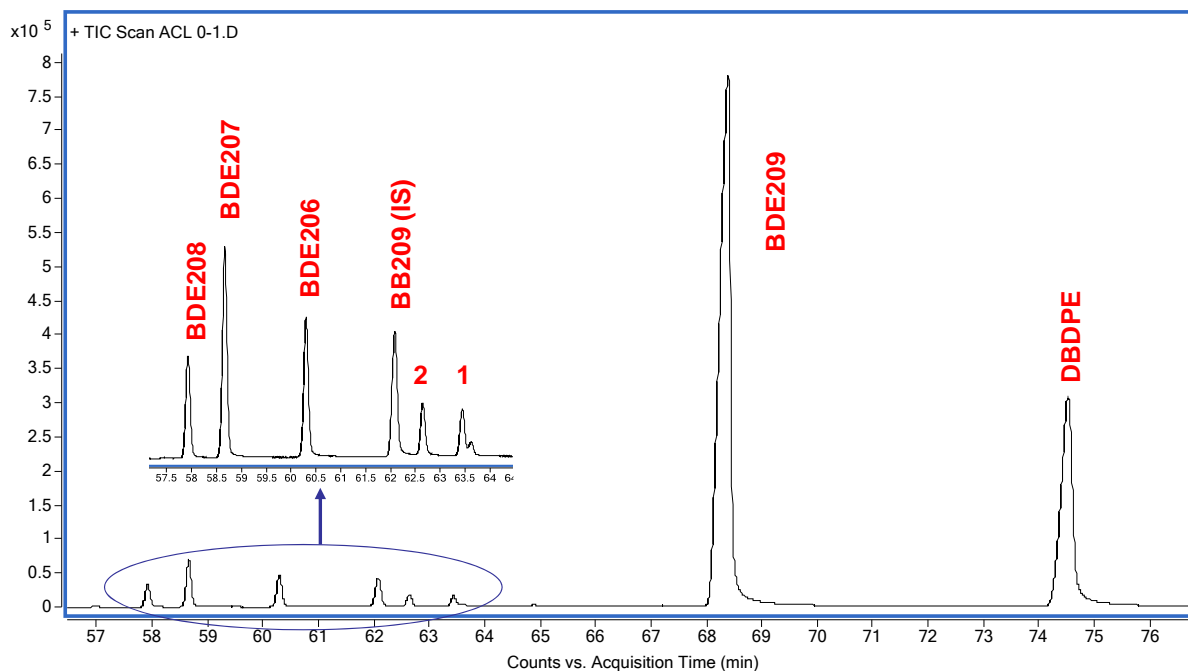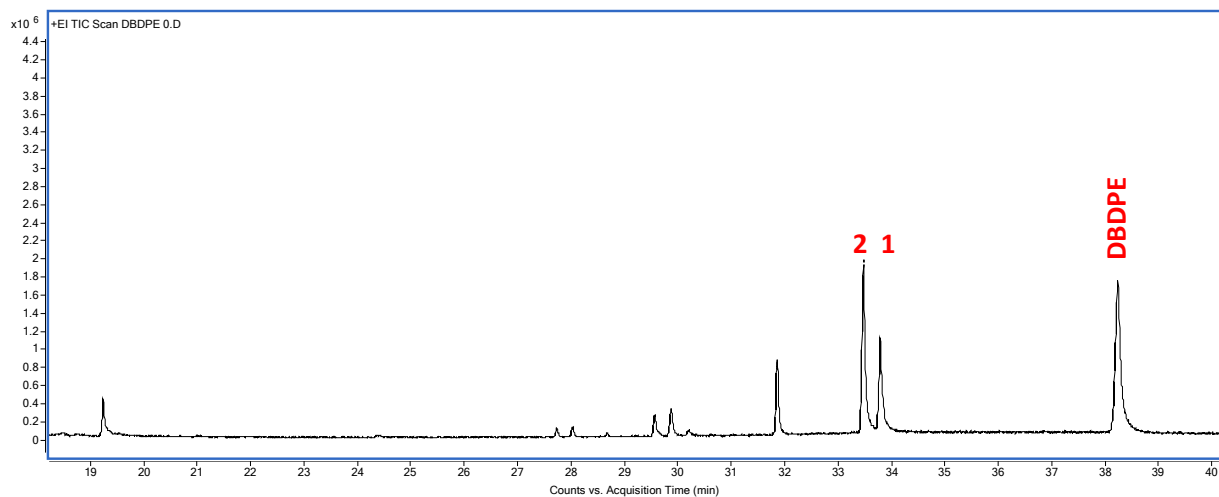
