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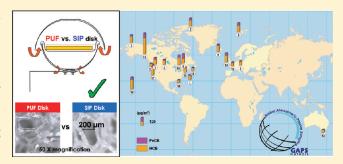
Application of Sorbent Impregnated Polyurethane Foam (SIP) Disk Passive Air Samplers for Investigating Organochlorine Pesticides and Polybrominated Diphenyl Ethers at the Global Scale

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Supporting Information

ABSTRACT: As part of continued efforts under the Global Atmospheric Passive Sampling (GAPS) Network to develop passive air samplers applicable to a wide-range of compounds, sorbent-impregnated polyurethane foam (SIP) disk samplers were codeployed and tested against conventional polyurethane foam (PUF) disk samplers. The SIP disk sampler has a higher sorptive capacity compared to the PUF disk sampler, due to its impregnation with ground XAD resin. The two sampler types were codeployed at 20 sites during the 2009, 3-month long spring sampling period of the GAPS Network. Air concentrations for chlordanes (trans-chlordane, cis-chlordane, and trans-



nonachlor) and endosulfans (endosulfan I, endosulfan II, and endosulfan sulfate) derived from PUF disk and SIP disk samplers showed near 1:1 agreement and confirmed previous results for polychlorinated biphenyls (PCBs). Discrepancies observed for α -HCH and γ -HCH in PUF disk versus SIP disk are attributed to lack of "comparability" of the PUF and SIP data sets, due to differences in effective air sampled by the two devices caused by saturation of these higher volatility compounds in the lower capacity PUF disk samplers. Analysis of PBDEs in PUF and SIP disks showed relatively good agreement but highlighted challenges associated with high blanks levels for PBDEs. The higher capacity SIP disk samplers allowed for the analysis of pentachlorobenzene (PeCBz) and hexachlorobenzene (HCBz) and revealed a relatively uniform global distribution of these compounds. The results of this study further validate the SIP disk sampler as a complement to the PUF disk sampler, with capabilities for a broad range of POPs targeted under international POPs treaties such as the Stockholm Convention on POPs and its Global Monitoring Plan.

■ INTRODUCTION

The Stockholm Convention on Persistent Organic Pollutants (POPs) under the United Nations Environmental Programme dientified a group of compounds which are of concern because of their physicochemical properties. These compounds are persistent in the environment, toxic, and can potentially bioaccumulate and transport to remote regions (e.g., Arctic). The list contains, among other compounds, organochlorine pesticides (OCPs) and hexachlorobenzene (HCBz).

Recent additions to the Stockholm Convention's list of POPs includes penta- and octa- formulations of polybrominated diphenyl ethers (PentaBDE and OctaBDE respectively), a class of brominated flame retardants, endosulfan (a current-use pesticide), and pentachlorobenzene (PeCBz), the most volatile POP listed thus far.²

Measurements of persistent organic pollutants (POPs) have been made since 2005 through the Global Atmospheric Passive Sampling (GAPS) Network. This network currently consists of \sim 55 sites on six continents at urban, rural, agricultural, and remote sites. Sampling is conducted seasonally over 3-month integration periods using polyurethane foam (PUF) disk samplers. Recently, a modified version of the PUF disk sampler—the

sorbent impregnated PUF disk or SIP disk sampler—was developed and tested for its ability to capture a broader range of chemicals having wider-ranging volatilities while at the same time producing results for the legacy POPs that are consistent with the PUF disk sampler. A pilot study was conducted in 2009 at a subset of 20 GAPS Network sites where SIP disks were codeployed with PUF disks for one 3-month sampling period. The SIP disk was shown to provide comparable results to the PUF disk for PCBs. The SIP disk samplers also allowed for measurement of more volatile species such as the polyfluorinated alkyl substances (PFAS) and volatile methyl siloxanes (VMSs) that are not captured efficiently by PUF disks.

The current study further contributes to the characterization of the SIP disk sampler by comparing SIP and PUF disk results at the 20 pilot study sites (see Supporting Information (SI) Figure S1) for legacy organochlorine pesticides (OCPs), endosulfan, and polybrominated diphenyl ethers (PBDEs). Results are also

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reported for the first time under the GAPS Network comparing PeCBz and HCBz.

