Supplementary Information (SI) for Environmental Science: Processes & Impacts.

Environmental Science: Processes & Impacts

Supplementary information

Improved Sampling Efficiency of Volatile Halomethoxybenzenes and Persistent Organic Pollutants Reveals Increasing Concentrations in Canadian Air

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Supplementary Information (SI)

SI 1. Sample extraction and analysis method

SI 1.1. Sample preparation

After sample collection, the samples were shipped to the laboratory for analysis. Before the analysis, the samples were spiked with an internal standard mixture for recovery correction which includes $^{13}C_6$-HCBD, $^{13}C_6$-PeCB, $^{13}C_6$-PeCA, $^{13}C_6$-HCB, and d_5-TBA. The average recoveries (%) ranged from 23.4% to 65.5%.

SI1.2. Sample extraction

An accelerated solvent extraction system (ASE350, Dionex) was used for the sample extraction. The extraction procedure for GLB samples began with the samples being initially extracted using hexane: acetone (6:1, v/v) over three cycles at 100°C, with a 290-second purge, a 10-minute static cycle and a rinse volume of 100%, whereas the Alert samples were extracted with hexane using the following conditions: 50°C, 5 min static cycle with a 100% flush at 800 psi. The extract was concentrated using a Turbovap system at 35°C under a gentle stream of nitrogen with isooctane as a keeper to a final volume of 1 mL. Subsequently, they were further concentrated to 0.5 mL under a stream of nitrogen and solvent-exchanged into iso-octane. The final volume of the extracts was adjusted to 0.5 ml for subsequent analysis.

SI1.3. Sample analysis

The sample extracts were analyzed using an Agilent 7010 triple quadrupole mass spectrometer (MS) with a 7890B gas chromatograph (GC) with a capillary DB-5 column (Agilent J&W Scientific, 30 m by 0.25-mm i.d. by 0.25-μm film thickness). The GC-triple quadrupole MS was operated in multiple reaction monitoring mode under electron ionization conditions.

SI 2. Apparent enthalpy calculations for surface-air exchange

To learn the possibility of emission from surface-air exchange, the Clausius-Clapeyron equation is applied as:

$$\ln P = -\frac{\Delta H_{SA}}{RT} + \text{constant}$$

Where:

P is the vapor pressure of a chemical in the gas-phase (Pa),

ΔH_{SA} represents the apparent enthalpy of surface-air exchange,

R is the gas constant, and

T is the ambient temperature (K).

To simplify this equation, we define $a = -\Delta H_{SA}/R$ as the slope, and b as the intercept, reformulating the equation as:

$$\ln P = a \times \frac{1000}{T} + b$$

By applying the measured values of ln P and T, the linear relationship can be determined, yielding the slope a and intercept b. The apparent enthalpy ΔH_{SA} is then calculated using:

$$\Delta H_{SA} = -a \times R$$

Where ΔH_{SA} represents the apparent enthalpy of surface-air exchange (kJ mol^{-1}),

R is the gas constant (8.314 J mol^{-1}).

Note that the factor of 1000 in the equation accounts for the unit conversion between ΔH_{SA} (kJ) and R (J).

Table SI 1. Instrument detection limits (IDLs, pg) and blank averages (pg sample^{-1})

	Great Lakes Basin (GLB) sites				Alert	
Chemicals	IDL		Blank average		IDL	Blank average
	2019-2021	2022	Point Petre	Evansville		2009-2020
unit	pg	pg	pg sample^{-1}		pg	pg sample^{-1}
DBA	6.82×10^{-2}	5.79×10^{-1}	68.1	223	30.8	41.3
TBA	1.77×10^{-1}	7.26×10^{-1}	558	959	76.5	347
TeCV	1.85×10^{-1}	5.54×10^{-1}	<IDL	<IDL	52.5	26.1
DAME	1.72×10^{-1}	6.63×10^{-1}	445	1880	21.7	296
PeCA	1.12×10^{-1}	3.26×10^{-1}	6230	1370	-	-
HCBD	3.06×10^{-3}	1.50×10^{-1}	924	1570	9.22	3080
α-HCH	7.54×10^{-2}	9.65×10^{-2}	4.28	13.0	27.4	54.5
γ-HCH	5.78×10^{-2}	6.77×10^{-2}	50.1	152	-	-
HCB	6.97×10^{-2}	1.23×10^{-1}	1420	282	12.8	586
PeCB	2.11×10^{-2}	1.93×10^{-1}	2060	466	5.53	244