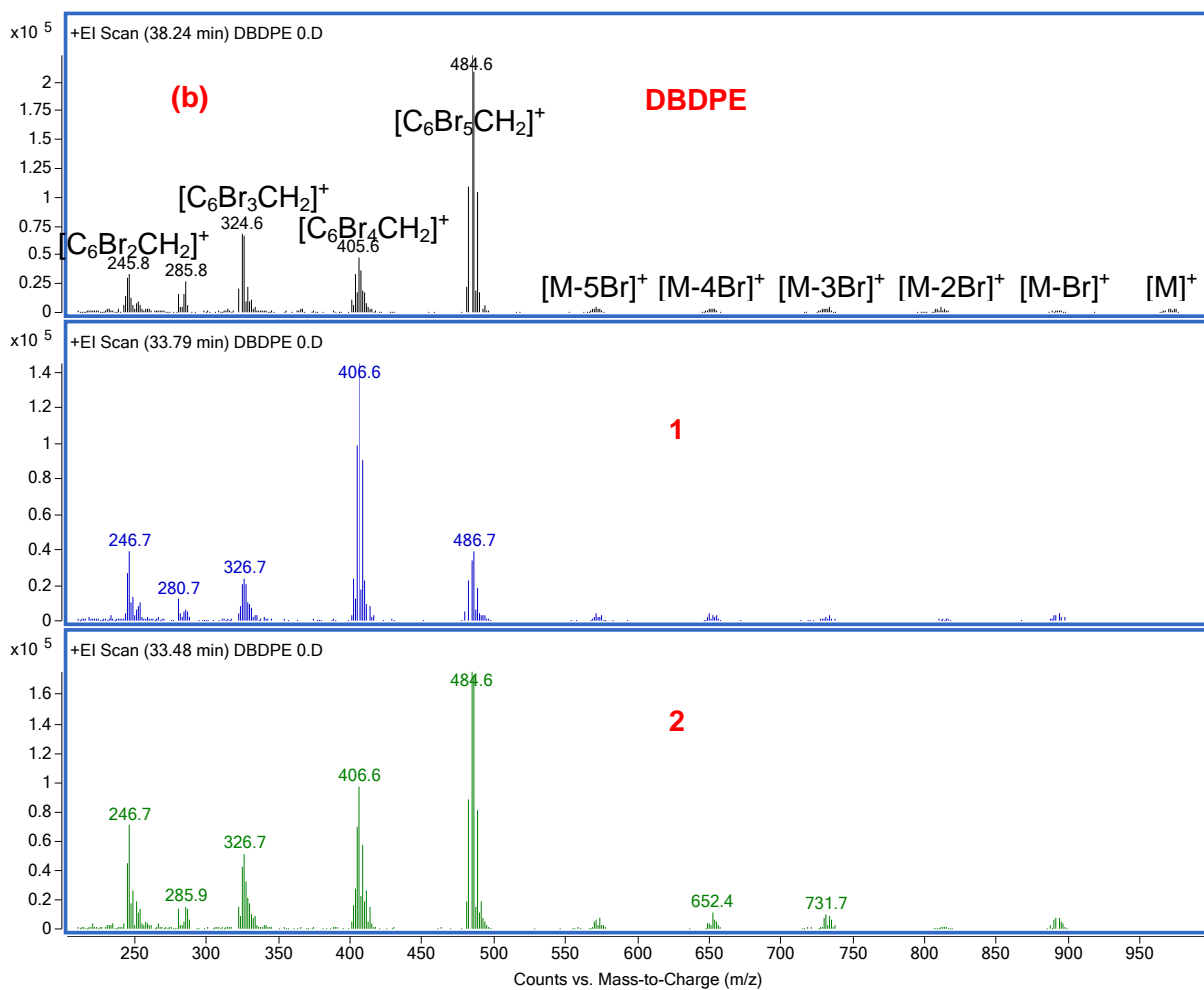(b)   Full MS SCAN of the DBDPE peak and peaks 1 and 2.
