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Occurrence and Distribution of Two Hydroxylated Polychlorinated Biphenyl Congeners in Chicago Air

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Abstract

We measured hydroxylated polychlorinated biphenyls (OH-PCBs) in both gas and particulate phases in 30 Chicago air samples, the first report of OH-PCBs in environmental air samples. Concentrations of 2OH-PCB2 and 6OH-PCB2 in both phases were similar to PCB2 measured in the same samples, from non-detect to 11 pgm^{-3} and 12 ngg^{-1} for the gas and particulate phases, respectively. We found that OH-PCB2s sorbed more to particulates than did PCB2; seasonal variability was larger than spatial variability across Chicago; and partial pressure and temperature strongly correlated with the two OH-PCBs ($p < 0.0001$). Similar 6OH-PCB2:2OH-PCB2 ratios were found in our air samples and Aroclors, suggesting that Aroclors are a legacy source of OH-PCB2s to the atmosphere and appear to be volatilizing proportionally to PCBs in Aroclors. Although degradation by the hydroxyl radical has been proposed as an efficient loss process for airborne PCBs, we found no evidence that this mechanism results in the formation of OH-PCB2s.

TOC

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Supporting Information

Tables of analytical details, raw data, sampling locations map, and (K_p) and correlation figures are included in the SI. This material is available free of charge via the Internet at <http://pubs.acs.org>.

The authors declare no competing financial interest.

Introduction

Hydroxylated polychlorinated biphenyls (OH-PCBs) are generally regarded as oxidation products of PCB metabolism in humans and other organisms; ^{1–5} however, they are beginning to be understood as environmental contaminants ^{6, 7} with distinct and important toxicological effects. ^{4, 8–10} The first report of OH-PCBs in the abiotic environment, by Ueno et al., reported on several OH-PCB congeners in rain, snow, and surface waters sampled in Ontario, Canada. ⁷ They hypothesized that the occurrence of OH-PCBs in precipitation could be by oxidation of PCBs by hydroxyl radicals ($\cdot\text{OH}$) in cloudwater and raindrops. ¹¹ Further, they posed that comparatively high levels of OH-PCBs in surface waters collected near sewage treatment plants could be the result of microbial oxidation and/or ozonation and advanced oxidation treatment processes in the presence of PCBs.

Recently, and for the first time, we reported the presence of OH-PCBs in freshwater sediments from the Indiana Harbor and Ship Canal, as well as in original Aroclor mixtures. ⁶ The presence of OH-PCBs in Aroclors is significant because it suggests the presence of OH-PCBs in the environment everywhere that Aroclor contamination is found, thus providing a previously unconsidered source of OH-PCBs.

Although OH-PCBs have never been measured in environmental air samples, it has long been hypothesized that oxidation of PCBs by $\cdot\text{OH}$ serves as an important permanent loss mechanism of PCBs in the natural environment. ^{7, 12–24} While it is generally understood that formation of OH-PCBs is the first step in this removal process, their atmospheric lifetimes are predicted to be quite short in comparison to their parent PCB compounds, ^{15, 25} making them difficult to detect in the air as reaction intermediates. Still, there is no definitive information regarding the presence of OH-PCBs (or lack thereof) in environmental air samples.

The primary aim of this work was to detect and measure OH-PCB congeners in air samples collected in Chicago. Due to a clear Aroclor PCB signal in Chicago air and the presence of OH-PCBs in Aroclor mixtures, we hypothesized that OH-PCBs are present in ambient urban air at levels proportional to their presence in commercial Aroclors and that volatilization from Aroclor contaminated sources is a major mechanism for the occurrence of OH-PCBs in air. To address this hypothesis, we analyzed air samples for 72 OH-PCBs for which commercial standards were available. For this research we focus on the most frequently detected OH-PCBs - two OH-PCB2 congeners. Further, we report PCB2 in the samples to investigate any relationship with the OH-PCB2 congeners.