Table SI 2. Descriptive statistics of volatile Halomethoxybenzenes (HMBs) and Persistent Organic Pollutants (POPs) (pg m^{-3}) at Alert and GLB (Point Petre and Evansville) sites

	compounds	mean	SD[*]	median	max	25%	75%	DF(%)[†]	min
Alert (2009 to January 2021) n[‡]=229									
HMBs	DBA	3.26	3.15	2.67	18.2	0.530	4.83	83.9	-
	TBA	17.5	16.6	12.2	75.6	4.87	25.8	100	0.0975
	TeCV	2.54	4.56	1.26	36.3	-	3.10	67.4	-
	DAME	30.8	31.5	21.3	256	11.7	35.6	100	1.48
POPs	HCBD	695	738	384	3600	177	971	100	16.8
	α-HCH	3.45	2.84	2.61	16.3	1.51	4.49	98.3	-
	HCB	47.3	22.9	47.9	119	29.2	64.0	100	5.03
	PeCB	14.3	8.28	13.0	42.1	8.17	18.8	100	1.38
Point Petre, GLB (October 2018 to December 2022) n=36									
HMBs	DBA	24.1	26.8	13.5	132	8.12	33.8	94.4	-
	TBA	51.2	38.1	41.5	148	19.4	71.1	100	4.77
	TeCV	5.74	8.75	2.16	39.1	0.00	8.41	58.3	-
	DAME	224	290	100	1490	48.8	289	100	16.2
	PeCA	81.9	104	49.1	382	8.67	108	80.6	-
POPs	HCBD	284	397	111	1810	64.9	391	97.2	-
	α-HCH	1.78	0.816	1.85	3.84	1.35	2.18	100	0.34
	γ-HCH	0.738	0.797	0.479	3.88	0.203	0.907	97.2	-
	HCB	45.9	35.8	41.4	154	15.5	64.8	100	7.35
	PeCB	23.9	17.4	21.6	75.5	9.49	35.8	100	0.676
Evansville, GLB (May 2019 to December 2022) n=31									
HMBs	DBA	12.1	19.5	4.14	96.8	1.08	16.6	81.0	-
	TBA	21.1	21.1	10.5	71.4	5.64	34.5	100	2.42
	TeCV	1.82	2.23	1.21	8.93	0.00	2.87	65.0	-
	DAME	56.2	48.3	53.9	185	14.9	74.2	100	4.75
	PeCA	15.1	16.5	10.6	73.3	5.35	16.9	90.3	-
POPs	HCBD	468	496	304	2150	210	476	100	74.6
	α-HCH	1.42	1.04	0.993	4.23	0.667	1.86	100	0.333
	γ-HCH	0.935	1.21	0.613	5.60	0.231	1.13	83.9	-
	HCB	17.2	17.6	7.61	64.3	6.28	19.8	100	2.63
	PeCB	16.5	12.7	9.94	46.9	6.90	23.1	100	3.83

SD: standard deviation

[†] DF(%): detection frequency, concentrations higher than the instrument detection limits and blank samples.

[‡] n= number of samples.

Table SI 3. Spearman correlation coefficients of HMBs at the GLB stations

		DBA	TBA	TeCV	DAME
Point Petre (n=36)					
TBA	Spearman Corr.	**0.507**			
	p-value	**1.62×10^{-3}**			
TeCV	Spearman Corr.	**0.334**	9.35×10^{-3}		
	p-value	**4.67×10^{-2}**	9.57×10^{-1}		
DAME	Spearman Corr.	**0.665**	**0.667**	6.44×10^{-2}	
	p-value	**1.0×10^{-4}**	**1.0×10^{-4}**	7.09×10^{-1}	
PeCA	Spearman Corr.	**0.484**	**0.476**	0.120	**0.691**
	p-value	**2.81×10^{-3}**	**3.35×10^{-3}**	0.484	**$<1.0\times10^{-4}$**
Evansville (n=31)					
TBA	Spearman Corr.	0.140			
	p-value	0.453			
TeCV	Spearman Corr.	**0.462**	**-0.522**		
	p-value	**8.89×10^{-3}**	**2.61×10^{-3}**		
DAME	Spearman Corr.	**0.545**	**0.534**	6.85×10^{-2}	
	p-value	**1.53×10^{-3}**	**1.96×10^{-3}**	0.714	
PeCA	Spearman Corr.	0.217	**0.447**	-9.10×10^{-2}	**0.645**
	p-value	0.242	**1.17×10^{-2}**	0.626	**9.07×10^{-5}**

Statistically significant correlations ($p<0.05$) are displayed in **bold**.

Table SI 4. Spearman correlation coefficients of volatile HMBs and POPs at the Alert station

2009-Jan 2021 (n=229)		T (K)	DBA	TBA	TeCV	DAME	HCBD	α-HCH	HCB
DBA	Spearman Corr.	0.103	1						
	p-value	0.123	--						
TBA	Spearman Corr.	0.0495	**0.841**	1					
	p-value	0.462	**9.53×10^{-61}**	--					
TeCV	Spearman Corr.	**-0.584**	**0.237**	**0.340**	1				
	p-value	**6.65×10^{-22}**	**3.55×10^{-4}**	**1.93×10^{-7}**	--				
DAME	Spearman Corr.	**-0.133**	**0.457**	**0.636**	**0.547**	1			
	p-value	**4.65×10^{-2}**	**7.42×10^{-13}**	**2.07×10^{-26}**	**9.82×10^{-19}**	--			
HCBD	Spearman Corr.	-0.114	**0.168**	**0.352**	**0.271**	**0.361**	1		
	p-value	9.21×10^{-2}	**1.25×10^{-2}**	**8.43×10^{-8}**	**4.62×10^{-5}**	**4.07×10^{-8}**	--		
α-HCH	Spearman Corr.	**-0.247**	**0.188**	**0.305**	**0.504**	**0.740**	0.0273	1	
	p-value	**1.88×10^{-4}**	**4.77×10^{-3}**	**3.55×10^{-6}**	**7.46×10^{-16}**	**1.01×10^{-39}**	0.687	--	
HCB	Spearman Corr.	**-0.291**	**0.233**	**0.444**	**0.498**	**0.777**	**0.452**	**0.728**	1
	p-value	**9.44×10^{-6}**	**4.43×10^{-4}**	**3.25×10^{-12}**	**2.01×10^{-15}**	**5.09×10^{-46}**	**1.79×10^{-12}**	**2.78×10^{-38}**	--
PeCB	Spearman Corr.	**-0.193**	**0.161**	**0.352**	**0.320**	**0.464**	**0.650**	**0.404**	**0.752**
	p-value	**3.79×10^{-3}**	**1.63×10^{-2}**	**6.93×10^{-8}**	**1.08×10^{-6}**	**3.46×10^{-13}**	**9.31×10^{-28}**	**3.62×10^{-10}**	**7.40×10^{-42}**

