

# Sampling Polycyclic Aromatic Hydrocarbons and Related Semivolatile Organic Compounds in Indoor Air

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## Abstract

*This paper summarizes the sampling and analysis methods used to collect and quantify polycyclic aromatic hydrocarbons (PAH) and PAH derivatives in indoor and outdoor air at 33 homes located in two cities, Columbus OH and Azusa CA. Two new samplers were developed for these studies: one with a flow rate of 200 l/min to collect material for bioassay and chemical analysis and a similar one with a flow rate of 20 l/min to collect samples for chemical analysis only. Both samplers simultaneously collect semivolatile organic compounds in the particulate and vapor phases. Both are quiet, transportable, and operate entirely within the sampled environment. The samplers' field performance was evaluated; that of the 20 l/min sampler is discussed in detail. Some typical data and findings from the studies are presented.*

## KEY WORDS:

Indoor air, Semivolatile organic compounds, Sampling, SVOCs, Polycyclic aromatic hydrocarbons, PAH

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## Introduction

Over the past five years, we have conducted several small studies of polycyclic aromatic hydrocarbons (PAH) and related semivolatile organic compounds (SVOCs) in indoor air in residences. Our primary goals were (1) to evaluate newly developed sampling and analysis methods under field conditions and (2) to estimate the effects of parameters such as smoking or operation of wood-burning fireplaces on the PAH and PAH derivative concentrations, to aid in the design of larger-scale indoor air studies. In this paper, we address primarily the methodology and its field evaluation.

Although several definitions of SVOCs have been put forward (Mølhave, 1990; WHO, 1989), the most general current definition consistent with the physical behavior of a wide variety of chemical classes is based on vapor pressures. The SVOCs are defined (Clements and Lewis, 1988) as compounds with vapor pressures at ambient temperatures from approximately  $10^{-2}$  to  $10^{-8}$  kPa ( $10^{-1}$  to  $10^{-7}$  torr). Because the SVOCs occur in air both in the vapor phase and adsorbed on or otherwise associated with airborne particles, sampling systems must incorporate both filters and sorbent media. We developed and tested two prototype quiet indoor air samplers for SVOCs: one with a sampling rate of 200 l/min, to collect sufficient material for bioassay and chemical analysis, and one with a flow rate of 20 l/min, to sample for chemical analysis only.

To aid in the design of large-scale indoor air studies, we measured the concentrations of selected PAH, nitro-PAH, and oxygenated PAH derivatives. Several indoor sources of PAH were included in our studies: cigarette smoking, use of a wood-burning fireplace, and use of natural gas for heating and cooking. Our first two studies included smoking; its contribution to measured PAH concentrations was overwhelmingly large compared to the contributions of the other sources. Thus the three later studies were conducted only at residences where smoking did not take place.

## Materials and Methods

### Samplers

Initially, we collected indoor air samples for use in microbioassay (Lewtas et al., 1987) as well as for chemical analysis. Approximately 1 mg of extractable organic material was required. The competing requirements of large samples for bioassay, small disturbances of airflows in the house, and minimum disturbance of the occupants by sampler noise led us to use a standard PS-1 sampling head (General Metal Works, Cleves OH), which holds a 10-cm quartz fiber filter followed by a Pyrex cartridge (60 mm i.d. x 125 mm) holding polyurethane foam (PUF) adsorbent (Chuang, Mack et al., 1987). Vacuum tubing led from the sampling head through a window port to the pump, which was located outdoors in a sound-insulated box. Sufficient material for microbioassay -- 0.38 to 28.93 mg from the filter or PUF, extracted separately -- was collected in 8 hr, sampling at approximately 200 l/min (7.2 cfm). A shorter sampling period, about 2 hr, would be required if only chemical analysis for PAH, polychlorinated biphenyls (PCBs), and pesticides is to be made (Howes et al., 1986).

