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Sampling and Analysis Artifacts Caused by Elevated Indoor Air Polychlorinated Biphenyl Concentrations

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Polychlorinated biphenyl (PCB) concentrations were measured in the indoor air of several public buildings in Bloomington, IN. The concentrations ranged from 5 to 300 times higher than outdoor concentrations and the indoor air PCB levels were highest in buildings with the earliest construction dates; see Figure 2. These elevated indoor air PCB levels have caused contamination in sample preparation as well as artifacts in sample collection. PCBs in procedural blanks in a laboratory with an indoor air PCB concentration of 6 ng/m³ averaged 1 order of magnitude lower than those measured in a laboratory with an indoor air PCB concentration of 300 ng/m³. Air sampling conducted on the roof of a building with high indoor air PCB concentrations and roof-top ventilation gave PCB concentrations that were approximately five times above ambient levels. However, sampling on the roof of another building without roof-top ventilation gave PCB concentrations that were not artificially elevated. We recommend measuring the indoor air of all sample preparation and collection sites to ensure that contamination can be recognized and mitigated.

Introduction

Prior to their ban in 1977, polychlorinated biphenyls (PCBs) were used in a wide range of closed-system electrical devices, such as transformers, capacitors, and fluorescent light ballasts (1). Because of the expense of replacing these devices, federal regulations allowed them to be kept in service without removing the PCBs even after the ban (2). Some of this equipment is still in use today, and as a result, these devices pose a contamination threat if the enclosed PCBs are released.

This is a particular problem for laboratories measuring ambient PCB concentrations. If PCB-filled devices are leaking into the indoor air of the building in which the laboratory is located, the elevated PCB concentrations could contaminate samples as they are being prepared for analysis. For example, it was noted that spare electronic equipment placed next to a laboratory entrance caused excessively high laboratory PCB blank values (3). Leaking

PCB-filled capacitors in the equipment were the source. Atmospheric sampling on the roofs of buildings that house PCB-filled equipment is a related but more subtle problem. If the building itself is a PCB source, the atmospheric samples may not be representative of the ambient environment.

We report here indoor air concentrations in four public buildings in Bloomington, IN. We have used laboratories in these building to prepare samples for PCB analysis, or we have used the roofs of these buildings as sampling platforms to collect air samples. We will show that elevated PCB concentrations in the indoor air of some of these buildings have caused contamination problems for both sample collection and preparation. We suggest that one should determine the indoor air concentrations of PCBs (and other semivolatile analytes) before using the building for either sampling or analysis.

Experimental Section

Buildings. The indoor air of three buildings on the campus of Indiana University was investigated: the Geology building (built in 1961), the Business School building (built in 1966), and the School of Public and Environmental Affairs (SPEA) building (built in 1980). The Batchelor School building (built in 1973), a city of Bloomington public school, was also studied. All of these buildings had, at one time or another, been used for air sampling (from the roof) or for PCB analyses (in two laboratories).

Air Sampling. All air samples, except the pre-1995 Geology indoor air samples, were collected using a high-volume air sampler (Sierra-Misco, Berkeley, CA). High-volume samples were collected for 24 h at a flow rate of approximately 1 m³/min; the sample volumes ranged from 800 to 1200 m³. Air was first drawn through a 0.2- μ m glass fiber filter to collect particles and then through a precleaned polyurethane foam (PUF) plug to collect vapor-phase PCBs. These PUF plugs were 10 cm in diameter and 10 cm long. A more detailed description of the experimental procedures can be found in the studies of Hermanson and Hites (4), Panshin and Hites (5), and Wallace and Hites (6, 7).

Different sampling techniques were used in the 1987, 1993, and 1994 Geology building indoor air samples. The 1987 and 1994 indoor Geology building samples were collected using a low-volume air sampling technique using small PUF plugs as described by Anderson and Hites (8) and Jones (9). The 1993 samples were collected using a diffusion denuder as described by Krieger and Hites (10).