Statistically significant correlations ($p<0.05$) are displayed in **bold**.

Table SI 5. Apparent first order half-life ($t_{1/2}$) or doubling time (t_2) of volatile HMBs and POPs at the Alert station

species	slope	p	$t_{1/2}$ (or t_2)[*]	trend
DBA	0.0959	6.68×10^{-4}	7.23	increasing
TBA	0.124	5.78×10^{-6}	5.58	increasing
DAME	0.0648	4.04×10^{-4}	10.7	increasing
HCBD	0.267	1.21×10^{-43}	2.60	increasing
α-HCH	-0.0205	2.54×10^{-1}	-33.8	Not statistically significant
α-HCH (2009-2015)	-0.350	1.78×10^{-15}	-1.98	decreasing
α-HCH (2016-2020)	0.167	1.02×10^{-3}	4.14	increasing
HCB	0.0427	2.11×10^{-3}	16.3	increasing
HCB (2009-2015)	-0.119	3.49×10^{-4}	-5.81	decreasing
HCB (2016-2020)	0.230	5.25×10^{-10}	3.02	increasing
PeCB	0.108	5.46×10^{-14}	6.42	increasing

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[*] $t_{1/2}$ (or t_2): negative value is $t_{1/2}$ (half-life time) or positive value is for t_2 (doubling time).

Table SI 6. Clausius–Clapeyron relation of HMBs at Point Petre and Evansville and calculated enthalpy (ΔH, kJ mol^{-1})

species	a (slope)	P-value	Pearson R[*]	$\Delta_{SA}H$[†]	$\Delta_{AW}H$[‡]	$\Delta_{OA}H$[§]	$\Delta_{SA}H$[**]	$\Delta_{SA}H$[††]	$\Delta_{VAP}H$[‡‡]
Point Petre									
DBA	-7.83±1.45	6.39×10^{-6}	-0.690	65.1±12.1	47.0	-61.6	-	29.7±7.2 (Råö); 23.9±7.2 (Pallas)	51.2±3.0
TBA	-5.10±1.33	5.27×10^{-4}	-0.549	42.4±11.1	49.7	-68.4	32.9 ± 3.6 (Toronto)	39.8±6.5 (Råö); 12.3±5.3 (Pallas)	51.6±3.0
DAME	-10.0±1.14	2.68×10^{-10}	-0.834	83.2±9.44	63.4	-77.2	74.7 ± 3.0 (Toronto)	47.3±9.4 (Råö); 61.4±9.4 (Pallas)	55.0±3.0
PeCA	-5.76±2.41	0.0239	-0.419	47.9±20.0	44.4	-71.5	71.2 ± 4.5 (Toronto)	-	54.1±3.0
TeCV	-8.83±1.46	8.08×10^{-6}	-0.811	73.4±12.1	63.4	-77.2	71.7 ± 3.0 (Toronto)	43.5±9.2 (Råö); 31.7±8.2 (Pallas)	53.8±3.0
Evansville									
DBA	-7.73±1.24	2.30×10^{-6}	-0.793	64.3±10.3	47.0	-61.6	-	29.7±7.2 (Råö); 23.9±7.2 (Pallas)	51.2±3.0
TBA	-2.78±1.42	5.89×10^{-2}	-0.342	23.1±11.8	49.7	-68.4	32.9 ± 3.6 (Toronto)	39.8±6.5 (Råö); 12.3±5.3 (Pallas)	51.6±3.0
DAME	-7.40±0.79	2.72×10^{-10}	-0.867	61.5±6.56	63.4	-77.2	74.7 ± 3.0 (Toronto)	47.3±9.4 (Råö); 61.4±9.4 (Pallas)	55.0±3.0
PeCA	-4.74±1.18	4.40×10^{-4}	-0.619	39.4±9.81	44.4	-71.5	71.2 ± 4.5 (Toronto)	-	54.1±3.0
TeCV	-5.31±0.96	2.91×10^{-5}	-0.794	44.1±7.98	63.4	-77.2	71.7 ± 3.0 (Toronto)	43.5±9.2 (Råö); 31.7±8.2 (Pallas)	53.8±3.0

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[*] Person R: Pearson Correlation coefficient

[†] $\Delta_{SA}H$: apparent enthalpy of surface-air exchange, calculated in this study

[‡] $\Delta_{AW}H$: enthalpy of air-water exchange. (1. F. Zhan, C. Shunthirasingham, Y. Li, J. Oh, Y. D. Lei, A. Ben Chaaben, A. Dalpé Castilloux, Z. Lu, K. Lee, F. A. P. C. Gobas, N. Alexandrou, H. Hung and F. Wania, Sources and environmental fate of halomethoxybenzenes, *Sci. Adv.*, 2023, **9**, eadi8082, DOI: 10.1126/sciadv.adi8082.)