Although the above sampling system performed satisfactorily, the intrusive and unique modifications required to set up the sampling system in each individual house and other practical problems, including

gradual settling of the outdoor box into the residue created by melting of snow from the heat generated by the pumping system, led to the development of a self-contained indoor SVOC sampler. It also incorporates the PS-1 sampling head. The pump, an Amtex bypass vacuum motor 116100-00 (Ametek, Lamb Electric Division, Kent OH), is in an acoustically insulated box. The box reduces the noise below NC-35, an acceptable value for a bedroom (Harris, 1979). The nominal operating flow rate is 225 l/min (8 cfm). Venting to the outside is not required in most cases (Wilson et al., 1989). The homes that we sampled, using this sampler, in three separate studies averaged 1500 ft<sup>2</sup> in size, and approximately 5-10% of the total air volume was sampled during an 8-hr sampling period. Therefore the effect of sampling, even at this relatively large sampling flow rate, did not alter the indoor concentrations substantially. A PM-10 inlet (Howes et al., 1986) and a compound annular denuder to allow measurement of the distributions of SVOCs between the particulate and vapor phases in air (Coutant et al., 1989) were also designed and constructed for this sampler.

Building upon the lessons learned in the operation of the 200 l/min sampling systems described above, we developed a similar, smaller indoor sampler for sampling for chemical analysis only (Kuhlman and Chuang, 1988). It operates at approximately 20 l/min and likewise incorporates a filter (47 mm) and a sorbent cartridge. The cartridge holds up to 30 g of XAD-2 resin and can be placed entirely within a Soxhlet extractor. The original design included acoustic insulation to reduce noise to meet a noise criterion of 35 and vibration isolation mounts for noise reduction, a carbon vane pump, a flow transducer for measurement of the flow rate, a 7-day programmable timer, a data logger, vents and a cooling fan. The entire package is the size of a small suitcase (51 x 35 x 21 cm) and is portable. A schematic diagram of this sampler is shown in Figure 1. Sufficient material

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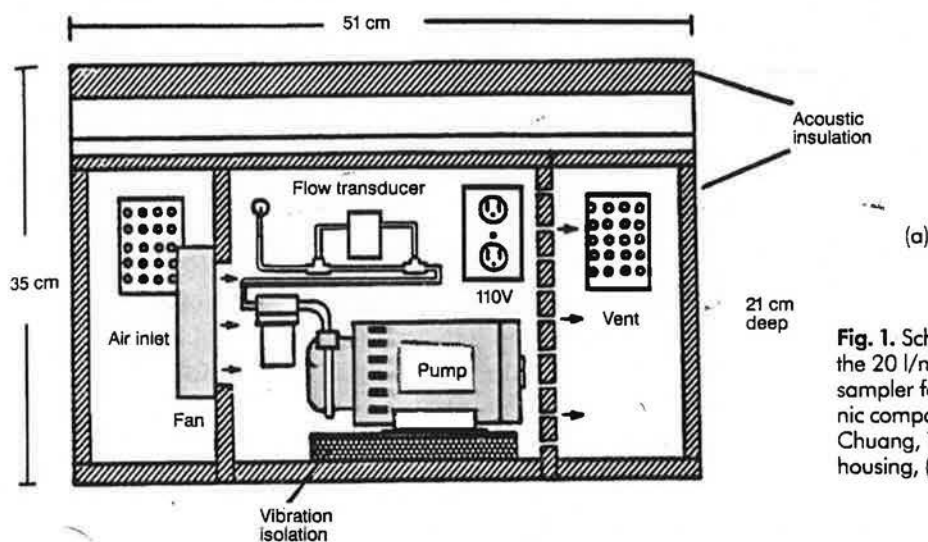
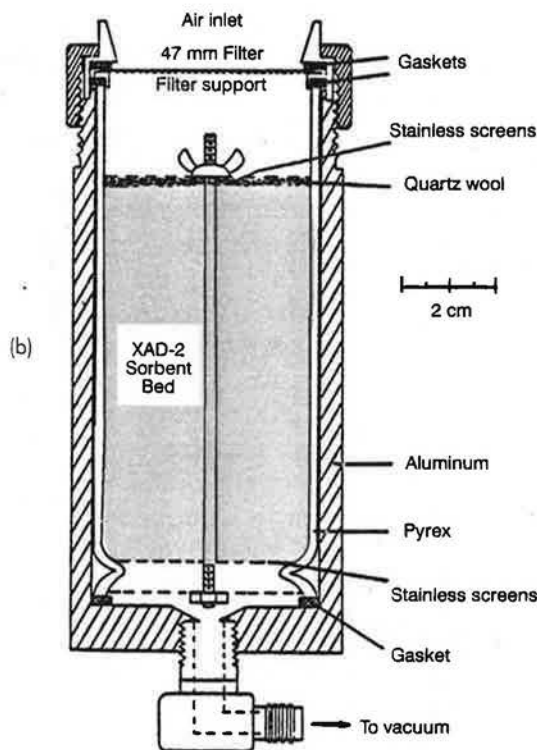