XAD-2 Procedural Blank Experiments. As part of another project, we noticed that when XAD-2 was used as a sorbent (Sigma Chemical Company, St. Louis, MO; mesh size, 20–60; surface area, 330 m²/g; pore diameter, 9 nm), it was becoming contaminated with PCBs during sample analysis. This sorbent had been solvent extracted prior to use to remove surfactants and other contaminants using the following procedure: Approximately 150 g of XAD-2 was rinsed with distilled water to remove fine particles, transferred to a Soxhlet extractor plugged with a small amount of glass wool, and extracted with the following solvents for at least 24 h each: methanol, acetone, hexane, dichloromethane, hexane, and 50% acetone in hexane. All

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solvents were from EM Science (Omnisolv; Gibbstown, NJ) and were pesticide grade. After cleaning, the XAD-2 was transferred to a beaker, covered with aluminum foil, and dried in an oven for 6 h at 65 °C. After drying, the XAD-2 was cooled and stored in sealed amber bottles at -20 °C until use

To perform a procedural blank experiment, about 20 g of XAD-2 was placed in a glass Soxhlet extractor tube plugged with glass wool. PCB congeners 14, 65, and 166 were added as recovery standards, and the XAD-2 was extracted for 24 h with 50% acetone in hexane. After extraction, the sample was rotary-evaporated to 2 mL, and the solvent was exchanged two times with 75-mL aliquots of hexane. The final aliquot was reduced to 2 mL. The extract was loaded onto a 4% water deactivated, 9 cm x 1.5 cm i.d. silica column topped with 1 cm of anhydrous sodium sulfate. The silica (Aldrich Chemical, Milwaukee, WI) was cleaned by heating it to 300 °C for 24 h. The sample was eluted with 25 mL of hexane; the resulting fraction contained the PCBs. The fraction was reduced to 0.5-1.0 mL by evaporation with a gentle stream of dry nitrogen, spiked with the internal quantitation standards, and analyzed as described below.

Sample Analysis. All air samples taken with PUF as the sorbent were analyzed using procedures given elsewhere (4-6). Briefly, PCBs were removed from the PUF by Soxhlet extraction. The sample extracts were passed through a silica or alumina column to remove polar interferences. The cleaned extract was concentrated to a small volume (0.1-1 mL) and injected into a gas chromatograph equipped with a constant-current electron capture detector. The GC conditions have been given elsewhere (6).

For the analysis of the XAD-2 samples, a Hewlett-Packard model 5890 gas chromatograph with a 60 m \times 250 μm i.d. (0.10 μm film thickness) J&W Scientific (Folsom, CA), DB-5 fused silica capillary column was used. One microliter of the sample was injected in the splitless mode. The GC temperature program conditions were as follows: held at 100 °C for 1 min, ramped at 1 °C/min to 240 °C, ramped at 10 °C/min to 280 °C, and held at 280 °C for 20 min. PCB congeners 30 and 204 were used as internal standards for quantitation.

Principal Component Analysis. Principal component analysis (PCA) was performed using SAS System Release 6.10 (SAS Institute, Cary, NC). A DEC AXP 7720 computer using DEC OSF/1 work-system software was used. For the principal component analysis, the 30 most abundant congeners found in the outdoor samples were used. These congeners were selected because they tended to be above 1% of the total PCB concentration. These congeners (listed in order of increasing gas chromatographic retention time) were IUPAC Nos. 18, 17, 16, 32, 25+26, 31+28, 53+33, 22, 52, 49, 44, 42+37, 41+71, 64, 74, 56+60, 92+84, 101, 99, 87+81, and 110+77. Congeners listed as pairs (e.g., 25+26) co-eluted from the GC column. For each sample, the concentration of each congener (or pair of congeners) was normalized to the total PCB concentration. These normalized concentrations were then used in the principal component analysis. The congeners selected for use in the principal component analysis are all trichloro-through pentachlorobiphenyls. While this is not a representative sampling of all PCB congeners, these congeners tended to be present in all of the samples measured in this study and are therefore best suited for inclusion in the principal component analysis. It has been our experience that, when

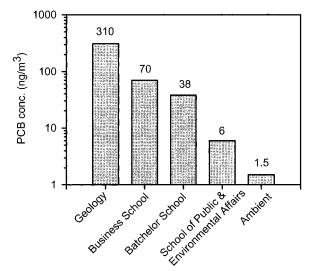


FIGURE 1. Indoor air PCB concentrations in the various buildings measured in this study. The far-right bar is the average ambient (outdoor) PCB concentration in Bloomington, IN.