[§] $\Delta_{OA}H$: enthalpy of octanol-air exchange (1. Ibid.)

[**] $\Delta_{SA}H$: apparent enthalpy of surface-air exchange in Toronto (1. Ibid.)

[††] $\Delta_{SA}H$: apparent enthalpy of surface-air exchange (2. T. Bidleman, A. Andersson, E. Brorström-Lundén, S. Brugel, L. Ericson, K. Hansson and M. Tysklind, Halomethoxybenzenes in air of the Nordic region, *Environ. Sci. Ecotechnology*, 2023, **13**, 100209, DOI: 10.1016/j.ese.2022.100209.)

[‡‡] $\Delta_{VAP}H$: enthalpy of vaporization (https://www.chemspider.com/)

Table SI 7. HCBD air concentrations in recent years from literatures

Location	Concentrations	Sampling Method	Year of sampling	References
Cape Hedo, Okinawa, Japan (background site)	330 – 2900 pg m^{-3}	quarts fiber filter, PUF and active carbon fiber filter	2009-2016	Takasuga T. et al. [3]
Cape Hedo, Okinawa Japan	mean: 3000 pg m^{-3}	quarts fiber filter, PUF and active carbon fiber filter	2016-2018	UNEP [4]
Fukue Goto, Japan	mean: 3300 pg m^{-3}		2016-2018	
Jinan	360 ± 170 ng m^{-3}*	fused silica-lined stainless steel vacuum canisters for collection; GC-MS for analysis (US EPA TO-14 and TO-15 methods)	June 2017 to April 2018	Yang et al. [5]
Tai'an	380 ± 300 ng m^{-3}			
Mount Tai	330 ± 220 ng m^{-3}			
Mixed factory park, Weifang	810 ± 350 ng m^{-3}			
Rubber plant, Guangrao	630 ± 220 ng m^{-3}			
municipal solid waste landfill area	790 ± 90 ng m^{-3}			
Latin America and Caribbean region	<20-120 pg m^{-3}	Passive sampler		Rauert et al. [6]
tetrachloroethylene factory (acetylene method), Inner Mongolia Autonomous Region, China	1.46–1170 μg m^{-3}	activated charcoal tube	30 August to 4 September, 2018	Liu et al. [7]
tetrachloroethylene factory (tetrachloride transformation) method, Shandong China	1.96–5530 μg m^{-3}			
Near a chlor-alkali plant Tarragona, Spain	0.21±0.31 μg m^{-3}	Stainless steel sorbent cartridge	2013-2015	van Drooge et al. [8]
landfill/compost plant, Shanghai, China	ND-146±49.8 μg m^{-3} (Detection Limit=3.1 μg m^{-3}). Geomean=8.7 μg m^{-3}	Tedlar® bag		Cheng et al. [9]

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* Converted from ppt. 1 ppt HCBD = 10.6 ng m^{-3} HCBD (298 K, 101325 Pa)

Table SI 8. Clausius-Clapeyron relation of HCHs, HCB and PeCB at GLB sites

Chemical	a (slope)	P-value	Pearson R*	$\Delta_{SA}H$ (kJ mol^{-1})
Evansville				
α-HCH	-3.38±0.759	1.15×10^{-4}	-0.638	28.1±6.31
γ-HCH	-4.50±1.72	1.51×10^{-2}	-0.471	37.4±14.3
HCB	-3.01±1.18	1.62×10^{-2}	-0.428	25.0±9.81
PeCB	-3.73±0.879	2.03×10^{-4}	-0.619	31.1±7.31
Point Petre				
α-HCH	-3.60±0.796	7.03×10^{-5}	-0.613	30.0±6.62
γ-HCH	-6.16±1.46	1.79×10^{-4}	-0.592	51.2±12.1
HCB	-4.57±1.27	1.00×10^{-3}	-0.525	38.0±10.6
PeCB	-5.97±1.75	1.67×10^{-3}	-0.505	49.6±14.5

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* Person R: Pearson Correlation coefficient

