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CHAPTER 6

Indoor and Outdoor Concentrations of Organic Compounds Associated with Airborne Particles: Results Using a Novel Solvent System

Datta V. Naik, Charles J. Weschler, and Helen C. Shields

ABSTRACT

An extraction method, using a mixed solvent, has been developed for the identification of semivolatile organic compounds (SVOCs) associated with airborne particles. The procedure yields a single extract, suitable for gas chromatographic analysis, that contains both polar and nonpolar compounds. The solvent is a one-to-one mixture of cyclohexane and 1-chlorobutane. The liquids are miscible and have virtually identical boiling points, which permits refluxing as well as evaporative concentration. The extraction procedure, followed by gas chromatograph/mass spectrometer (GC/MS) analysis, has been used to examine SVOCs associated with size-fractionated airborne particles. The particles were collected indoors and outdoors, for a 6-week period, at a site in Neenah, Wisconsin. The procedure adequately extracts the major organic compounds associated with these particles. The results indicate that there are strong indoor sources for certain branched and *n*-alkanes, fatty acids, and phthalate esters at the Neenah site. It is also apparent that the organic compounds associated with indoor coarse particles have indoor sources. This study reaffirms the earlier observation that partitioning between the vapor phase and the surface of airborne particles is more pronounced indoors than outdoors.

INTRODUCTION

Semivolatile organic compounds (SVOCs), especially those associated with fine airborne particles, can penetrate deep into the lungs and remain in contact with lung tissue for an extended period. Consequently, such compounds are a potential threat to human health. SVOCs can also contribute to failures in sensitive

electronic equipment. Examples of such failures include promoting arcing between contacts of switches and relays leading to increased contact erosion, forming resistive films on precious metal contacts, and contributing to computer head crashes and disc drive failures.

Numerous studies have examined SVOCs associated with outdoor airborne particles (see, for example, References 1 to 8 and references therein). Still other studies have analyzed SVOC associated with indoor airborne particles and contrasted the results with those from outdoor samples.[9-12] In the majority of these studies the organic compounds were extracted from the collected particles using a variety of solvents. Chromatographic procedures were then applied to the resulting extracts. The collection of particles and the chromatographic analysis are usually straightforward. The extraction step, however, can be time consuming and subject to contamination and losses. For example, Daisy and co-workers[5,6] have studied SVOC associated with particles using a sequential three-solvent extraction (solvents of increasing polarity: cyclohexane, dichloromethane, and acetone). Weschler et al. have employed methanol,[9] toluene,[10] petroleum ether,[12] and a thermal desorption method[10,12] to examine SVOC. Multiple solvent extraction is a lengthy process, and each extraction step can lead to loss of analytes, potential contamination, and artifacts. Thermal desorption is relatively fast but gives lower signal-to-noise ratio and consequently poor detection limit. In addition, the use of a low desorption temperature (250 to 300°C) makes it difficult to quantify high molecular weight compounds. Higher desorption temperatures may be used, but that can lead to decomposition of unstable compounds that may be present.

Solvent extraction is still a preferred method for the analysis of SVOC associated with particles as it allows for higher sensitivity. An extraction procedure that yields a single extract is desirable, as this avoids problems associated with multiple solvent extractions. In addition, use of a single extract facilitates subsequent chromatographic analysis.

In this study we report a simple extraction procedure using a mixed solvent system that yields a single extract, containing both polar and nonpolar compounds, suitable for chromatographic analysis. This method, followed by gas chromatograph/mass spectrometer (GC/MS) analysis, has been used to examine SVOCs associated with size fractionated airborne particles collected indoors and outdoors at a site in Neenah, Wisconsin.