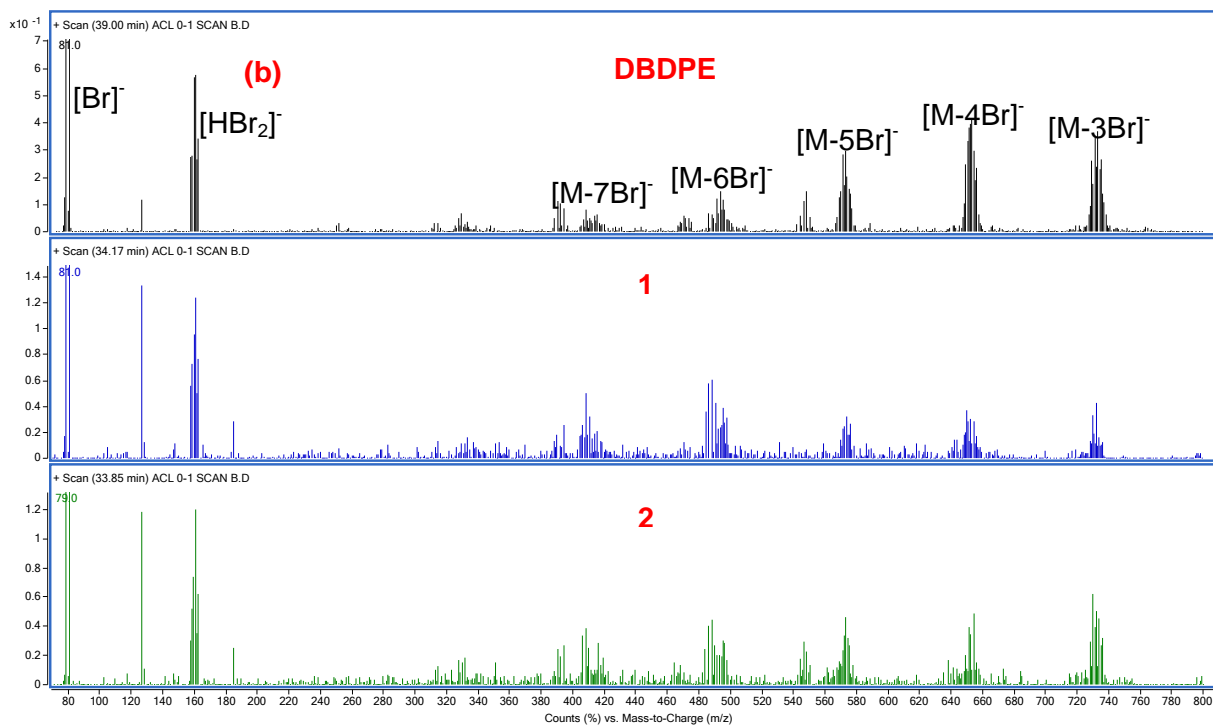
E1.   Detection of nonaBDPEs in sediment, using Agilent 6890/5973 GC/MS with electron capture negative ionization (ECNI).