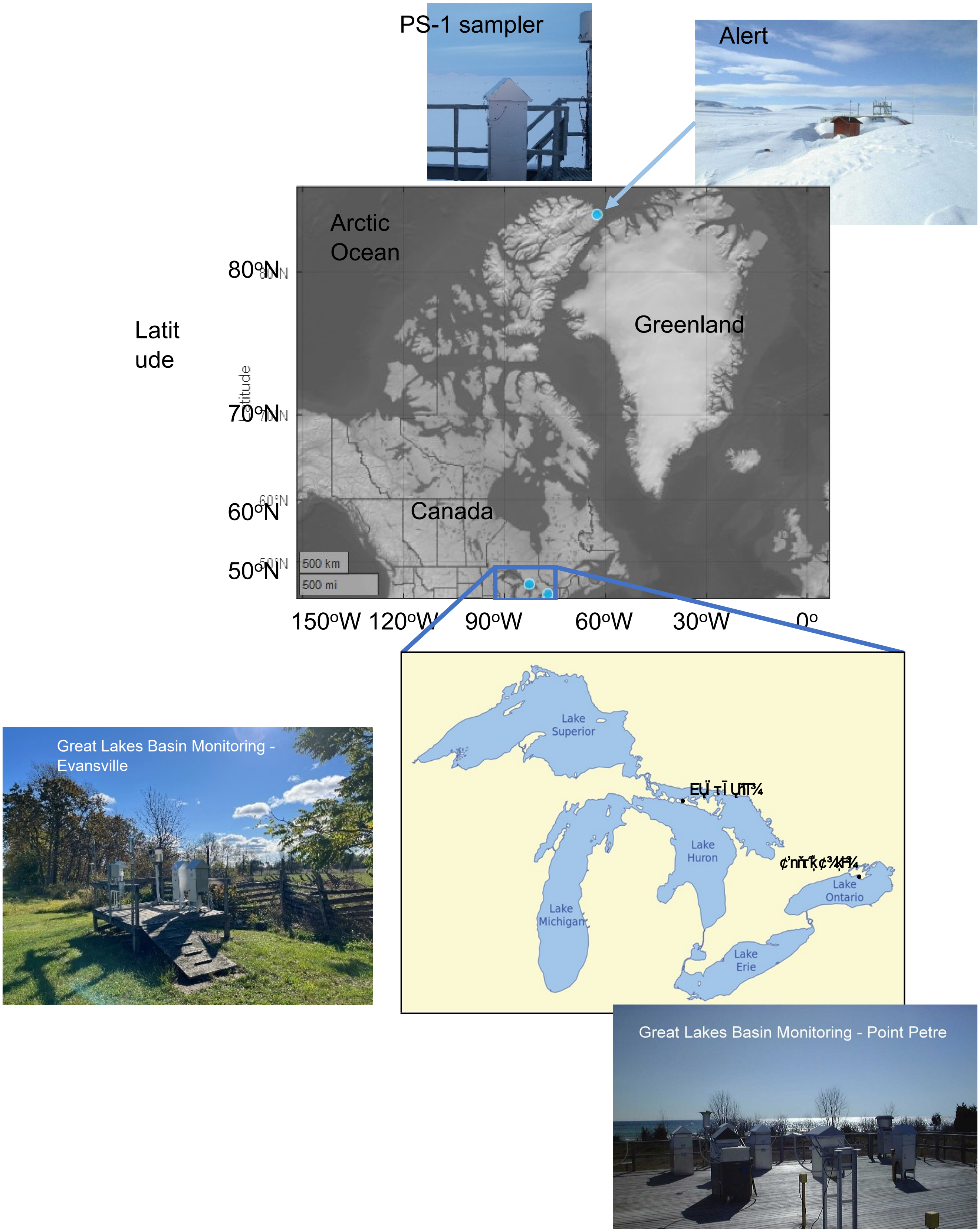

Figure SI 1. Map of sampling locations with photos of each site and the sampling devices.