■ EXPERIMENTAL SECTION

Details on sample collection, preparation, extraction, quantification, quality assurance/quality control (QA/QC), and derivation of sample volumes from depuration compounds have been previously published. Briefly, samples collected from 12 background, 4 polar, 3 urban, and 1 agricultural site were spiked with a range of recovery standards and extracted using Soxhlet extraction. Extracts were reduced by rotary evaporation and nitrogen flow to a final volume of 500 μ L. After adding internal standards, samples were analyzed using gas chromatography/mass spectrometry (GC/MS).

Air sample extracts were quantified for 19 OCPs [α -, β -, γ -, and δ -hexachlorocyclohexanes (HCHs), trans-chlordane (TC), cis-chlordane (CC), trans nonachlor (TN), heptachlor (HEPT), aldrin, dieldrin, endosulfan I and II (endo I and II), endosulfan sulfate (endo-SO₄), o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD, o,p'-DDT, and p,p'-DDT]; pentachlorobenzene (PeCBz), hexachlorobenzene (HCBz), and selected polybrominated diphenyl ethers (BDE-17, 28, 47, 66, 71, 85, 99, 100, 138, 153, 154, 183, 190, and 209).

Field blanks (n = 40) were collected at all 20 sites for both PUF and SIP disk samplers.

Depuration compounds (i.e., labeled compounds having a range of volatilities and that do not exist in air) were spiked on PUF disks prior to their deployment in order to determine site-specific sampling rates used for estimating sample air volumes (for details see Genualdi et al., 2010).⁷

■ RESULTS AND DISCUSSION

QA/QC. Concentrations of the OCPs, PeCBz, and HCBz in all field blanks were below the limit of quantification (defined as the lowest calibration point); therefore no blank correction was necessary.

The average site-specific sampling rate during this study was $6.0 \pm 3.9 \,\mathrm{m}^3/\mathrm{day}$ (n=20) or 5.9 ± 4.0 (n=19) when excluding one site that was subjected to a exceptionally high sampling rate likely associated with strong winds. This average sampling rate is slightly higher but not significantly different (p < 0.05) than the average sampling rates estimated for all GAPS sites by Pozo et al. of 4.78 ± 2.3 (n=12) or 3.9 ± 2.0 (n=25). The higher sampling rates observed under this pilot study compared to the usual GAPS Network sites may reflect a greater proportion of coastal sites which are typically windier, leading to increased sampling rates.

Eleven OCPs were below the limit of quantification in both PUF and SIP disks. These included β - and δ -HCH, heptachlor (HEPT), aldrin, dieldrin, o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD, o,p'-DDT, and p,p'-DDT.

For the PBDEs, field blanks were relatively high for both PUF and SIP disk samplers. This indicates that the source of contamination was not solely attributed to either XAD or PUF. We could compare blank values in PUF and SIP disk samplers in 25 cases, where the concentrations were above detection limits in both PUF and SIP disk samplers (12 for BDE 47, 3 for BDE 138, 7 for BDE 153, 2 for BDE 190, and 1 for BDE 209). In most of the cases (19 of 25) concentrations were higher in the SIP disk samplers, by an average by 65%. When averaged over all cases,

the concentration in SIP disk samplers was 20% higher (n = 25). Unlike OCPs that were not detected in blanks, PBDEs were present in both SIP and PUF blanks for several congeners. Method detection limits were calculated for these congeners and are listed in SI Tables SI5 and SI7. The reported data for PBDEs fell below the MDL values in most cases and blank corrected concentrations are reported.

Comparison of PUF Disk Versus SIP Disk. Air concentration results for OCPs and PBDEs that were detected in both PUF and SIP disks are summarized in SI Tables S2—7. The air concentration levels are generally consistent with previously published results from the GAPS Network³ and will not be discussed further in terms of absolute magnitudes. The main objective of the pilot study was to compare the performance of the SIP and PUF disk samplers for additional target compounds. Results of this comparison for OCPs is presented in Figure 1 as linear regressions of SIP vs PUF derived air concentrations. In Figure 2, air concentrations derived from SIP disks are normalized to results from PUF disks for all detected compounds. We elaborate on this comparison according to compound classes.