TABLE 1
Indoor Air PCB Concentrations in the Geology
Building on the Dates Indicated

date	no. of samples	av air concn (ng/m ³⁾	standard deviation	ref
1987	2	220	7	8
1993	6	210	60	10
1994	6	330	60	9
1995a	2	480	7	this study
av		310	85	-

^a After ballast replacement.

all congeners are included, the PCA becomes very sensitive in discriminating between zero and non-zero values to the exclusion of most other effects. By only picking major congeners, this problem is minimized.

Results and Discussion

Indoor Air. Over the years, we have used several buildings in Bloomington to conduct our research, both for analysis and for sampling. The indoor air PCB concentrations in these buildings are given in Figure 1. In all of the buildings, except the Geology building, the air concentrations represent a single measurement. The value plotted for the Geology building is the average value of the 16 indoor air samples collected from 1987 to 1995 (see Table 1). Note that the concentration scale in Figure 1 is logarithmic and that it ranges over 3 orders of magnitude. The roofs of the Business School and Batchelor School buildings have been used as sampling platforms, and both the Geology and the School of Environmental and Public Affairs (SPEA) buildings have been used to prepare samples. The ambient (outdoor) PCB air concentrations in Bloomington average approximately $1-2 \text{ ng/m}^3$ (4, 5), and this is shown at the far right of Figure 1. The indoor air PCB concentrations are elevated in all of the buildings relative to outdoor levels, in some cases by a factor of at least 100. It is also apparent that the elevated PCB concentrations are not the same in all of the buildings; the Geology building concentration is over 50 times larger than the concentration measured in the SPEA building. We believe that the variations on these indoor PCB levels relate to the time of construction of the four

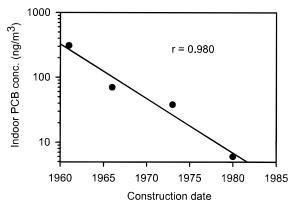


FIGURE 2. Indoor air PCB concentrations in the buildings shown in Figure 1 plotted as a function of their construction dates.

buildings. Buildings constructed before the PCB ban in 1977 would have higher PCB indoor concentrations than those constructed after the ban, presumably because the latter buildings contain fewer PCB-filled electrical devices.

PCB concentrations are highest for the buildings that were built first. In fact, there is a strong correlation between construction date and indoor PCB concentration; see Figure 2. Apparently, PCBs leaking from the various electrical devices accumulate in a building over time and cause substantially elevated air concentrations. It is interesting to note that PCB concentrations in the SPEA building are above ambient concentrations (see Figure 1), even though this building was constructed well after the U.S. PCB production ban. This elevated concentration may be caused by the connection between this building and the Business School building (the two buildings share a first floor). PCB-containing air from the Business School building could mix with air in the SPEA building, resulting in elevated concentrations in the latter.

It should be noted that while the PCB concentrations measured in the indoor air of these buildings are much higher than ambient levels, these indoor air levels are probably not high enough to cause human health problems. The Occupational Safety and Health Administration (OSHA) recommends a maximum time-weighted average exposure of 10 and 5 mg/m³ for Aroclors 1242 and 1254, respectively (11). The National Institute for Occupational Safety and Health (NIOSH) sets a recommended exposure limit at 1 $\mu g/m^3$ for both Aroclor 1242 and Aroclor 1254 (11). The more conservative NIOSH limit was the result of NIOSH considering the Aroclors to be potential occupational carcinogens, while OSHA does not. In any case, the highest recorded indoor air PCB concentration in this study (490 ng/m³) was lower than the more stringent NIOSH level by a factor of 2.