Fig. 1. Schematic diagram of the 20 l/min quiet indoor air sampler for semivolatile organic compounds (Kuhlman and Chuang, 1988). (a) Sampler housing, (b) Inlet design.



for chemical analysis of many SVOCs can be obtained in about 2 hr. To collect nitro-PAH, oxygenated PAH, and other less abundant SVOCs, sampling for 8 to 12 hr is necessary. No breakthrough of the target compounds occurred in sampling for 12 to 24 hr with this sampler.

**Sorbents**

A number of sorbents can be used to collect vapor-phase SVOCs. We originally used PUF, which has low resistance to airflow and has been validated for the collection of pesticides and PCBs (Lewis et al., 1988). However, PUF does not collect or retain well PAH with fewer than four rings, such as anthracene or similar compounds. Additionally, the PUF sample extracts gave widely varying results in microbioassay compared to extracts from other sorbents. Therefore, in all but one (Chuang, Kuhlman and Wilson, 1990) of our later studies, we used XAD-2 resin (Rohm and Hass, Philadelphia PA). Non-polar two-ring and larger aromatic compounds and most of the more polar aromatic derivatives are efficiently collected and retained on XAD-2 (Chuang, Hannan and Wilson, 1987). To collect simultaneously both PAH and nicotine or other small SVOCs, XAD-4 resin, which is chemically similar to XAD-2, but with smaller pores and greater surface area, proved to be superior (Chuang, Kuhlman and Wilson, 1990).

**Analysis**

Because the SVOCs are distributed between the gaseous and particulate phases in air, both particles and vapor must be collected and analyzed to determine the true concen-

trations. Although one can analyze filters and sorbents separately, reporting the quantitative results separately ignores changes in the amounts on the filters and sorbents during sampling or storage before analysis. These changes may occur due to revolatilization, chemical reaction, or other artifacts. The changes can be large; in some cases the artifact that results from revolatilization is of the same magnitude as the amount originally in the gas phase (Coutant et al., 1988). Therefore, except in studies specifically designed to measure phase distributions, we extract each filter-sorbent pair together and report the total concentrations.

We usually use Soxhlet extraction with 5% ether/hexane for PUF and with dichloromethane (DCM) for XAD-2 or XAD-4. If PUF is used, we extract the filter and sorbent separately and combine the extracts for analysis. A compression method (Chuang, Mack et al., 1987) also works well to extract PUF. To remove nicotine and PAH from XAD-4, we extract sequentially with ethyl acetate and DCM. Significant contamination of the indoor air during sampling by DCM or other solvent used to clean the XAD resin before use can occur. Residual DCM can be removed by fluidizing the resin in a stream of pure nitrogen gas while heating at 40 °C (Chuang, Holdren and Wilson, 1990).

Other extraction solvents may be used. Benzene/methanol has been used effectively for polar analytes (Offerman et al., 1990). Supercritical fluid extraction (SFE) with CO<sub>2</sub> is effective in some cases for non-polar PAH (Hawthorne and Miller, 1986; Wright et al., 1987), although modifiers may be necessary to remove polar materials. The use of SFE for air sample analysis is still under investigation.