EXPERIMENTAL

Sampling

Size fractionated aerosol samples were collected simultaneously indoors and outdoors at a telephone switching office in Neenah during the 6-week period, January 25 to March 8, 1989. Automatic dichotomous samplers (Anderson Sierra Model No. 245) were used to collect coarse particles (2.5 to 15 μm) and fine

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Procedure

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particles (< 2.5 μm) on Teflon membrane filters (37-mm diameter). Prior to their use, the filters were subjected to 6 successive ultrasonic washes (30 min each), first with methanol, then water, then methanol, etc., to remove any contaminants that may be present on virgin filters. The dichotomous sampler was programmed to use a pair of filters (1 coarse, 1 fine) per week. The details of the sampling system are given elsewhere.[13]

Procedure

A solvent system was sought in which a nonpolar solvent would be used to extract nonpolar organic compounds from particles; a polar solvent would then be added to the nonpolar extract to remove remaining polar compounds from the particles. The second solvent has to be miscible with the first, and on mixing should raise the polarity of the mixture enough to extract the polar compounds. The increase in solvent polarity should not be so much as to precipitate the nonpolar compounds already extracted. Additionally, since the extract will then be subjected to evaporative condensation from a few milliliters to a few microliters, this evaporation should not significantly enrich either of the solvent components in the extract.

Cyclohexane and 1-chlorobutane were chosen as the two solvents that meet the criteria mentioned above. Cyclohexane (bp 81°C) is frequently used as a solvent for extraction of nonpolar compounds (e.g., Daisey et al.[5,6]) The boiling point of 1-Chlorobutane (78°C) is close to that of cyclohexane, and the two solvents are miscible. The dielectric constant of 1-chlorobutane (7.4) is similar to that of dichloromethane (8.9) — a solvent commonly used for extraction of polar organic compounds.

The extraction procedure involved sonicating the Teflon membrane filters (six coarse filters or six fine filters), containing particles collected over the 6-week period, in 1.00 ml of cyclohexane for 2 h. This was followed by 12 to 15 h of soaking, and then 1.00 ml of 1-chlorobutane was added. The mixture was again sonicated for 2 h followed by an additional 24 h of soaking. The Teflon filters were then removed and the extract condensed to 10 to 50 μl in a fume hood at room temperature. Aliquots of 1 μl were injected into the GC/MS. Six pre-washed blank filters were also carried through the same extraction procedure and analysis.

Organic compounds contained in the extract were analyzed using a Hewlett Packard 5995C (GC/MS). The separation was achieved with a 12 m × 0.2 mm, cross-linked dimethyl silicone, fused silica capillary column, and a helium flow rate of 1 ml/min in split operation. The GC injection port was kept at 240°C, and the oven was programmed to start at 50°C for 2.6 min, followed by a temperature rise to 300°C at a rate of 8°C/min.

The response of the mass spectrometer was calibrated using an internal standard (d_{10}phenanthrene) and pure analytes (*n*-alkanes, phthalate esters, fatty acids) with selected ion monitoring (SIM) techniques. The ions monitored during a GC/MS run are listed in Table 1. The specific ions used for quantitation of respective

Table 1. Ions Monitored During GC/MS Analysis Using the SIM Technique

m/e Monitored*	Compound(s)
57	*n*-Alkanes with C > 34
73	Fatty acids
85	*n*-Alkanes with C > 34
127	$n\text{-}C_{17,23,33,34}$ and br-$C_{30,32}$
141	Branched alkanes
149	Phthalate esters
160,**188**	d_{10}-Phenanthrene (internal standard)
223	Dibutyl phthalate
256	Palmitic acid
279	Di(2-ethylhexyl)phthalate
284	Stearic acid
293	Dinyl phthalate
307	Didecyl phthalate
338	$n\text{-}C_{24}$
352	$n\text{-}C_{25}$
366	$n\text{-}C_{26}$
380	$n\text{-}C_{27}$
394	$n\text{-}C_{28}$
408	$n\text{-}C_{29}$
422	$n\text{-}C_{30}$
436	$n\text{-}C_{31}$
450	$n\text{-}C_{32}$
464	$n\text{-}C_{33}$
478	$n\text{-}C_{34}$

* The bold m/e peaks were used for the quantitation of the respective compounds.