Hexachlorocyclohexanes. Regressions of PUF disk and SIP disk-derived air concentrations for α -HCH and γ -HCH (Figure.1, top panels) show poor to moderate agreement with slopes ranging from about 0.25 to 0.9, and C_{air} values for SIP disks that are typically about a factor of 2 higher compared to PUF. In the absence of high volume air monitoring data for HCHs at all sites covering the entire deployment period, the reason for this discrepancy is not entirely clear but may be related to the fact that the two samplers are integrating over different time periods. The SIP disk sampler, due to its greater sorptive capacity is sampling linearly over the entire 3-month deployment period, resulting in an equivalent sample volume $(V_{\rm air})$ of about 700 m³. Based on previous calibration work for the PUF disk sampler 11 it is estimated that only 60-80% (average for all sites) of this integration period is covered by the PUF disk in the current study. The air volumes derived for the PUF disks for α -HCH and γ -HCH are based on previous calibrations by Shoeib and Harner (2002)¹¹ and calculated using a template available from the GAPS Network (gapsnetwork@ec.gc.ca), consequently these data sets are not truly "comparable" since they represent different sample air volumes. This is illustrated in Figure 2 that compares C_{air} and V_{air} values for SIP and PUF by expressing them as a ratio. Values close to unity for $C_{\rm air}$ ratios indicate good agreement and values close to unity for V_{air} ratios indicate comparability of the data sets.

Chlordanes and Endosulfans. Unlike the HCHs, air concentrations for chlordanes (TC, CC, and TN) and endosulfans (endo I, endo II, and endo-SO₄) agree well between PUF and SIP disks with regression slopes (Figure 1) close to unity. In this case the data sets are truly comparable because the two samplers are integrating over the same time period and represent nearly the same equivalent air volume (V_{air}) as shown in Figure 2. The small discrepancy in $V_{\rm air}$ values (values of SIP/PUF slightly greater than 1) is due to reduced air sampling by the PUF disks toward the end of the sampling period as the chemicals finally begin to approach saturation in the PUF disk. This agreement is similar to what was reported by Genualdi et al. (2010) for polychlorinated biphenyls (PCBs) that showed best agreement between PUF and SIP disks for the medium and high molecular weight congeners that had similar sample air volumes in the two sampler types. These results lend further support for the use of SIP disk samplers as a complement to or replacement for the PUF disk

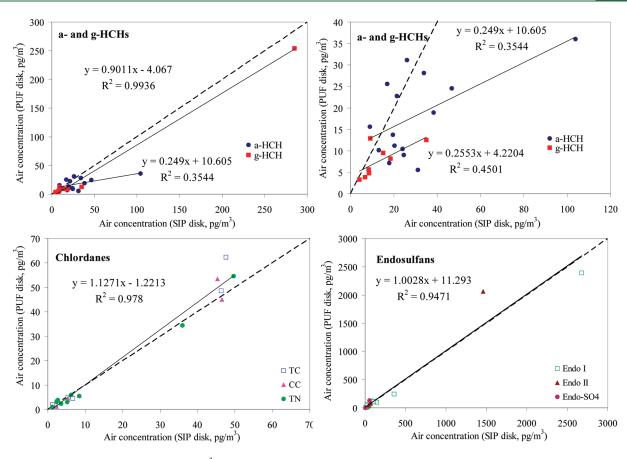


Figure 1. Regression of air concentrations (pg/m³) for selected OCPs derived from PUF disks against results from SIP disks. The 1:1 dashed line represents perfect agreement. The top right panel for HCHs excludes the single high value for g-HCH. All regressions are significant (p < 0.05).

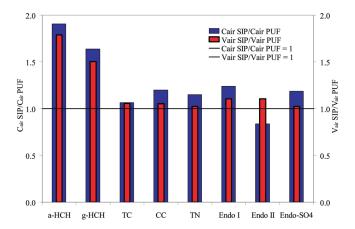


Figure 2. Average ratios for target compounds of (1) SIP vs PUF derived air concentrations ($C_{\rm air}$, blue bars) and (2) SIP vs PUF equivalent air sample volumes ($V_{\rm air}$, red bars). Departure from unity for $V_{\rm air}$ (e.g., as observed for α - and γ -HCH) indicates that data sets are not truly comparable due to different air masses represented by SIP and PUF disks. This leads to an increased likelihood that SIP/PUF $C_{\rm air}$ ratios will exhibit departure from unity (i.e., will disagree).