For PAH analyses, we use GC/MS rather than HPLC, because it requires little clean-up and is therefore more cost-effective (Chuang, Mack et al., 1987). To achieve the highest sensitivity, positive chemical-ionization (PCI) GC/MS gives good results for

PAH. For nitro-PAH and other electrophilic compounds, we use negative CI GC/MS; to achieve quantifiable levels of the nitro compounds, it has been necessary to use sample volumes of at least 300 m<sup>3</sup> of air. Extracts are concentrated to 1 ml, and 1.0-2.0 µl is injected in splitless mode onto the GC column, typically an Ultra No. 2 (Hewlett-Packard) or a DB-5 (J&W) fused silica capillary column (50-60-m length, 0.31-mm i.d., 0.17-0.25-µm film thickness), with the column outlet located at the inlet of the MS source. The GC is held at 50 °C for 2 min, then programmed to 100 °C over 2.5 min, then to 300 °C at 8 deg/min. An internal reference, usually 9-phenylanthracene, which is absent from most environmental samples, is convenient for quantification. The analytical detection limit is approximately 1 pg on-column. Methane is the carrier gas for the NCI analyses, and the ionization is 70 eV in the electron impact mode and 150 eV in the CI mode. The measurement detection limit, using selected ion monitoring, is roughly 10 pg/m<sup>3</sup>.

### Target Compounds

The original target compounds were chosen because of their abundance in air and thus potential utility as indicators of overall PAH levels (naphthalene, anthracene, phenanthrene, fluoranthene, pyrene), their carcinogenicity or mutagenicity (benz[a]anthracene, quinoline, chrysene, benzo[fluoranthene] isomers, benzo[a]pyrene, indeno[1,2,3-c,d]pyrene, 1-nitropyrene, 2-nitrofluoranthene), their association with specific sources, e.g., motor vehicle exhaust (cyclopenta[c,d]pyrene, benzo[g,h,i]perylene, coronene) or tobacco smoke (quinoline, isoquinoline), and their historic importance (benzo[a]pyrene, benzo[e]pyrene). We later included a possible wood-smoke marker, retene (1-methyl-7-isopropyl phenanthrene), a vapor-phase tobacco smoke marker (nicotine), and several other PAH and PAH nitration and oxidation products that we identified in air samples (acenaphthylene, 1-nitronaphthalene, 2-nitro-

other electrophilic reactive CI GC/MS; to some of the nitro compounds necessary to use sample volume of air. Extracts are 1.0-2.0  $\mu$ l is injected to the GC column, (Hewlett-Packard) or a capillary column i.d., 0.17-0.25- $\mu$ m column outlet located source. The GC is then programmed to 300 °C at 8 deg/min, usually 9-phenylanthracene from most environmental detection limit is approximately. Methane is the major component in the analyses, and the ionization is by electron impact in EI mode. The measurement is using selected ion monitoring.

Compounds were chosen based on their concentration in air and thus their contribution to the total of overall PAHs. These include anthracene, phenanthrene, their carcinogenic derivatives, benz[a]anthracene, benzo[a]fluoranthene, indeno[1,2,3-c,d]pyrene, nitrofluoranthene, specific sources, e.g., cyclopenta[c,d]pyrene, coronene) or benzo[a]pyrene, and also included a possible source (1-methyl-7-isobenzofluoranthene) and several other compounds and oxidation products in air samples (acetylnaphthalene, 2-nitro-

naphthalene, 9-fluorenone, naphthalene-1,8-dicarboxylic acid anhydride, anthracene-9,10-dione, pyrene carboxaldehyde, benz[a]anthracene-7,12-dione, and pyrene-3,4-dicarboxylic acid anhydride).