Sampling on the roof of a contaminated building may give artificially high PCB concentrations for samples taken at that location. For example, Wallace and Hites (6) recently developed a low-volume air sampler for measuring semi-volatile organic compounds in the atmosphere. To demonstrate that the new low-volume sampler performed similarly to a conventional high-volume air sampler, concurrent measurements with each sampler were taken to determine if the concentrations agreed (they did). Both PCBs and endosulfan were measured. Endosulfan is a contact and stomach insecticide that is currently used against a variety of insects that attack cereals, fruit, oilseeds, potatoes, and vegetables (12). Endosulfan is not a con-

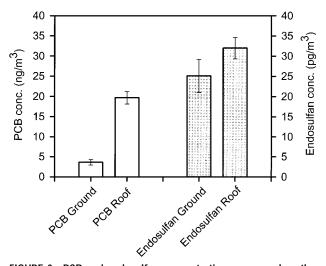


FIGURE 3. PCB and endosulfan concentrations measured on the roof of the Indiana University Business School building at 22 °C and at ground level at 19 °C. Each bar represents the average of 19 samples collected over a 5-day period; the error bars represent the 95% confidence limits of the mean.

taminant in indoor air. The low volume samples were taken on the roof of the Business School building, approximately 8 m from a large air-conditioning vent. These 19 samples were taken during the week of September 6, 1994. Eleven days later, 19 more samples were taken at ground level near a residence 4 km southeast of the Indiana University campus (7). Both sets of samples were collected, extracted, and analyzed identically, although the temperature was slightly warmer during the roof-level sampling (22 °C) than the ground-level sampling (18 °C).

Figure 3 shows the average ground-level and roof-level PCB and endosulfan concentrations. Endosulfan concentrations measured on the roof were higher than those at ground-level by approximately 25%, which may reflect the slightly warmer temperature during the roof-level sampling. Notice that the confidence limits of the means of the endosulfan samples almost overlap. The roof-level PCB concentrations, however, were over 400% higher than those measured at ground-level, and the confidence limits do not overlap. This difference cannot be explained by temperature differences between the two sampling periods. Instead, we suggest that these elevated roof-level PCB concentrations were caused by contamination of the air samples by indoor air that had come from the Business School building through the air-conditioning vents. Because there was no endosulfan in the Business School building, the roof-top measurements for this compound did not show a substantial elevation.

In the past, we have used the roof of Batchelor School building as a sampling platform. This building was constructed in 1973 and has elevated PCBs in its indoor air (38 ng/m^3) . Using the above argument, we might expect artificially elevated PCB concentrations at this site as well. However, in the 18-month study of Hermanson and Hites (4) and the 3-month study of Panshin and Hites (5), PCB concentrations ranged between 0.040 and 4.8 ng/m^3 over the temperature range of -9.3 to +35 °C. These concentrations are in agreement with other PCB measurements made in the United States as outlined by Leister and Baker (13) as well as ground-level measurements made in Bloomington (7). Hence, simply sampling on a building with high indoor air PCB concentrations does not guarantee

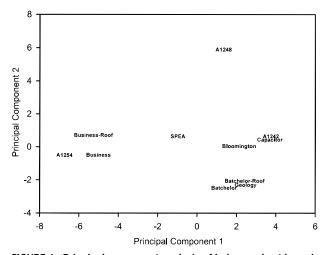


FIGURE 4. Principal component analysis of indoor and outdoor air samples collected in Bloomington compared to Aroclor fluids ("A" prefix). The indoor air samples are from the Business School, the School of Public and Environmental Affairs (SPEA), the Batchelor School, and the Geology buildings. Air samples taken on the roof of Batchelor School and the Business School are shown as well as a fluid sample from a capacitor removed from a fluorescent light ballast in the Geology building. The point labeled "Bloomington" represents the ambient (outdoor) air in Bloomington.

that samples will be contaminated. In this case, the Batchelor School building is not air conditioned, and thus, it does not have roof-top air vents. Presumably, PCBs emanating from the building are diluting to ambient levels before reaching the roof of the building.

