Bull. Environ. Contam. Toxicol. (2002) 68:485–489

DOI: 10.1007/s00128-001-0280-y

Bulletin of Environmental Contamination and Toxicology

Trace Analysis of Nicotine in Indoor Air by a SPME Method

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Received: 14 May 2001/Accepted: 1 December 2001

One of the major public health concerns in recent years has been indoor air pollution and its effect on the individuals (Thompson, et al, 1989). Environmental Tobacco Smoke (ETS), which is a complex mixture of gases and particles, has been recognized as an important component of indoor air pollution due to their serious health implications. Nicotine is often used as a marker for ETS because it is unique to tobacco and is a major constituent of tobacco smoke (Chuang, et al, 1990). It is important to determinate the nicotine concentration in indoor environment in order to know the indoor air quality, so the need exists to develop a simple, rapid and accurate method for analyzing nicotine. Various testing methods have been used for the determination of nicotine in indoor air, but they are all either complex, time-consuming, or limited by their relatively high limits of detection. A recent advance in sampling concentration and preparation technique is solid-phase microextraction (SPME), which is solvent-free, simple, rapid, portable and has a number of other advantages over conventional methods (Namiesnik, et al, 2000). In the SPME method ,the analytes are absorbed directly from an aqueous or gaseous phase onto a thin fused-silica fiber coated with a polymeric phase, hence, sampling, extraction and concentration are incorporated into a single step (Namiesnik, et al, 2000). The fiber is then inserted into the injector port of a gas chromatography, so the analytes on the fiber are desorbed in the GC injector and the analysis of the sample is carried out. Due to the above attracting features, SPME has become very popular since it advents, especially in environmental analysis. However, this new method, to our knowledge, has seldom been used to determinate the concentration of nicotine in indoor air.

In this work, we used the SPME method to determinate the quality of indoor air with respect to the concentration of nicotine. Analytical determinations were carried out using a gas chromatograph coupled with a mass spectrometer.

MATERIALS AND METHODS

A manual SPME holder was used with a 100 μm polydimethylsiloxane fiber (Supelco, Inc). GC/MS (Finnigan MAT GCQ). A pump for sampling (self-made).

A number of pilot experiments were completed to confirm the suitability of the

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sampling and analytical procedures. These included the estimation of the fiber exposure time required for the sampling and the determination of the appropriate analytical conditions of GC/MS. In the main study, two methods of gaseous nicotine sampling were applied. One was a low-volume active sampling method in which a small pump drew air at 2ml/min directly from the indoor environment and the fiber of the SPME appliance was put in the gas path. The second method was a passive sampling method, in which the nicotine was collected on the SPME fiber solely by molecular diffusion without the help of the pump. Full details of the two methods were presented in Table 1. Samples were collected at two sites which are well away from known or suspected pollution sources. During the sampling, the door and the windows were closed. The SPME holder was placed centrally in the investigated rooms at a height corresponding to the breathing zone of smokers (1-1.5m) above the floor. Before sampling, the extraction fibers were conditioned at 250℃ in the GC injection port for 30 mins, then fiber blank analysis was conducted on the GC/MS.

Table 1. Details of sampling procedure

Sampling method	Sampling principle	Sampling site 1	Sampling site 2	Temperature
Active sampling	Pump, dynamic	40 m^2 office, 4 workers, two of whom are smokers	32 m^2 lab, 4 workers, no smokers.	12℃
Passive sampling	Molecular diffusion			

Table 2. Details of analysis conditions for GC/MS

Injection made	Injection temperature	Carrier gas	Temperature program	Desorption time	MS method
Splitless	220℃	N_2 at 40cm/s	50°C hold 5 mins, then 10°C/min to 100°C, 4°C/min to 250°C, hold 10 mins.	40s	SIM

Once the sample had been collected on the fiber of SPME, analysis was conducted by GC/MS. Details of analysis conditions are shown in Table 2. The experiments were made in duplicate. Identification of the nicotine was based on comparison of its mass spectrum with that available in the mass spectra library of GC/MS, (NIST in this study). Quantification was done according to the experiments of Martos and Pawliszyn, in which the unknown concentrations of target analyte were determined by measuring the analyte mass loaded on the fiber and the sampling temperature.

RESULTS AND DISCUSSION

Several factors have been found to affect the precision of the SPME technique for

GC/MS analysis. These include the location of the fiber in the injection port, the desorption temperature, the desorption time, and the column temperature.

