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Project Summary

Evaluation of Sampling and Analytical Methods for Nicotine and Polynuclear Aromatic Hydrocarbon in Indoor Air

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The objective of this project was to evaluate a potential collection medium, XAD-4 resin, for collecting nicotine and polynuclear aromatic hydrocarbon (PAH) and to determine whether one collection system and one analytical method will allow quantification of both compound classes in air.

The extraction efficiency study was performed to determine the extraction method to quantitatively remove nicotine and PAH from XAD-4 resin. Various solvents were used with both the solvent-spiking and XAD-4 matrix-spiking methods. The results showed that a two-step Soxhlet extraction consisting of dichloromethane followed by ethyl acetate resulted in the best recoveries for both nicotine and PAH.

In the sampling efficiency study, XAD-2 and XAD-4 resin were compared, in parallel, for collection of PAH and nicotine. Quartz fiber filters were placed upstream of both adsorbents to collect particles. Prior to sampling, both XAD-2 and XAD-4 traps were *spiked with known amounts (2μg) of* perdeuterated PAH and D_3-nicotine. The experiments were performed with cigarette smoking and non-smoking conditions.

The spiked PAH were retained well in both adsorbents after exposure to more than 300 m^3 of indoor air. The spiked XAD-4 resin gave higher recoveries for D_3-nicotine than did the spiked XAD-2 resin. The collection efficiency for PAH for both adsorbents is very similar but higher levels of nicotine were collected on XAD-4 resin.

This Project Summary was developed by EPA's Environmental Monitoring Systems Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Many polynuclear aromatic hydrocarbons (PAH) are mutagens, carcinogens, or both. There is increasing concern over human exposure to these compounds in indoor air from the workplace, home and school.

Several studies have identified environmental tobacco smoke (ETS) as a major contributor to levels of PAH in indoor air pollution. Battelle conducted a pilot study to determine PAH in residential air. The results indicated that cigarette smoking is one of the major contributors to levels of PAH. The study found higher levels of particulate matter and PAH in homes occupied by smokers. Other studies have also indicated that cigarette smoking can substantially increase the mutagenicity of the indoor air.

It appears that nicotine can be an important marker for ETS because it is unique to tobacco smoke and is a major

constituent of the smoke. However, it is not clear whether air sampling and analytical methods typically used for PAH can be applied to nicotine. Different collection media such as treated filters, which require separate sample preparation and analyses, are normally used to determine nicotine in air. It is desirable to determine both compound classes in indoor air with only one collection system and analysis method.

The following study was carried out to evaluate a potential collection medium, XAD-4 resin, for nicotine and PAH and to determine if one collection system and analytical method can be used to determine both compound classes. This study consisted of the following subtasks:

1. Extraction efficiency studies to determine the optimum procedure for determination of nicotine and PAH.

2. A sampling efficiency study under cigarette smoking and non-smoking conditions, using a filter and either XAD-4 or XAD-2.

3. Evaluation of different analytical methods to measure nicotine and PAH to determine if nicotine and PAH can be measured within one analysis.

Procedure

Different solvents or combinations of solvents were evaluated in the extraction efficiency study for XAD-4 resin. Prior to extraction, an aliquot of the spiking solution which contains selected PAH and nicotine was spiked into either the solvent reservoir (solvent spike) or the clean XAD-4 resin (matrix spike). The extraction was carried out for 16 hr. Prior to concentration, the extracts were filtered through clean pre-wet quartz fiber filters. The extract was concentrated to about 1 ml and was analyzed by gas chromatography/mass spectrometry (GC/MS).

Two prototype indoor air samplers were placed parallel to each other and were separated from each other by about 1 ft. Quartz fiber filter and XAD-2 resin in series was used in one sampler, and quartz fiber filter and XAD-4 resin in series was used in the other sampler. Prior to sampling, an aliquot of the perdeuterated solution was spiked onto both XAD-2 and XAD-4 cartridges. Then the air was sampled at approximately 8 cfm flow rate for about 24 hr.

The samplers were exposed to cigarette smoke during the sampling period at uneven intervals for the first four tests. In the fifth test, the samplers were not exposed to cigarette smoke. After sampling, XAD-2 and XAD-4 samplers were Soxhlet extracted by dichloromethane followed by ethyl acetate and were concentrated to about 1 ml for GC/MS analysis.

Results

A cleanup method for XAD-4 resin was developed including Soxhlet extraction with methanol, ethyl acetate and dichloromethane sequentially. The results of the extraction solvent spike demonstrated that good recoveries were obtained for the spiked PAH and nicotine from various solvents tested including dichloromethane, ethyl acetate, and 0.01% trimethylamine in ethyl acetate. This finding indicated that the losses of target compounds through sample handling and concentration are negligible. We also noted that the precipitates were formed during the concentration of 0.01% trimethylamine in dichloromethane. This observation suggested that ammonium salts were formed between trimethylamine and dichloromethane during concentration. Thus this solvent system was excluded from the matrix spike experiment.

The results of the extraction matrix spike indicated that recoveries of PAH and XAD-4 were generally good, but not for nicotine (27%) when dichloromethane was used as the extracting solvent. The recovery of nicotine improved to greater than 90 percent with ethyl acetate. But the recoveries of some PAH such as acenaphthylene and anthracene were only from 70 to 60 percent with this solvent. The recoveries of these PAH did not improve when basic ethyl acetate was used. Thus two-step extractions were evaluated which consisted of dichloromethane followed by either 0.01% pyridine in ethyl acetate or 100% ethyl acetate. Overall, good recoveries for both PAH and nicotine were obtained from these two-step extractions.

The results of the sampling efficiency study revealed that most PAH levels found in the XAD-2 and the XAD-4 samples are indistinguishable from each other. This finding suggested that both XAD-2 and XAD-4 resin have similar sampling efficiency for PAH. But levels of nicotine found in XAD-4 samples were higher than those in the corresponding XAD-2 samples indicating that XAD-4 resin has better sampling efficiency for nicotine.

Both electron impact (EI) and positive chemical ionization (PCI) GC/MS methods were employed to analyze the standard solution containing PAH and nicotine. As expected, the PCI method provides better detection sensitivity for nicotine than the EI method. The detection for PAH with both methods is very similar. Fused silica capillary columns with two film thickness (0.17 μm and 0.50 μm) were evaluated. Better detection sensitivity for nicotine was detected with the thinner film column (0.17 μm).

Conclusions and Recommendations

The following conclusions are based on the results of this study:

1. The collection system, a quartz fiber filter and XAD-4 resin in series, can be used to collect both PAH and nicotine in indoor air.

2. The extraction method, Soxhlet extraction with dichloromethane followed by ethyl acetate can quantitatively remove both PAH and nicotine from XAD-4 resin.

3. The analysis method, positive chemical ionization, gas chromatography/mass spectrometry operated in the multiple ion mode, can be used to determine both PAH and nicotine.

Because XAD-4 resin has better sampling efficiency for nicotine than does XAD-2 resin and both adsorbents show comparable sampling efficiency for PAH, quartz fiber filters and XAD-4 resin are recommended for future field studies. The recommended sample preparation procedure is Soxhlet extraction with dichloromethane followed by ethyl acetate. The positive chemical ionization gas chromatography/mass spectrometry (PCI GC/MS) method operated in the multiple ion detection (MID) mode is also recommended for use in future studies to determine both PAH and nicotine.

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The complete report, entitled "Evaluation of Sampling and Analytical Methods for Nicotine and Polynuclear Aromatic Hydrocarbon in Indoor Air," (Order No. PB 88-124 615/AS; Cost: $12.95; subject to change) will be available only from:
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