Materials and Methods

Air Sampling

The sampling method used to collect air samples is described in detail elsewhere. ^{26, 27} Briefly, modified high volume air samplers (Hi-Vols) were mounted to the rear of two mobile medical vans (Mobile C.A.R.E. Foundation of Chicago) and run for a period of 6–8 hours while the medical vans remained at a sampling site. The Hi-Vols collected both gas and particulate phases via Amberlite XAD-2 resin (XAD) and quartz fiber filters (QFF),

respectively. Samplers operated at a flow rate of $0.4 \text{ m}^3 \text{ min}^{-1}$. Thirty XAD and thirty QFF samples throughout 2009 from ten sites across Chicago were extracted and analyzed for this investigation (Table S1 and Fig S1). Of the ten sites, four sites were measured once during the summer, four sites were measured four times during the year, and two sites were measured five times during the year. The sampling sites were selected to form a north to south transect through the city of Chicago. After collection, samples were shipped to the University of Iowa, where they were kept refrigerated at -20°C until extraction.

Analytical Methods

The analytical methods for PCBs and OH-PCBs were according to Hu et al.²⁷ and Marek et al.,⁶ respectively. Prior to extraction, samples and method blanks consisting of cleaned XAD and QFF were spiked with 5 ng of OH-PCB surrogate standards ^{13}C -4'-OH-PCB12 (^{13}C -4'-hydroxy-3,4-dichlorobiphenyl), ^{13}C -4'-OH-PCB120 (^{13}C -4'-hydroxy-2,3',4,5,5'-pentachlorobiphenyl), and ^{13}C -4OH-PCB187 (^{13}C -4-hydroxy-2,2',3,4',5,5',6-heptachlorobiphenyl); and 12.5 ng of PCB surrogate standards PCB14 (3,5-dichlorobiphenyl), PCB65-d5 (2,3,5,6-tetrachlorobiphenyl-d5, deuterated), and PCB166 (2,3,4,4',5,6-hexachlorobiphenyl). After sample extraction using pressurized solvent extraction (Dionex ASE 300), PCB and OH-PCB fractions were separated by liquid-liquid partitioning.⁶ The PCB fraction was cleaned using acidified silica gel and concentrated.²⁷ The OH-PCB fraction was derivatized with diazomethane, converting OH-PCBs to methoxylated PCBs (MeO-PCBs) prior to cleanup with acidified silica and concentration.⁶ Before instrumental analysis, extracts were spiked with 20 ng of internal standards PCB30-d5 (2,4,6-trichlorobiphenyl-2',3',4',5',6'-d5, deuterated) and PCB204 (2,2',3,4,4',5,6,6'-octachlorobiphenyl).

Gas chromatography with tandem mass spectrometry (GC-MS/MS, Agilent 7000) equipped with Supelco SPB-Octyl capillary column in multiple reaction monitoring (MRM) mode was used to identify and quantify all 209 PCBs as 171 individual or coeluting congeners and 72 OH-PCB congeners as 65 individual or coeluting congeners. Details of the GC-MS/MS parameters are included in the SI (Tables S2, S3 and S4).

QA/QC

XAD and QFF samples were analyzed separately in batches of nine to eleven samples (three XAD and three QFF batches total). Depending on the sampling media, either a cleaned XAD or a combusted QFF sample was run as a method lab blank with each batch to monitor potential contamination and also to calculate the method detection limit (MDL) for each PCB and OH-PCB congener. MDLs were calculated as the upper limit of the 95% confidence interval of each individual or coeluting congener mass from the method blanks. Masses below the MDL were substituted with zero. Precision in the quantification was assessed with surrogate standard recoveries for each sample and each chemical category, summarized in the SI.

Congener identity was confirmed by reanalyzing the samples using the same instrument methods equipped with a DB-5 and a DB-1701 capillary columns (Agilent Technologies). Figure 1 shows MRM chromatograms for the 2 OH-PCB2 congeners with their respective

calibration standard solution for the three capillary columns used. Additional QA/QC information is presented in the SI.