The Monsanto Corporation, the only U.S. producer, marketed PCBs under the trade name Aroclor (2). Each Aroclor has a characteristic congener distribution. For example, Aroclor 1242 has a congener distribution enriched in less chlorinated PCBs, while Aroclor 1254 is enriched in the more highly chlorinated PCBs. By comparing congener distributions of different PCB samples, information can be gained about the source of the PCBs. Principle component analysis (PCA) is a useful technique for comparing PCB congener distributions. We used the 30 most abundant congeners for PCA; see the experimental section for the list of individual congeners. These congeners tended to be present in all samples of this study. The concentrations of the 30 congeners in a given sample were normalized to percent of the total concentration. This eliminates the possibility that the PCA will discriminate among samples simply on the basis of total concentration.

Figure 4 shows a plot of the first two principal components of the air samples taken in and on ("roof" suffix) the four buildings and at an outdoor location in Bloomington, IN. This figure also shows the first two principal components for Aroclor fluids 1242, 1248, and 1254 ("A" prefix). A sample of insulating fluid from a capacitor taken from a fluorescent light ballast in the Geology building (capacitor) is also shown. The first two principal components account for 87% of the cumulative variability of the 30 congeners used in the PCA.

Figure 4 indicates that most of the samples fall into two clusters. Both of the samples from the Business School are near Aroclor 1254. The other samples, with the possible exception of the SPEA sample, are near Aroclor 1242. Aroclor 1248 occupies its own area of principal component space. It is apparent from these data that the capacitor from the Geology building is filled with Aroclor 1242. It is

TABLE 2

Masses of PCBs (in Nanograms) Measured in Procedural Blank Experiments Performed in the Geology Building and the School of Public and Environmental Affairs (SPEA) Building

	PCB mass (ng) when extracted in	
	Geology building	SPEA building
XAD-2 cleaned in Geology building XAD-2 cleaned in SPEA building XAD-2 cleaned by Illinois	32 49 16	4.1 2.2 0.9
State Water Survey XAD-2 cleaned by Acurex Environmental Inc.	20	5.3
XAD-2 exposed to Geology building air for 24 h	96	

also apparent that the Batchelor roof, indoor Batchelor, indoor Geology, and the outdoor Bloomington samples are closely related to Aroclor 1242. The two Business School samples have congener distributions that are markedly different from all of the other samples taken in Bloomington, but they are closely related to Aroclor 1254. This result suggests that these samples were contaminated with indoor air from the Business building and that this building was contaminated with Aroclor 1254.

It should be noted that the congener distributions for the samples shown in Figure 4 do not exactly match the Aroclor fluids. One possible reason for this discrepancy is that leaking capacitors might not be the sole source of contamination. For example, some caulks and sealants that were used in buildings built in the mid-1970s contained PCBs, and these building materials caused elevated PCB levels in indoor air (14, 15). We have no specific knowledge of PCB-containing caulks or sealants being used in the buildings in this study, but it is a possibility we cannot dismiss.

Sample Preparation Contamination. Another potential problem associated with elevated indoor air PCB concentrations is the contamination of samples during their analysis. It has recently been shown by a group of British researchers that ancient soil and peat samples, which initially had little or no PCBs, rapidly sorbed PCBs when exposed to laboratory air with elevated PCB concentrations for a few days (*16*). These ancient samples sorbed so much PCB that their apparent concentrations exceeded contemporary British soil concentrations (20–30 ppb relative to 2–6 ppb). We have observed the same problems with the preparation and extraction of XAD-2.

We use XAD-2 in our laboratory as a sorbent for the collection of semivolatile organic compounds from air. To clean the XAD-2 before use, we Soxhlet extract it as described above. The masses of PCBs measured in a series of procedural blank experiments using XAD-2 cleaned at four locations are given in Table 2. A procedural blank consists of clean XAD-2 (*no* air is drawn through it) taken through the sample preparation procedure; this blank experiment indicates how much PCB contamination is introduced during the sample preparation process itself. The cleaned XAD-2 to be used for the procedural blank experiments was split into two portions; half was Soxhlet extracted in the Geology building and the other half in the SPEA building. As shown in Table 2, the amount of PCBs

was consistently higher when the XAD was extracted in the Geology building (16-49 ng) than in the SPEA building (0.9-5.3 ng).