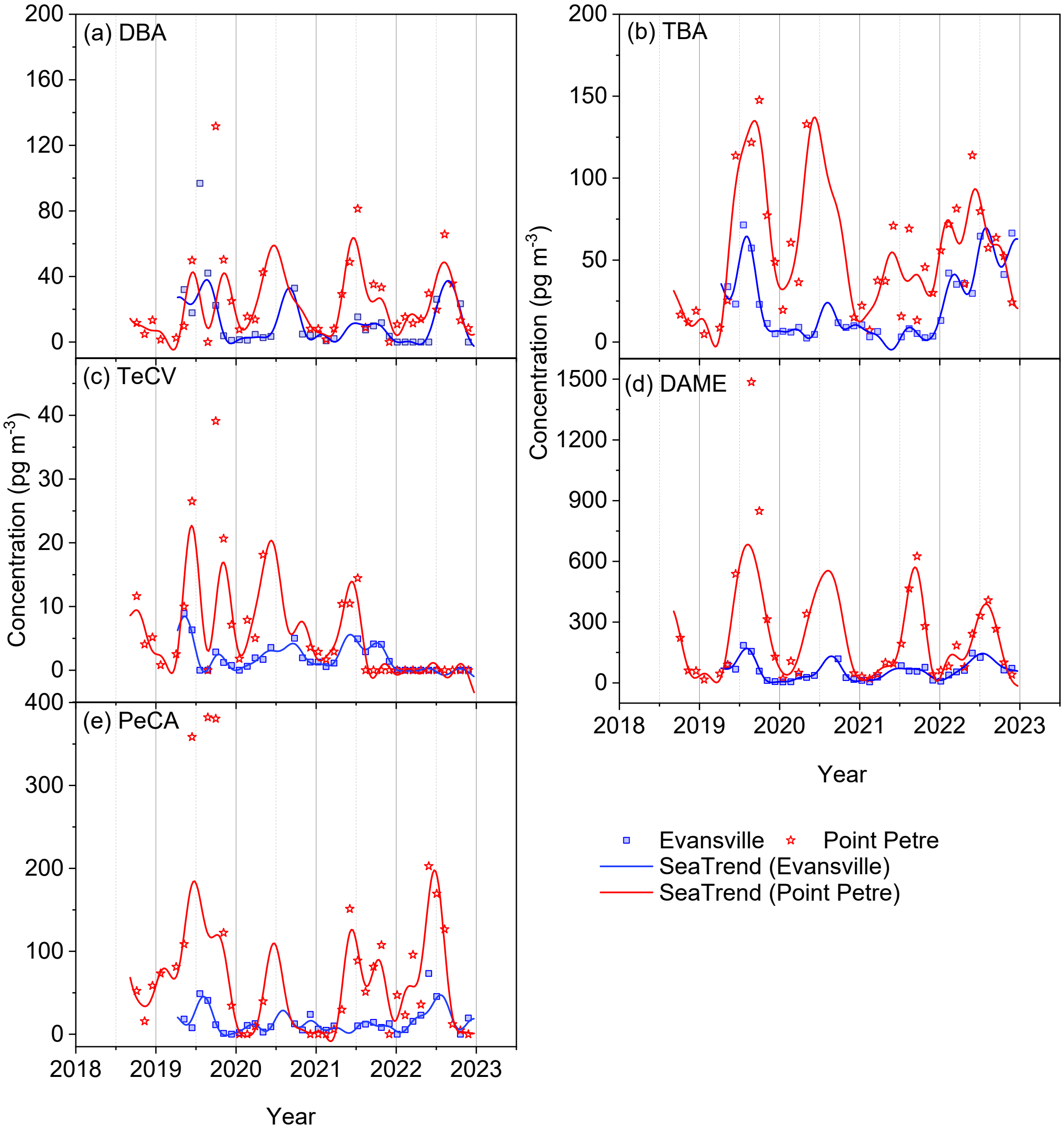

Figure SI 2. Concentrations (pg m^{-3}) and seasonal trends (SeaTrend) of (a) DBA, (b) TBA, (c) TeCV, (d) DAME and (e) PeCA from 2018 to 2022 at the Evansville and Point Petre stations.

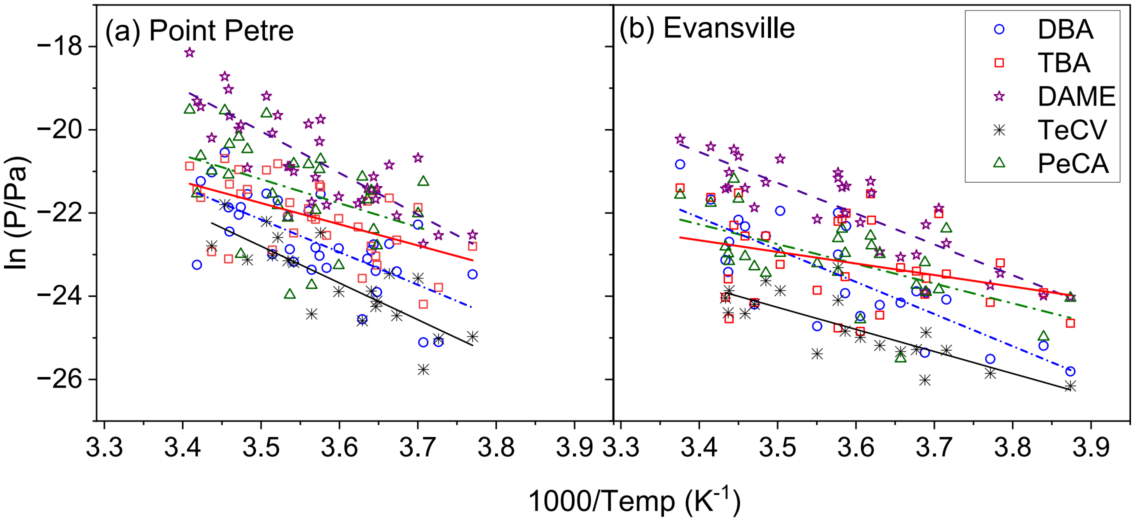

Figure SI 3. Clausius- Clapeyron relation of 5 HMBs at (a) Point Petre and (b) Evansville.