analytes appear in the table as underscored figures. Samples were quantified by comparison of mass spectral response of selected ion for each analyte to that of the same ion for the respective standard, both normalized to the molecular ion (188) of the internal standard. A few analytes (br-alkanes, *n*-alkanes with C>34, dinonyl and didecyl phthalate) for which standards were not used were estimated by assuming their MS response to be similar to other structurally related standards. Recoveries of standards were determined by spiking a composite of six pre-washed filters with the standards and subjecting these filters to the extraction and analysis. The recoveries were quantitative for $n\text{-}C_{17}$, palmitic acid, and stearic acid. For other standards the recoveries were between 59 and 99%; the appropriate recovery factors were used in the quantitation step. No blank corrections were needed since the GC/MS analysis (SIM mode) of the extract of the six pre-washed blank filters did not detect any of the analytes of interest. All chemicals (solvents and standards) were of HPLC of Gold Label grade, and were obtained from the Aldrich Chemical Co. and Supelco Inc. The solvents were subjected to GC/MS analysis after evaporative condensation of 2.0 ml of the 1:1 mixture (cyclohexane/1-chlorobutane) to a few microliters. No contaminants were detected.

RESULTS AND DISCU

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RESULTS AND DISCUSSION

A 1:1 mixture of the two solvents was found to reflux at 77°C. The solvent composition for 2.00 ml of the 1:1 mixture was studied as a function of evaporation, at room temperature, using a GC. The composition changed gradually with evaporation, progressing towards slight enrichment of cyclohexane. When 2.00 ml of the mixture was evaporated down to 10 ml, the final composition was 54.2% cyclohexane and 45.8% 1-chlorobutane — not a large enough enrichment to cause precipitation of extracted compounds.

A trial analysis was conducted on an aggregate of six 1-week outdoor coarse samples (collected at Neenah) using the above extraction procedure and GC parameters. The mass spectrometer was operated to present a total ion current profile ranging in mass from 40 to 450. The total ion chromatogram of the trial extract is presented in Figure 1. The analytical procedure was found to extract both polar and nonpolar compounds of interest. For this particular sample (coarse airborne particles) n-alkanes as large as C_{43} were detected.

Given the very small amounts of SVOC present (0.02 to 13 ng/m^3), all the GC/MS analyses on real samples were performed using SIM techniques. The SIM method increases the signal-to-noise ratio and improves the detection limit by as much as 1000-fold compared to total ion current techniques.[14]

The average outdoor and indoor concentrations of the major SVOC associated with fine and coarse airborne particles, collected over the 6-week period (January 25 to March 8, 1989), are summarized in Table 2 and Table 3. The average mass concentration of combined fine and coarse airborne particles outdoors was 28,200 ng/m^3, and that indoors was 1290 ng/m^3. During the sampling period the building fans were operated continuously and the air was circulated through filters with an ASHRAE Dust Spot Rating of 85%. The significantly lower concentration of total indoor airborne particles (<5% of the outdoor level), and extremely low concentration of indoor coarse particles (<0.7% of the outdoor level) are attributable to the efficient air filtration at this site coupled with the relatively small number of indoor sources generating airborne particles.

The major nonpolar SVOC detected in the outdoor and indoor samples (Table 2) are the n-alkanes, C_{26} to C_{33}, and the branched alkanes, C_{30} to C_{32}. The combined (fine plus coarse) concentrations for the nonpolar compounds range between 0.3 and 2.4 ng/m^3 in the outdoor samples, and 0.4 and 13 ng/m^3 in the indoor samples. The most abundant (>2.2 ng/m^3) alkanes associated with the outdoor particles are *n*-C_{29} and *n*-C_{31}. Among the nonpolar SVOC detected indoors, the abundant (>2.2 ng/m^3) compounds are *n*-C_{29}, *n*-C_{31}, *n*-C_{33}, br-C_{31}, and br-C_{32} with the concentration of *n*-C_{31} the highest (13 ng/m^3). More will be said about *n*-C_{31} below. As a class the n-alkanes display an odd/even carbon number predominance (Figure 2) in indoor as well as outdoor samples. This suggests that a significant fraction of these compounds are derived from vascular plant waxes.[4,8] Common indoor sources for the *n*-alkanes include sealants, lubricants, and polishes.[15] In addition to the compounds listed in Table 2, the chromatograms also displayed a "hump" starting at