sampler and for producing consistent results for comparable data sets. Figure 3 summarizes all the data for the chlordanes and endosulfans from the 20 study sites and shows good agreement for a wide range of air concentrations spanning from 1s to 10s of pg/m^3 which is more typical for the chlordanes, to the 100s to

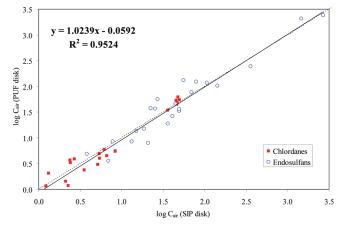


Figure 3. Regression of derived air concentrations from PUF and SIP disk samplers for comparable data (n = 45) spanning more than 3 orders of magnitude. Comparable data are included for the chlordanes (TC, CC, TN) in red squares and endosulfans (endo I, endo II, and endo-SO₄) in open blue circles. Perfect agreement is represented by the 1:1 dashed line.

even 1000s of pg/m³ for endosulfan, which is still in use as a pesticide in many parts of the world.

Polybrominated Diphenyl Ethers. PBDEs have been previously reported in GAPS Network samples but with some difficulties.³ Due to their main application as flame retardants,

PBDEs are ubiquitous in the indoor environment including instrumentation. ¹² This results in elevated blank levels compared to other target compounds that are not as prevalent indoors.

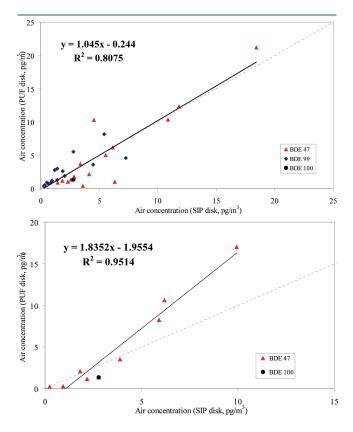


Figure 4. Top: Regression of derived air concentrations from PUF and SIP disk samplers for comparable data, that is, same sample air volumes in PUF and SIP without blank correction. Data points (n=31) include BDEs 47, 99, and 100; and Bottom: Resulting regression (n=10) after blank correction. The 1:1 dashed line represents perfect agreement.

Although PBDEs were easily detected in field blanks at high levels, the results for the lab blanks suggests that most of the contamination is occurring in the laboratory and not due to sample transport and deployment.

Figure 4 shows the regression of PUF and SIP derived air concentrations for PBDEs prior to and after blank correction.

Prior to blank correction the regression line falls close to the 1:1 line, however the data exhibit considerable scatter. After correcting the samples for blank levels, only 10 paired values remained for comparison of PUF and SIP disk samplers (9 for BDE 47 and 1 for BDE 100). This results in a tighter correlation (Figure 4) but higher slope for the PUF disks which generate \sim 1.5 times higher air concentrations compared to SIP disks.

Efforts in the Environment Canada (Hazardous Air Pollutants) HAPs laboratory to isolate the PBDE blanks problem have been inconclusive so far. The results suggest in addition to inputs from contaminated laboratory air, solvent contamination may also be an issue. The ability to maintain low laboratory blanks is one of the main challenges associated with using passive air samplers for trace air analysis of priority chemicals. This is particularly true for target compounds that are ubiquitous in indoor air and laboratory instrumentation.

In the context of blank contamination under this study, there were some differences in blank levels of PBDEs in PUF versus SIP. On average, the concentration of PBDEs in SIP disk field blanks was 20% higher than in PUF disk blanks (n = 25). This may be related to differences in the methodology of their pretreatment and preparation (which is more involved for the SIP disk) and also to the higher sorption capacity of SIP versus PUF. These difficulties arising from blank issues highlight potential challenges for using SIP disk samplers for sampling PBDEs in ambient air. However, we expect that contamination issues are partly laboratory-specific and so the severity of the blank problem for PBDEs will vary among laboratories.