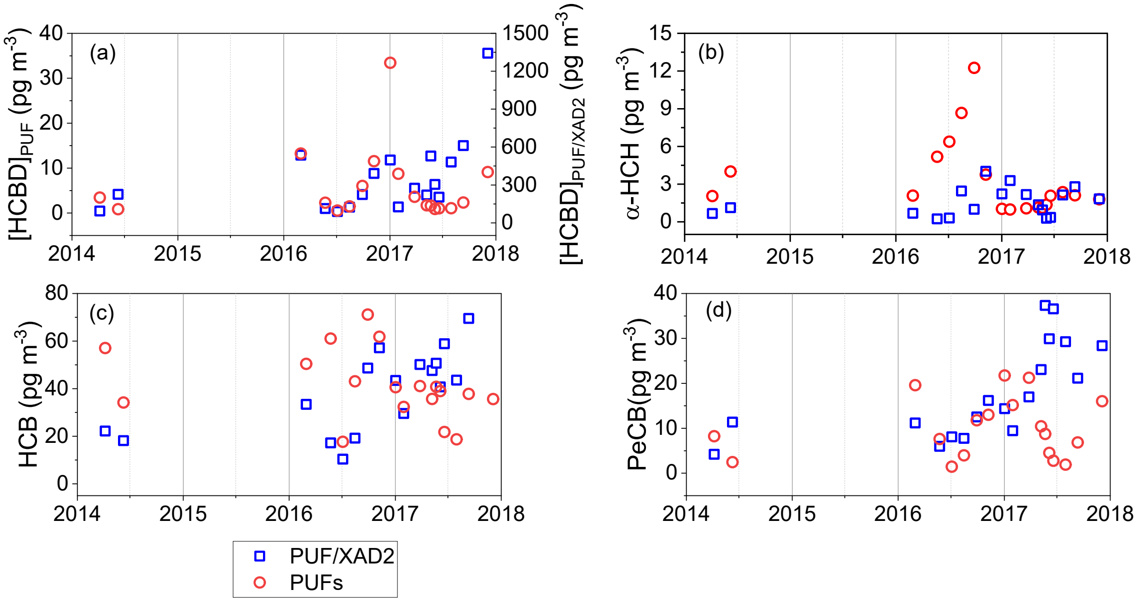

Figure SI 4. Collocated volatile POP measurement comparison with the sampling media of 2 PUFs (○) and PUF-XAD2-PUF (□) at the Alert site (2014 – 2017). (a) HCBD concentrations (pg m^{-3}) collected by 2 PUFs ([HCBD]$_{PUF}$) and PUF-XAD2-PUF([HCBD]$_{PUF/XAD2}$); (b) α-HCH; (c) HCB; (d) PeCB concentrations (pg m^{-3}).

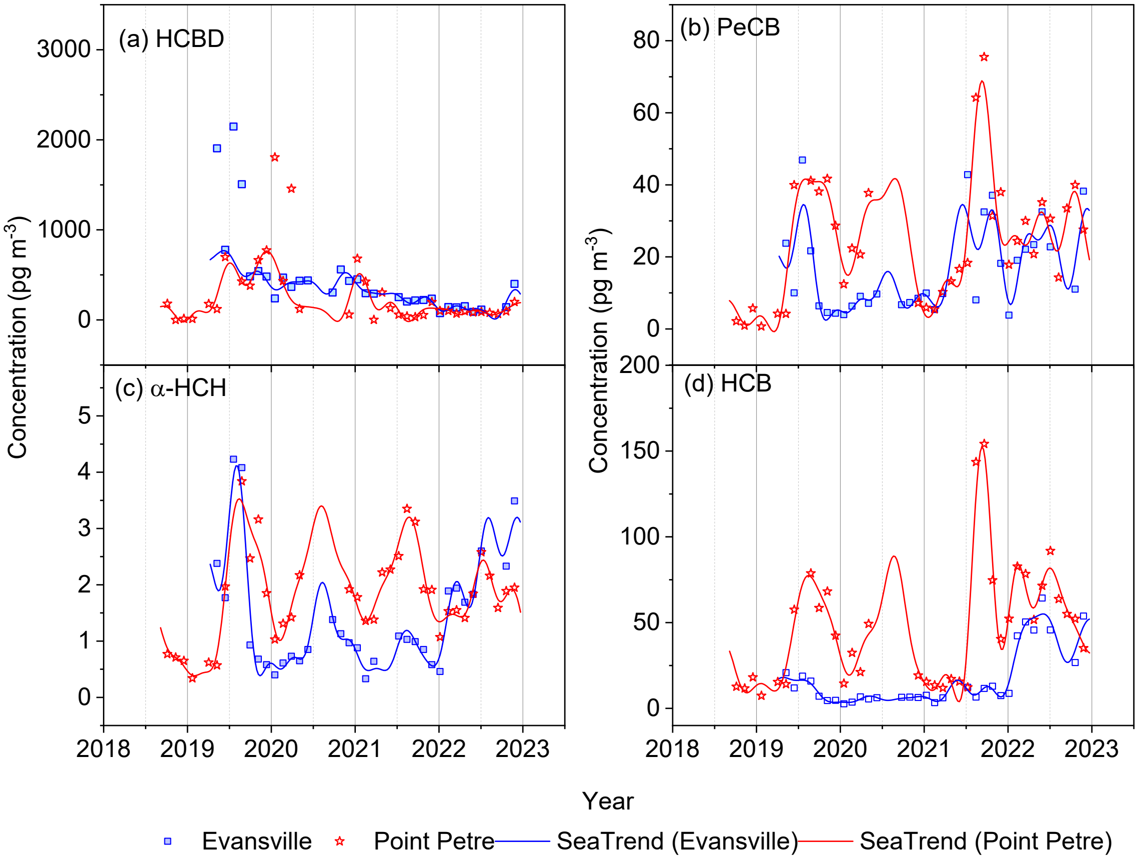

Figure SI 5. Concentrations (pg m^{-3}) and seasonal trends (SeaTrend) of (a) HCBD, (b) PeCB, (c) α-HCH, and (d) HCB from 2018 to 2022 at the Evansville and Point Petre stations.