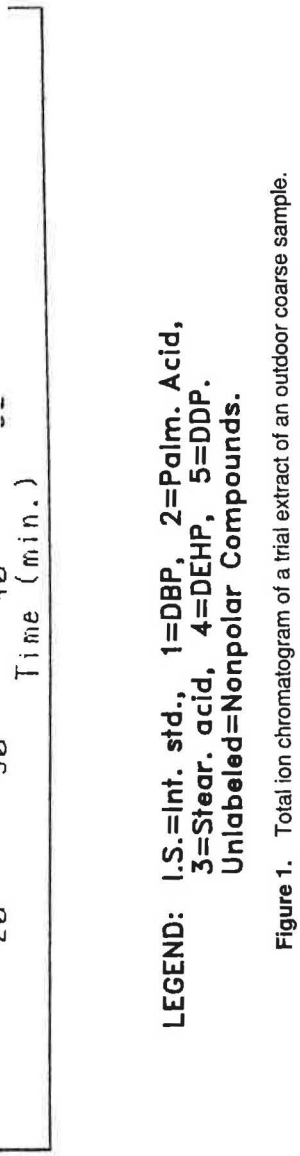

Figure 1. Total ion chromatogram of a trial extract of an outdoor coarse sample.

Table 2. Average Outdoor and Indoor Concentrations for Major Nonpolar Organic Compounds Associated with Airborne Particles at Neenah, Wisconsin

Compound	Outdoor ng/m³		Indoor, ng/m³	
	Fine	Coarse	Fine	Coarse
Airborne particles	14300	13900	1200	90
n-C_{26}	0.21	0.10	0.42	0.01
n-C_{27}	0.39	0.51	1.20	n.d.
n-C_{28}	0.42	0.10	0.40	0.02
n-C_{29}	1.29	0.96	2.53	n.d.
n-C_{30}	0.76	0.96	1.24	n.d.
br-C_{30}	0.9	n.d.	1.9	n.d.
n-C_{31}	1.78	0.62	12.94	n.d.
br-C_{31}	0.7	n.d.	4.0	n.d.
n-C_{32}	0.46	0.14	1.50	n.d.
br-C_{32}	0.6	n.d.	4.9	n.d.
n-C_{33}	1.62	n.d.	4.06	n.d.

Table 3. Average Outdoor and Indoor Concentrations for Major Polar Organic Compounds Associated with Airborne Particles at Neenah, Wisconsin

Compound	Outdoor, ng/m³		Indoor, ng/m³	
	Fine	Coarse	Fine	Coarse
Airborne particles	14300	13900	1200	90
Dibutylphthalate	0.08	0.05	0.04	0.02
Di(2-ethylhexyl)phthalate	1.4	0.35	2.5	0.5
Palmitic acid	10.6	2.8	1.2	1.1
Stearic acid	9.8	1.1	1.9	1.0

about n-C_{26} and extending to n-C_{36}. This broad band is due to the elution of high molecular weight branched and substituted cyclic alkanes with boiling points so close that they could not be separated with the capillary column used in this study. Such a hump is commonly referred to as an unresolved complex mixture (UCM). Other studies[7,16] have reported similar high-boiling UCMs in extracts of particulate phase organic compounds and have ascribed the source to be anthropogenic emissions (primarily fossil fuel combustion). Although the UCM is apparent in the total ion chromatograms (see Figure 1), it does not interfere with SIM analyses due to the selective nature of the latter technique.

n-C_{31} has been detected in high concentration in other indoor environments (e.g., Wichita, KS and Lubbock, TX.[10,12] Its unusually high indoor concentration at each of these sites suggests a strong, yet fairly common, indoor source. A possible candidate is smoking. In chamber studies, Eatough et al. have identified n-C_{31} as one of the major particle-phase compounds found in environmental tobacco smoke (ETS).[17] It is the only linear alkane so identified. Although smok-