Pentachlorobenzene and Hexachlorobenzene in SIP Disks. Figure 5 shows the spatial map of PeCBz and HCBz air concentrations derived from SIP disks. This is the first time that



Figure 5. Pentachlorobenzene and hexachlorobenzene concentrations in air (pg/m^3) derived from SIP disk samplers deployed at 20 GAPS Network sites in April-June, 2009.

pentachlorobenzene (PeCBz) and hexachlorobenzene (HCBz) have been reported using SIP disks under the GAPS Network and the first global scale data for PeCBz. Because of their low log $K_{\rm OA}$ values (log $K_{\rm OA}$ = 6.3 for PeCBz and 6.9 for HCBz at 25 °C¹³), the sorptive capacity of PUF disks for PeCBz and HCBz is limited, resulting in very low samples air volumes. SIP disks however have a much greater sorptive capacity which allows them to accumulate PeCBz and HCBz in the linear phase so that the amounts in SIP disks can be converted to air concentrations, which has been previously reported.

Air concentrations ranged from (2.5–75) pg/m³ for PeCBz and (37–240) pg/m³ for HCBz. Detailed results are provided in SI Table S5. The lowest concentrations of both compounds were found at Ucluelet, BC and the highest concentrations were detected at samples from Little Fox Lake, YK. The results demonstrate the uniformity (i.e., generally within an order of magnitude) of air concentrations of PeCBz and HCBz over continental scales which is consistent with other investigations. ^{14–17} For example Shen et al. used XAD-based passive air sampler to investigate pesticides in the North America and found PeCBz ranging from (17–136) pg/m³ and HCBz in the range (32–131) pg/m³.

The relatively uniform global distribution of PeCBz and HCBz is related to their emission history and physical chemical properties. PeCBz in not manufactured for any commercial uses, but is generated as a byproduct of a variety of industrial processes. PeCBz is also generated during solid waste combustion and biomass burning. 18 PeCBz and HCBz have long residence times in the atmosphere, which give them potential for long-range transport and allow them to be widely distributed and exhibit fairly uniform air concentrations. ¹⁴ In 2009 PeCBz joined HCBz on the list of targeted chemicals under the Stockholm Convention on Persistent Organic Pollutants (POPs), the largest global POPs Treaty.² It was also targeted under the POPs protocol of the Convention on Long-Range Transboundary Air Pollution (LRTAP) under the United Nations Economic Commission for Europe. HCBz has been historically used for variety of purposes including as a fungicide on seeds; as a wood-preserving agent; and a multitude of industrial processes, for example, as a porositycontrol agent or as a chemical intermediate in dye manufacturing. 19

Implications. This study adds to the characterization of SIP disk samplers for a larger set of air contaminants that are of interest to researchers, risk assessors and regulators. Findings show that air concentrations derived from SIP disks agree with results using conventional PUF disks, for OCPs that are listed under the Stockholm Convention on POPs.² This is consistent with an earlier publication showing that SIP and PUF disks generated comparable results for PCBs.7 This further supports the prospect of using SIP disks as a complement, or alternatively, as a replacement for the PUF disks. SIP disks are able to capture a broader range of air contaminants and provide another costeffective tool for measuring POPs in air under the Global Monitoring Plan of the Stockholm Convention on POPs. The agreement between SIP and PUF for high K_{OA} chemicals suggests that uptake by SIP disks is governed by the same sampling kinetics as the PUF disks (i.e., same sampling rate) and that this is air-side controlled. SIP disks have the advantage of a higher sorptive capacity and are able to maintain linear-phase sampling for long deployment periods compared to PUF disks. A relatively small amount of XAD powder (\sim 0.5 g) is required to achieve this enhanced sorption capacity. The ground XAD used in SIP disks has a higher sorptive capacity compared to XAD spheres due to an enhanced surface area per unit mass.²⁰ It is also

easier to clean the XAD powder as more surfaces are exposed to solvent during extraction. This reduces the potential for a "memory effect" or blank issues. Maintaining low blank levels in passive air samplers will be an ongoing challenge as many of the emerging chemical classes are associated with indoor applications (e.g., PBDEs).

In addition to demonstrating the good agreement between PUF and SIP disks samplers for OCPs and PBDEs, this study also generated the first global-scale data for PeCBz and HCBz from SIP disk samplers. More work is required to calibrate the SIP disk sampler for PeCBz, HCBz, and other volatile POPs of interest under field conditions and to assess the limits of its sorptive capacity so that maximum deployment periods and sample air volumes can be assessed with greater certainty.

ASSOCIATED CONTENT

Supporting Information. Additional details on sample collection, site details and air concentration results for OCPs. This material is available free of charge via the Internet at http://pubs.acs.org.

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