The top of the depth gauge is adjusted to #4 on the holder so that the fiber is in the suitable place. In this position, the largest detector response of Finnigan MAT GC/MS instrument was obtained. Once the above factor has been established, the optimum desorption temperature and time were determined by monitoring the peak area as a function of both time and temperature. The desorption temperature and time were set at 280°C, 220°C, 150°C and 5mins, 2mins, 40s respectively. We found that as the temperature rises, the desorption becomes faster and quantitative, but the fiber layer bleed becomes serious. However, when the temperature is too low, the analyte can not be desorbed from the fiber completely. The determination of an appropriate desorption time is also important because the analyte should be desorbed from the fiber completely to reach the highest possible sensitivity and to avoid carryover. But if the desorption time becomes too long, the chromatographic band will become too wide. In this study, desorption for 40s at 220°C was found to be optimal. No analyte peak was discovered when analyzing the fiber blank directly after the previous injection, which indicates that thermally desorption of the fiber for 40s at 220°C is enough to desorb nicotine completely. At the same time, no bleed peaks of the fiber layer was found.

The procedure outlined above obtained a well-separated nicotine peak. Analytical results of the nicotine in indoor air are shown in Table 3, with representative chromatogram shown in Figure1.The amounts measured accord well with previously published data for similar sampling conditions (Rothberg, et al, 1998). In the passive sampling mode, sampling time 1h, 2h, 4h were selected respectively, the determined nicotine concentration increases when the sampling time increases, but the concentration determined at 4h were lower than that at 2h, which indicates that 2h is long enough to sample nicotine in indoor air by SPME. The lower concentration at 4h may be due to the desorption of nicotine again from the fiber during sampling. The control experiment involved deployment of passive gas sampler for a 4h period in a non-smoker lab, no nicotine peak was found. Only one result is presented under active sampling condition because it seems that no significant difference can be found between active and passive sampling by SPME.

Precision was determined by analyzing duplicate for each experiment, which were shown in the Table 3. The RSD values varied from 3% to 5%, which are normal for trace analysis. The limits of detection and quantification were determined according to published guidelines (Thompson, et al, 1989). These are determined as a peak area 3 and 10 times the baseline peak-to-peak noise level near the nicotine peak, respectively. According to these criteria, under the analytical conditions described above, the limit of detection(LOD) was equivalent to 0.019 μ g/m^3 nicotine, and the limit of quantification(LOQ) was 0.075 μ g/m^3. This level is lower than that of other methods for determination of nicotine in indoor air, such as NIOSH method(30 μ g/m^{3}LOD); trap-thermal desorption method (0.07 μ g/m^3 LOD and 0.17 μ g/m^3 LOQ) (Thompson, et al, 1989), which confirms that the method presented here has adequate sensitivity to cope

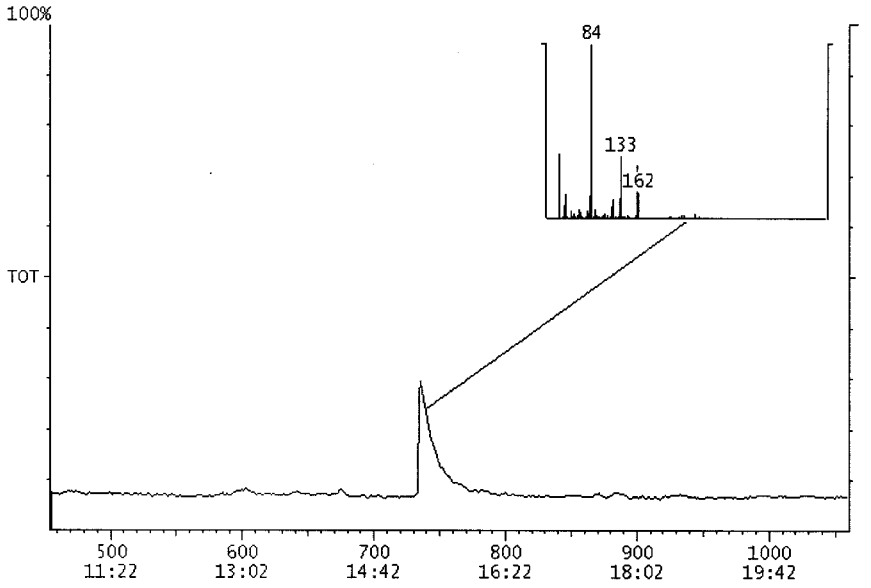

Figure 1. Chromatogram of nicotine under 2h passive sampling.

Table 3. Analytical results of nicotine in indoor environments.

Location	No.of sample	Sampling mode	Sampling time	Nicotine concentration ($\mu g/m^3$)	RSD
Office	1	Passive sampling	1h	0.118	5.02%
Office	2	Passive sampling	2h	0.219	3.11%
Office	3	Passive sampling	4h	0.201	4.33%
Office	4	Active sampling	4h	0.199	3.89%

with both active and passive sampling of nicotine under indoor environments.

Nicotine exists both in the gas and particulate phase, but >95% of environmental nicotine is in the gas phase (Caka, et al, 1990), so nicotine in gas phase sampled by this SPME method can represent the concentration of nicotine in indoor air.

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