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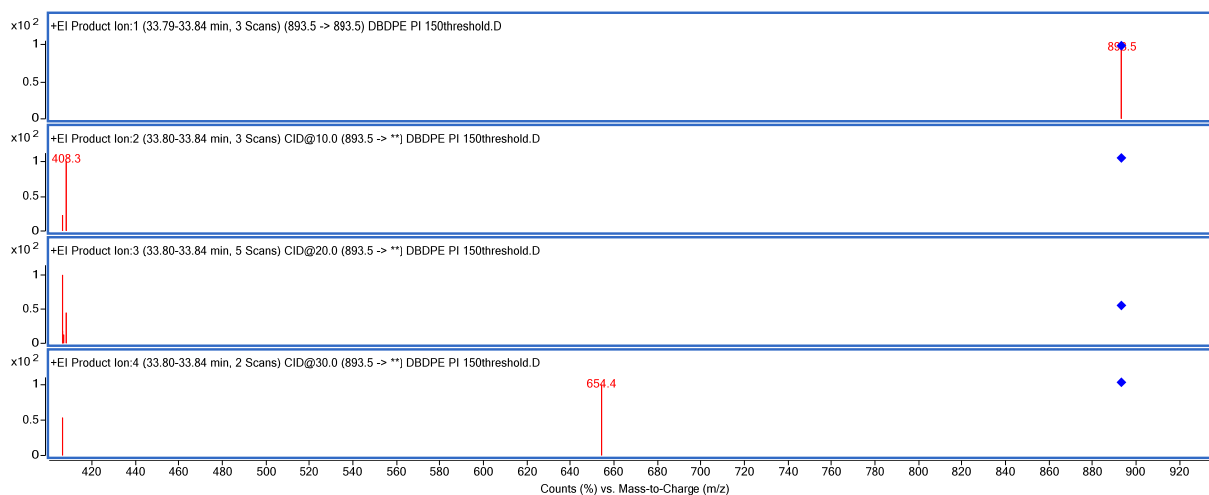(a) GC/QQQMS Analysis of a DBDPE solution after 30 min sunlight exposure

(b) Full MS1SCAN of the DBDPE peak and peaks 1 and 2 in EI.
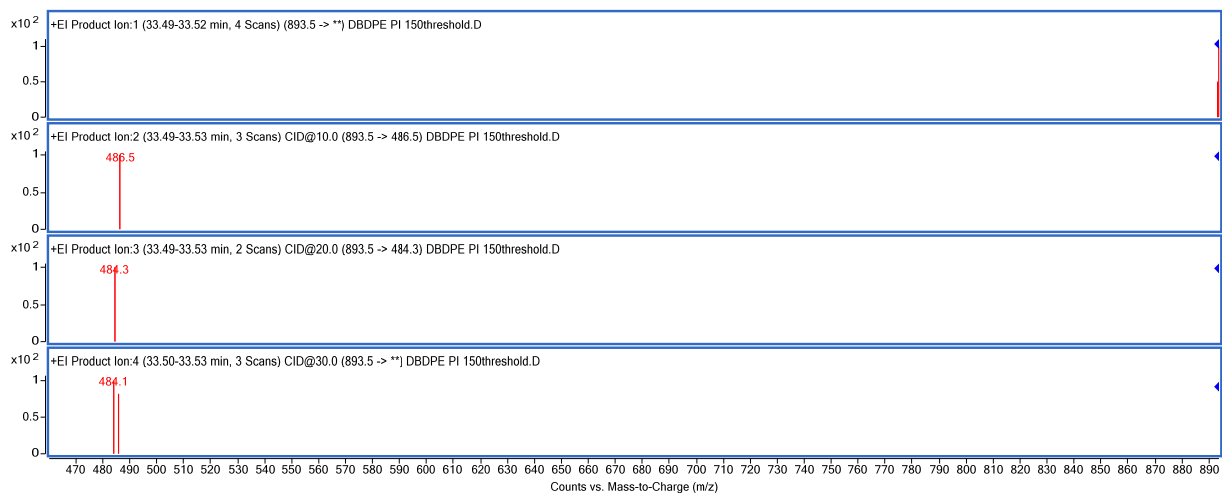
(c) Peak #1 product ion scan

(d) Peak #2 product ion scan

E2. **Analysis of DBDPE standard solution after 30 min sunlight exposure**. The analysis was conducted using Agilent 7890/7000 GC/QQQMS with MS1 scan and product ion scan in electron impact ionization (EI). In the product ion scan mode, the molecular ion (m/z = 893.5) was used as the precursor.

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