## Results and Discussion

### Performance of the 20 l/min Sampler

The performance of the 200 l/min sampler has been described elsewhere (Wilson et al., 1989; Howes et al., 1986; Chuang, Mack et al., 1987). Here we discuss only the 20 l/min sampler. Operation in the laboratory at ambient temperatures from 20 to 27 °C raised the temperature inside the sampler as high as 38 °C, but did not raise the temperature of the sampling inlet and caused no problems. The sampler noise was low and met noise criterion NC 39, which is suitable for an office or conference room -- a sound level similar to that of a quiet fan or the operation of a personal computer. Breakthrough was insignificant over several different sampling periods. Flow rates were stable during continuous operation for up to seven days. Continuous sampling over a 7-day period gave results equivalent to those obtained over seven consecutive 24-hr sampling periods, with the exception of reactive PAH or reaction products such as 9-fluorenone, 9-nitroanthracene, and pyrene carboxaldehyde (Kuhlman and Chuang, 1988).

Recently, we used this sampler inside and outside five homes as part of a methods evaluation for a planned study involving more than 200 homes. These homes are located in Azusa, California, a small city east-northeast of Los Angeles. No heating systems were in use during the study, which took place in late March and early April. Overall operation of the samplers was satisfactory (Chuang et al., 1989). Sufficient material for chemical analysis was collected. The noise levels were entirely acceptable to the occupants of the sampled residences. However, we encountered some problems. Unexplained gaps in

the recorded flow rates, although normal flow calibrations were obtained before, after, and during the experiments, indicated that recording flow controllers should be used instead of flow transducers, despite the higher cost. We had set the thermal cut-off switches inside the samplers at 140 °F; although this was satisfactory in the laboratory and inside residences, it was too low for outdoor operation in southern California. Solution of the above problems should provide a relatively inexpensive, generally useful sampler for indoor SVOCs.

### Target Compound Concentrations

The ranges and typical values of PAH concentrations in all 33 homes that we sampled in California and in Ohio are shown in Tables 1 and 2. Detailed analytical results are being reported elsewhere (Chuang et al., 1991; Wilson et al., 1991, 1992). Statistical analysis (Mack et al., 1989; Menton et al., 1989) showed that the results are consistent from study to study (in terms such as the relation-

Table 1 Indoor concentration ranges of selected PAH<sup>a</sup>, ng/m<sup>3</sup>

Compound	Lowest	Highest
Quinoline	0.29	1100
Phenanthrene	9.2	210
Fluoranthene	2.40	37.37
Pyrene	1.4	18.1
Cyclopenta [c, d]pyrene	0.00	2.38
Benz[a]anthracene	0.0	3.81
Chrysene	0.18	8.61
Benzo[a]fluoranthene	0.0	7.81
Benzo[a]pyrene	0.00	4.13
Indeno [1, 2, 3-c, d]pyrene	0.00	3.53
Benzo[g, h, i] perylene	0.00	5.4
Coronene	0.00	4.75
1-nitropyrene	0.00	0.28
2-nitrofluoranthene	0.006	0.20

<sup>a</sup> Range of concentrations measured in 33 homes in CA and OH, both day and night, winter and summer, for 8- to 24-hr sampling periods.

**Table 2** Typical PAH concentrations in homes<sup>a</sup>, ng/m<sup>3</sup>

Compound	Smoking	Non-smoking	
	Gas heat Gas stove	Electric heat Electric stove	Gas heat Gas stove
Quinoline	240	3.1	2.8
Phenanthrene	87	19	31
Fluoranthene	8.6	2.99	3.7
Pyrene	5.2	2.13	2.8
Cyclopenta[c, d]pyrene	1.1	0.17	0.21
Benz[a]anthracene	1.4	0.20	0.46
Chrysene	2.6	0.37	0.67
Benzo[fluoranthenes	2.5	0.47	0.93
Benzo[a]pyrene	1.2	0.28	0.63
Indeno[1, 2, 3-c, d]pyrene	0.97	0.32	0.82
Benzo[g, h, i]perylene	1.4	0.53	0.88
Coronene	0.60	0.52	1.3
1-Nitropyrene	0.044	0.020	0.021
2-Nitrofluoranthene	0.052	0.012	0.022

<sup>a</sup> Concentrations in selected individual homes having approximately median concentrations for 33 homes, averaged over three locations in the homes: living room, bedroom, and kitchen.

ships of target compounds to each other, compound concentration ranges, relationships of the target compounds to specific pollutant sources, effects of combustion sources, and home characteristics such as air exchange rates) although we sampled different homes and used different samplers and/or sorbents. An exception is that breakthrough of the smaller PAH and other small non-polar compounds occurred with PUF. Thus PUF is inappropriate as a back-up adsorbent for PAH or similar SVOCs.