The relatively high amount of PCBs in the blank experiment carried out in the Geology building indicates a problem for the analysis of low levels of PCBs. For example, we are currently engaged in the analysis of PCBs in air collected near the Great Lakes. Typically, PCB concentrations in these samples range from 0.05-1 ng/m³. We collect approximately 800 m³ of air in a typical sample; thus, approximately 40-800 ng of PCBs is sorbed to the XAD-2 in the field. The average PCB blank value in the Geology building is about 30 ng. This contamination level poses problems for quantitation. In this case, if an 800-m³ air sample with a PCB concentration of 0.05 ng/m³ is extracted in the Geology building, the signal to blank ratio will be 40/30 or 1.3, which is not acceptable. The same sample extracted in the SPEA building would give a signal to blank ratio of 40/3 or 13, which is more than sufficient for accurate quantitation. Based on this observation, we currently perform all PCB analyses in the SPEA building.

The elevated PCB levels in the blank samples prepared in the Geology building probably come from partitioning of PCBs from the laboratory air to solvents, sorbents, and glassware during sample processing. This conclusion is supported by the observation that XAD-2 exposed to Geology building air for 24 h sorbed 96 ng of PCBs (see the bottom row of Table 2), which is about three times larger than non-exposed XAD-2. Apparently, PCBs do not partition to the XAD-2 sorbent during cleaning by Soxhlet extraction, because the procedural blank in which the XAD-2 was cleaned in the Geology building and extracted in the SPEA building (see Table 2) had a relatively small amount of PCBs in the extract (4.1 ng). Based on this observation, it seems clear that PCBs contaminate the extract after Soxhlet extraction but before analysis; that is, during the sample cleanup procedure.

Remediation. We have recently undertaken a remediation effort in our laboratory in the Geology building to try to correct the relatively high indoor air levels. To our knowledge, the fluorescent light ballasts are the largest reservoir of PCBs in the Geology building. The electrical transformers in the basement of the Geology building no longer contain PCBs; they were filled with a non-PCB insulating fluid in 1985. Thus, we replaced the fluorescent light fixtures with new ones that contained PCB-free components in May 1995. It was our hope that by removing the PCB-filled ballasts, indoor air concentrations would decrease over time. Table 1 gives the PCB concentrations measured in our laboratory air in the Geology building in 1987 (8), 1993 (10), 1994 (9), and 1995; note that the 1995 samples were taken 3 months after removing the PCBfilled ballasts. Clearly, Table 1 shows that there has been no decrease in the indoor air PCB concentrations over time; in fact, there may have been an increase.

Perhaps the PCB concentrations are not decreasing in our laboratory, despite our remedial action, because PCB-filled ballasts in other offices and laboratories in the building continue to contaminate the indoor air of the entire building. Another possibility is that PCBs are sorbed to surfaces (such as walls and ceiling tiles) in the Geology building itself; vaporization of the PCBs from these surfaces may be keeping the indoor air concentrations elevated.

It only takes a very small amount of leakage from a ballast (or other source) to cause the indoor air PCB concentrations

we observed in the Geology building. This building has a volume of about 30 000 m^3 and a ventilation rate of approximately 5 air changes/h. To sustain an air PCB concentration of 300 $\mathrm{ng/m}^3$ (see Table 1), only 1 g of PCBs needs to volatilize into the building's air per day. This is relatively small amount given that there are still hundreds of PCB-filled ballasts in use throughout the Geology building.

Recommendations. Many older buildings have elevated PCB concentrations, and these levels will continue to pose contamination problems for measurement scientists well into the next century. We suggest the following if one is considering initiating any type of study measuring PCBs: First, determine the indoor air concentration of PCBs in the laboratory where samples will be prepared. Compare these levels to the concentrations expected in the field; if the indoor air concentrations are high, select another laboratory location. Repeat until a satisfactory location is found. Second, only use a building as a sampling platform when the indoor air of that building will not contaminate air collected on the roof of that building. If these precautions are taken, the risk of PCB contamination from indoor air exposure should be minimized.

Acknowledgments

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