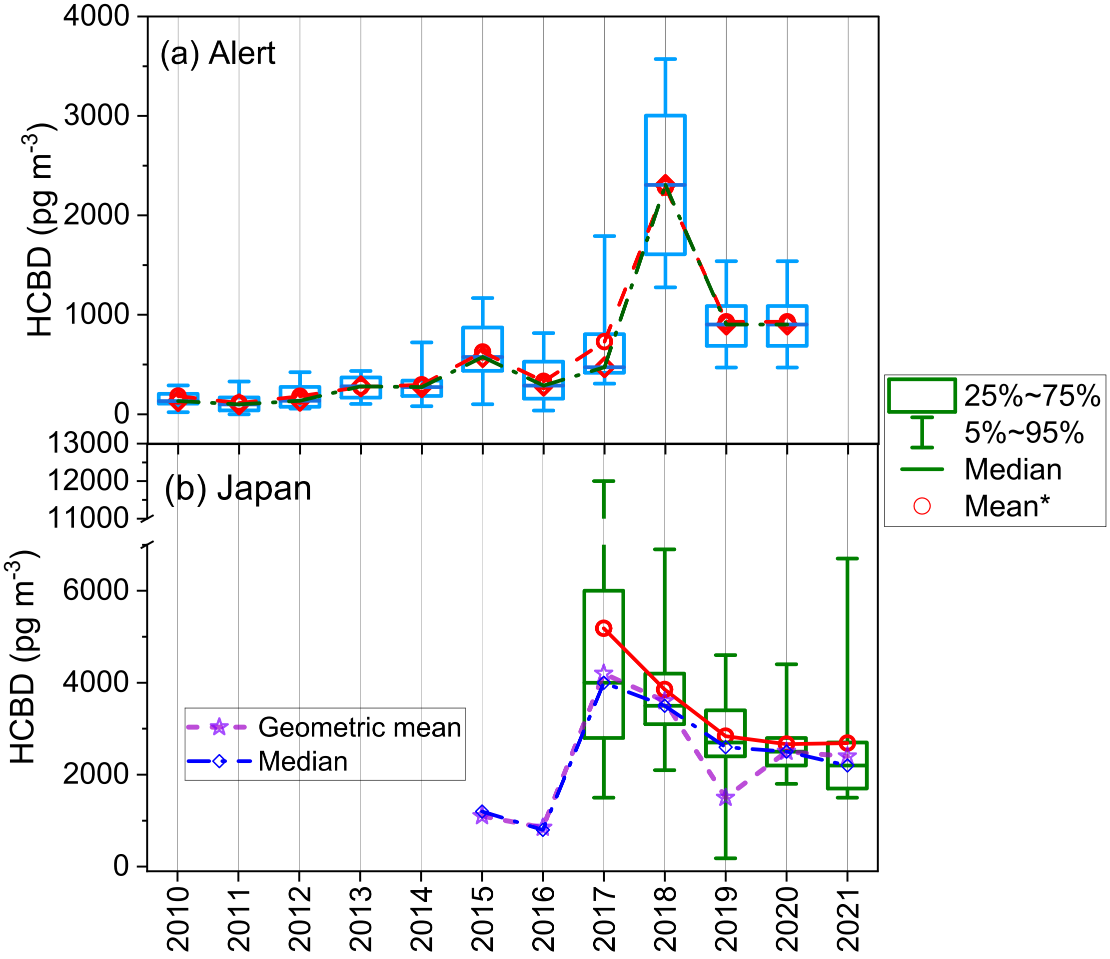

Figure SI 6. Box plot of HCBD concentrations (a) from 2010 to 2020 at the Alert site; (b) from 2015 to 2021 in Japan (*note 1*). (mean*: arithmetic mean)

Note 1. HCBD concentrations in Japan.

HCBD concentrations were measured in Japan by Ministry of the Environment, Japan since 2015. Each time, 24-hour samples were collected consecutively for three days at a single site. Sampling was conducted at 34-37 sites across the country, with a total of 102 to 111 samples taken each sampling event. The available data for comparison is from the warm season sampling event (September or October).

Data source:

Geometric mean and median: [10] (https://www.env.go.jp/chemi/kurohon/en/http2022e/index.html)

2017 HCBD concentration: [11] (https://www.env.go.jp/chemi/kurohon/en/http2018e/index.html)

2018 HCBD concentration: [12] (https://www.env.go.jp/chemi/kurohon/en/http2019e/index.html)

2019 HCBD concentration: [13] (https://www.env.go.jp/chemi/kurohon/en/http2020e/index.html)

2020 HCBD concentration: [14] (https://www.env.go.jp/chemi/kurohon/en/http2021e/index.html)

2021 HCBD concentration: [10] (https://www.env.go.jp/chemi/kurohon/en/http2022e/index.html)

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