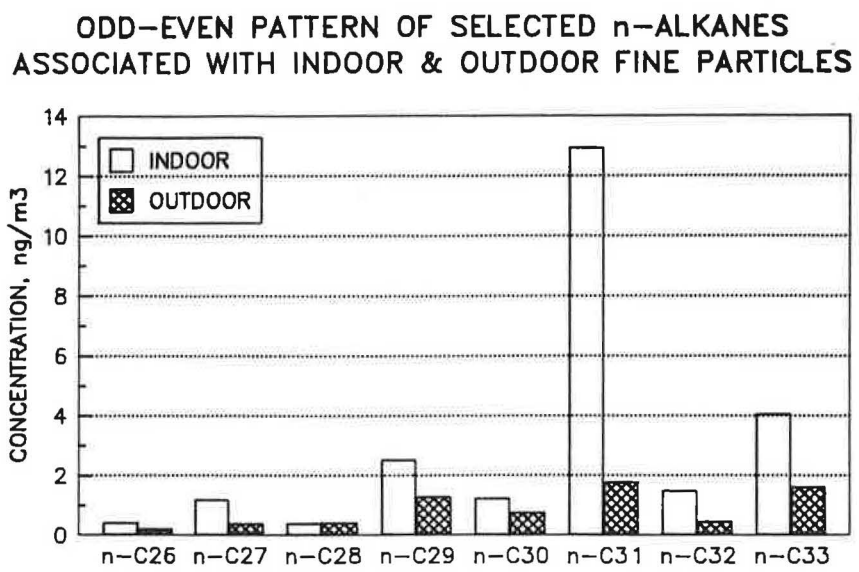

Figure 2. Distribution of selected *n*-alkanes associated with indoor and outdoor fine particles.

ing is prohibited in telephone switching areas, the Neenah office does have a "breakroom" where employees are permitted to smoke. A certain fraction of the air from this room is recirculated throughout the office by the building's air handling system. Fine-mode potassium (K) has also been identified as a potential tracer of ETS.[17] Indeed, in other studies of telephone switching offices (including Wichita and Lubbock), elevated levels of fine-mode potassium have been ascribed to smoking.[18,19] In a separate study, the water-soluble salts associated with fine and coarse particles from Neenah have been measured.[20] The concentration of indoor fine-mode potassium averaged 9 ng/m^3. Hence the ratio, by mass, of the average indoor concentration of n-C_{31} to K at Neenah is (12.9/9) = 1.4. The ratio, by mass, of n-C_{31} to K in Eatough's chamber studies was 1.2.[17] This simple analysis does not take into account the relative contribution of outdoor sources to the indoor concentrations of these species. However, it does show that smoking is a reasonable explanation for elevated indoor levels of both n-C_{31} and K.

The major polar compounds detected in the outdoor and indoor samples (Table 3) are dibutyl phthalate, di(2-ethylhexyl)phthalate (DEHP), and fatty acids (palmitic and stearic acids). Phthalate esters are frequently used as plasticizers,[15] and their association with airborne particles is expected from the widespread use of plastics. Palmitic and stearic acids have many natural sources, including vascular plant waxes,[4,21] and are also present in a variety of commercial products.[15]

The indoor/outdoor ratios (I/O) for selected nonpolar and polar compounds associated with the airborne particles collected over the 6-week period are listed in Table 4. The average I/O values for the mass of the aerosol particles (0.08 for

Table 4. Indoor/Outd

Compound
Airborne Particles
n-C_{27}
n-C_{28}
n-C_{29}
n-C_{30}
br-C_{30}
n-C_{31}
br-C_{31}
n-C_{32}
br-C_{32}
n-C_{33}
Dibutylphthalate
Di(2-ethylhexyl)phthalate
Palmitic acid
Stearic acid

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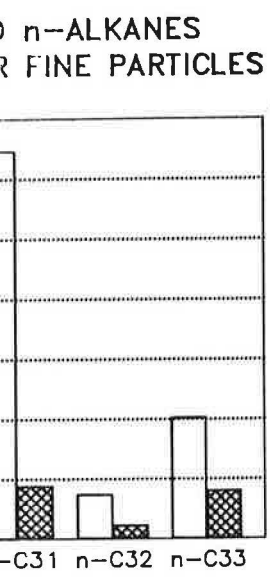