Results and Discussion

Concentrations, spatial and temporal variability

In the gas-phase, 2OH-PCB2 (2-hydroxy-3-chlorobiphenyl) and 6OH-PCB2 (6-hydroxy-3-chlorobiphenyl) were detected with 100% frequency. In the particulate phase, 2OH-PCB2 and 6OH-PCB2 were detected with 41 and 66% frequency, respectively. We did not detect the remaining 69 OH-PCB congeners from our standards in either the gas or particulate phases, which included all remaining OH-PCB2 congeners. Significantly higher concentrations of gas phase 2OH-PCB2 were observed in relation to 6OH-PCB2 (Kruskal-Wallis, $p = 0.001$, Table 1). OH-PCBs in the particulate phase showed a different result, where no significant difference was found between these two OH-PCB2 congeners (Kruskal-Wallis, $p = 0.6$). Tables S5 and S6 in the SI show the individual concentration of each congener and each location.

Previously, we frequently detected PCB2 in air, sediment, and human blood serum.^{3, 27–29} To investigate any possible associations, similarities or differences between 2OH-PCB2 and 6OH-PCB2 and their corresponding PCB2, we compared PCB2 and OH-PCB2 data from this study. In this study, we detected PCB2 in 100% of gas phase samples and 89% of particulate phase samples. PCB2 was significantly higher than the two OH-PCB2 congeners in the gas phase (Kruskal-Wallis, $p < 0.006$), but not significantly different in the particulate phase (Kruskal-Wallis, $p = 0.064$). Further, the particulate fraction of PCB2 was very similar to the OH-PCB2 congeners, approximately 10%.

The mean particle/gas partition coefficient ($K_p = C_p/C_g$) for the two OH-PCBs ranged from 2.9 to 3.1 (g m^{-3}) log units, respectively (Table 1). PCB2 averaged a log (K_p) of 2.4, which is lower than the OH-PCB2 congeners. Interestingly, PCB2 was only significantly lower compared to 6OH-PCB2 (Kruskal-Wallis, $p = 0.005$), but not compared to 2OH-PCB2 (Kruskal-Wallis, $p = 0.09$). These results might suggest that OH-PCBs sorb more onto particles than PCBs.

OH-PCBs vary more over seasons (or temperature) than over space within the city. Our measurements of gas phase OH-PCBs and PCB2 at the same site and over the course of the year varied on average by a factor of 24, whereas measurements collected at the 10 sites at a very similar time of the year (summer) varied on average by a factor of 10. It was not possible to determine any spatial or temporal variability across Chicago for the particulate phase because of the lower detection of the chemicals (~ 50%).

Gas-phase OH-PCBs in Chicago are strongly correlated with temperature. Almost 50% of the OH-PCB concentration variability in Chicago air was explained by temperature (Clausius-Clapeyron correlation, Figure 2).^{26, 27} The slope obtained from the Clausius-Clapeyron correlation for OH-PCBs ranged from 34 to 37 kJ mol^{-1} which is lower than experimentally determined values for PCB congeners with the same number of chlorines.³⁰ Conversely, PCB2 showed no correlation with temperature ($p = 0.39$). These results suggest

that volatilization was one of the major release processes of OH-PCBs to the atmosphere but not necessarily for PCB2. It is possible that release of PCB2 to the atmosphere is from non-volatilization emission sources, or that PCB2 levels are affected by other atmospheric processes, which do not affect the OH-PCBs detected in this study to the same degree.

Congener profile evaluation

It is well known that the historical use of Aroclors remains a major source of airborne PCBs in urban areas. We have previously reported that OH-PCBs are present in Aroclors and this study suggests that Aroclors function as a source of airborne OH-PCBs. The ratio of 2OH-PCB2 to 6OH-PCB2 is remarkably similar to the four Aroclors (Fig S3). Additionally, the gas phase data for these two OH-PCB2 congeners show strong correlation ($p < 0.001$), indicating that these chemicals are likely coming from the same source, their rates of volatilization are very similar, and they behave very similarly in the atmosphere.