The indoor concentration ranges of selected PAH are shown in Table 1. These data represent 33 homes, some of which were sampled for three consecutive 8-hr periods on one or two days and some of which were sampled on seven consecutive days. In general, the variability from home to home was quite large, whereas the day-to-day variability in the same home was small. Indoor concentrations were, with rare exceptions, higher than those outdoors, and were associated with indoor combustion sources.

In homes where smoking took place, its effects dominated the results, with typical increases in PAH concentrations over non-smoking homes by a factor of three or more. These increases are clearly evident in the typical concentrations presented in Table 2. Each of the columns in the table gives the average of the individual PAH concentrations over all rooms (kitchen, living room, and bedroom) and sampling periods for a single home of the type indicated at the top of the column. The use of natural gas for heating and cooking also produced a small but statistically significant increase in indoor PAH (significant at the 90% confidence level for seven PAH, one nitro-PAH, and two oxygenated PAH). We are currently investigating this further.

Although the use of wood-burning fireplaces that were open to the room did not appear to have a statistically significant effect on indoor PAH levels, a reanalysis of the data using pattern recognition techniques showed that homes with fireplaces cluster and score strongly in the principal components that represent the more volatile compounds associated with wood combustion (Mitra and Wilson, 1990). A principal component plot showing such clustering is given in Figure 2. Using pattern recognition may allow us to glean additional information from our indoor air measurements. However, further examinations of the effects of fireplace use that include detailed real-time air exchange rate measurements are necessary before we can understand the effects of fireplace use on indoor air pollution.

The indoor air concentrations of PAH and PAH derivatives in the five California homes, where no heating systems were in operation, were similar to those obtained in Columbus, Ohio homes, most of which were studied in the winter heating season. Exceptions were some compounds associated with mobile sources, such as cyclopenta[c,d]pyrene, benzo[g,h,i]perylene and coronene, which were higher both inside and outside

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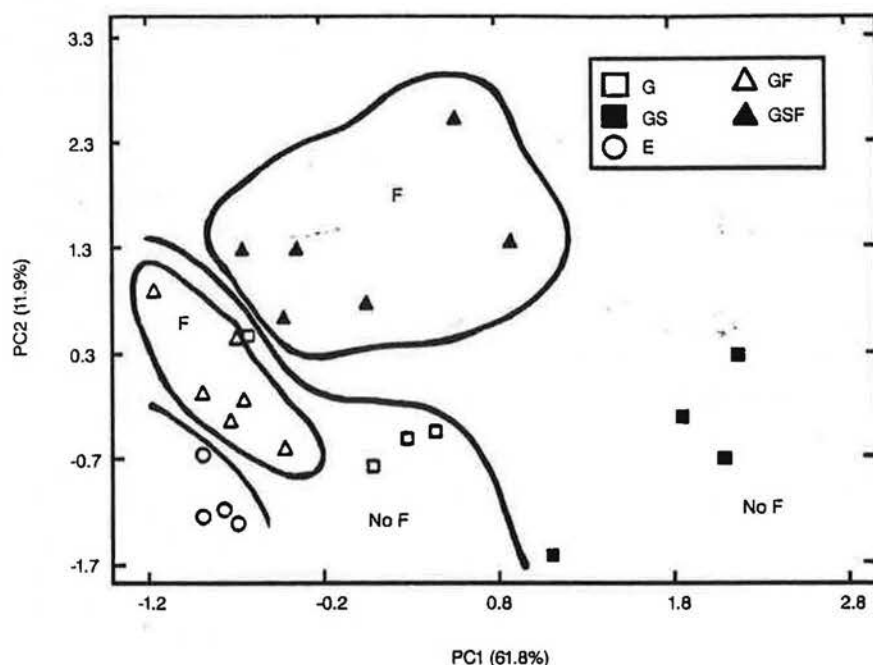