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Table 4. Indoor/Outdoor Ratios for Selected Nonpolar and Polar Compounds

Compound	I/O fine	I/O coarse
Airborne Particles	0.08	0.006
n-C_{27}	3.1	—
n-C_{28}	0.9	0.2
n-C_{29}	2.0	—
n-C_{30}	1.6	—
br-C_{30}	2.1	—
n-C_{31}	7.3	—
br-C_{31}	5.7	—
n-C_{32}	3.3	—
br-C_{32}	8.2	—
n-C_{33}	2.5	—
Dibutylphthalate	0.5	0.4
Di(2-ethylhexyl)phthalate	1.7	1.4
Palmitic acid	0.1	0.4
Stearic acid	0.2	0.9

fine mode and 0.006 for coarse mode) provide a framework in which the I/O relationships for the SVOC can be considered. If the I/O value for a fine-mode SVOC is significantly greater than 0.08 (0.006 in the case of a coarse-mode SVOC), then that compound is assumed to have a strong indoor source.

For most of the coarse-mode alkanes, I/O values could not be calculated since their indoor concentrations were below our detection limit (0.02 ng/m^3). Apparently, indoor sources of these alkanes in the coarse mode are insignificant. For all of the fine-mode alkanes, the I/O values indicate significant indoor sources. The data suggest stronger indoor sources for n-C_{27}, n-C_{31}, n-C_{32}, n-C_{33}, br-C_{31}, and br-C_{32} than for n-C_{28}, n-C_{29}, n-C_{30}, and br-C_{30}. As mentioned above, common indoor sources for the alkanes include waxes, polishes, and lubricants.[15]

Comparisons of the I/O values for the phthalate esters with the corresponding values for the airborne particles indicate strong indoor sources of dibutyl phthalate and DEHP in the fine as well as in the coarse mode. Dibutyl phthalate is frequently used as a plasticizer in floor polishes. DEHP is used to plasticize vinyl products and can be found in everything from floor tiles to vinyl binders. The abrasion of materials such as floor tiles could contribute to the relatively high concentration of these SVOC in the coarse mode. Comparisons of the I/O values for the fatty acids with the corresponding values for the airborne particles suggest that there are also indoor activities which directly introduce course-mode particles containing these compounds. Indoor fatty acid sources are comparatively less important in the fine mode. Potential indoor sources of the fatty acids include detergents, lubricants, waxes, and polishes.[15] Further analyses that may clarify the source(s) of these coarse-mode fatty acids are in progress.

The fine/coarse ratios (F/C) for selected nonpolar and polar compounds are

Polar Compounds

	F/C Indoor
	13.8
	>100
	20
	>200
	>100
	>90
	>1000
	>200
	>100
	≥245
	>300
	2.0
	5.2
	1.1
	1.8

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environment. Elevated concentrations are even more likely when ventilation rates are reduced through energy conservation measures.

The F/C values for the polar compounds range from 1.6 to 8.8 for outdoor samples and from 1.1 to 5.2 for indoor samples. Comparison of these values with the corresponding particulate ratio (F/C) suggest that partitioning of these compounds is relatively unimportant in both outdoor and indoor environments, and that they tend to be integrally associated with the airborne particles.

CONCLUSIONS

A mixed solvent (1:1 cyclohexane/1-chlorobutane) extraction procedure has been developed which adequately extracts polar and nonpolar SVOC associated with airborne particles. The results of the analysis of 6-week samples of coarse and fine particles collected indoor and outdoor at the Neenah site indicate that strong indoor sources exist for n-C_{27}, n-C_{29}, n-C_{31}, n-C_{32}, n-C_{33}, br-C_{30}, br-C_{31}, and br-C_{32}, and for dibutyl phthalate, DEHP, palmitic acid, and stearic acid. This study reaffirms the earlier observation[12] that partitioning between the vapor phase and the surface of airborne particles is more pronounced indoors than outdoors.

ACKNOWLEDGMENTS

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INTRODUCTION

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