Implications:

Our results suggest a volatilization process is responsible for the presence of these two OH-PCB congeners in the air, and not reactions between PCBs and $\cdot\text{OH}$. The only hydroxylated forms of PCB2 we detected contain their OH on the chlorinated ring and in ortho positions - contrary to what is expected for PCB OH reaction products.^{11, 20} This finding, in conjunction with the strong similarity in OH-PCB2 ratio to Aroclors and strong temperature correlation, points to Aroclors as the source of OH-PCBs to the atmosphere. Further, if the OH-PCB2 congeners we detected were products of a first order reaction between PCB2 and $\cdot\text{OH}$, we should expect to find a correlation between the concentrations of PCB2 and the hydroxylated PCB2 congeners as PCB2 is hydroxylated according to a pseudo first order reaction. However, no correlations were found ($p > 0.8$) (Fig S4). We investigated the “degradation ratio” suggested by Ueno et al. regarding the possibility of decay of PCBs to OH-PCBs in the environment.⁷ Our ratios of gas phase concentrations between 2OH-PCB2:PCB2 and 6OH-PCB2:PCB2 did not show any characteristic degradation pattern. We also found higher values than reported for snow, rain and surface water collected in Ontario, Canada. Further, we evaluated these ratios temporally and spatially and we did not find any particular trend, except from what we have already stated. In general, the OH-PCB/PCB ratios are higher in the summer and lower in the winter, because OH-PCBs are higher in summer and PCB2 is generally constant during the entire year, except from one sample (Zapata, 1/6/2009). However, the differences between the ratios were not significant ($p > 0.5$).

The occurrence of airborne OH-PCBs in Chicago air is important as it presents a previously unknown human exposure to potentially toxic chemicals.^{5, 8–10} Our results provide more insight to the discovery of OH-PCBs in Aroclors.⁶ Aroclors were widely-used as electrical insulating fluids in capacitors and transformers and also blended with construction materials such as caulks and sealants.^{31, 32} Because these materials were widely used both indoors and outdoors, these materials are likely to be sources of potentially toxic OH-PCBs. Finally, if Aroclors and materials blended with Aroclors can be identified and remediated, human exposure to both PCBs and OH-PCBs will be reduced.

Supplementary Material

Refer to Web version on PubMed Central for supplementary material.

Acknowledgments

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Figure 1. Multiple reaction monitoring chromatograms for the 218/168 ion pair using: (i) Supelco SPB-Octyl, (ii) DB-5 and (iii) DB-1701 capillary columns from Agilent Technologies. Lines in red are the calibration standard solution and lines in black are the air samples. 2'OH-PCB2 was not detected in the air samples.

Figure 2.

Clausius-Clapeyron correlation ($\ln P = -H_v/RT + C$) for OH-PCBs detected in Chicago air. P is the partial pressure, H_v is the apparent enthalpy of vaporization, R is the ideal gas constant and T is the absolute temperature. 2OH-PCB2 (green triangle, $H_v = 34 \pm 6.6 \text{ kJ mol}^{-1}$, $p < 0.001$), and 6OH-PCB2 (cyan diamond, $H_v = 37 \pm 5.8 \text{ kJ mol}^{-1}$, $p < 0.001$).

Table 1

Range, geometric mean (GM), and geometric standard deviation (GSD) of gas and particulate phases concentrations of 2OH-PCB2, 6OH-PCB2 and PCB2 in Chicago air samples (n=30).

	Gas phase (pg m^{-3})			Particulate phase (ng g^{-1})			Log K_p ($\text{m}^3 \text{g}^{-1}$)		
	Min	Max	GM (GSD)	Min	Max	GM (GSD)	Min	Max	AM (SD)
2OH-PCB2	0.18	11	1.2 (2.2) [^]	< MDL	5.8	0.89 (2.7)	2.2	4.7	2.9 (0.72)
6OH-PCB2	0.13	4.5	0.62 (2.2) ^{*^^}	< MDL	12	0.83 (3.9)	2.3	4.6	3.1 (0.58) ⁺
PCB2	0.61	33	2.4 (2.5)	< MDL	30	0.44 (5.0)	0.96	3.8	2.4 (0.77)

Asterisks indicate significant difference between OH-PCBs (Kruskal-Wallis, * = $p = 0.001$). Carets indicate significant difference between PCB2 and OH-PCB2 congeners (Kruskal-Wallis, ^ = $p = 0.006$, ^^ = $p < 0.001$).

Plus indicate significant difference between PCB2 and OH-PCBs (Kruskal-Wallis, + = $p = 0.005$).

Particle/gas partition coefficient (K_p) for 2OH-PCB2, 6OH-PCB2 and PCB2 in Chicago air are also presented in Fig S2.