Fig. 2. Principal component scores for fireplace operation, smoking, and heating in ten homes. PC1 (61.8%) is the first principal component, which accounts for 61.8% of the variability in the data, and PC2 (11.9%) is the second principal component, which accounts for 11.9% of the variability. Each PC is a transformed variable made up of the measured concentrations of the target compounds, weighted to maximize the number of concentrations that are high or near zero and to minimize the number that have intermediate values (Jolliffe, 1986). The curved regions shown are not exact, but serve only to aid in visual separation of the scores for different home types. G = homes with gas heating and cooking, GF = homes with gas heating and cooking and fireplace use, GS = homes with gas heating and cooking, and cigarette smoking, GSF = homes with gas heating and cooking, fireplace use, and cigarette smoking, E = all-electric homes with no fireplaces and no smoking (Mitra and Wilson, 1990).

the California homes. Outdoor levels of these three compounds were up to three times higher than those found in the four Ohio studies. In the California homes, indoor and outdoor concentrations were similar; therefore ambient air intrusion appears to be a major influence.

#### Marker Compounds

Quinoline concentrations paralleled those of nicotine in all homes. Both nicotine and quinoline concentrations were highly correlated with the number of cigarettes smoked [significant at the  $< 0.01$  ( $> 99\%$  confidence) level for both compounds]. Since nicotine is a

specific product of the combustion of tobacco and has been used (Löfroth et al., 1989) as a marker for the presence of environmental tobacco smoke (ETS), quinoline may also serve as such a marker. This would simplify both sampling (separate samplers for quinoline and PAH are not required) and analysis (GC/MS dynamic range problems are eliminated) compared to nicotine (Chuang, Kuhlman and Wilson, 1990).

We were interested in finding one or more abundant compounds that could indicate the concentrations of less abundant, but potentially hazardous, PAH and PAH derivatives. The concentrations of three abundant PAH:

phenanthrene, pyrene, and fluoranthene, were highly correlated with concentrations of such compounds as benzo[a]pyrene and benzo[g,h,i]perylene. Pyrene was most suitable, with a mean  $r^2$  of 0.45 for 17 individual PAH measured in 33 homes in three 8-hr or two 12-hr sampling periods per home (Mack et al., 1989; Menton et al., 1989). The correlations for phenanthrene and fluoranthene were lower, with mean  $r^2$  values of 0.35 and 0.41 respectively. Although anthracene, naphthalene, and biphenyl are often abundant in combustion emissions and hence may be expected to be abundant in ambient and indoor air, they are not collected and retained efficiently by our sampling systems, especially at higher sampling temperatures or with PUF sorbent. Thus these lower-mass compounds are less appropriate for use as markers of PAH.

### Conclusions and Recommendations

To sample PAH and related SVOCs in indoor air, a sampler incorporating a quartz fiber filter and XAD-2 or XAD-4 resin is generally useful. For chemical analysis only, a flow rate of 20 l/min provides adequate samples for most compounds in 8-12 hr.

Concentrations of PAH and related compounds in indoor air vary widely, from a few  $\text{pg}/\text{m}^3$  to several hundred  $\text{ng}/\text{m}^3$ . The overall levels are much higher in homes occupied by smokers. Other influences on the PAH levels are more subtle. Use of natural gas heating systems and cooking appliances appears to increase the PAH levels slightly. Spatial variability within a given home was small and not statistically significant. The effects of fireplace operation were not statistically significant, but could be observed using pattern recognition techniques. Further studies are necessary to determine the influences of gas versus electric home heating and cooking, and of other indoor sources. These studies should be done in the absence of smoking.

The abundant compounds fluoranthene, pyrene, and phenanthrene show promise as predictors of the levels of the other target PAH, such as benzo[a]pyrene, which occur at levels that are several orders of magnitude lower. Pyrene is the best marker compound for most of the individual target compounds investigated.

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