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A Convenient Method for the Determination of Ambient Nicotine*

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An analytical method was developed for the determination of airborne nicotine. The method utilizes the impingement of air into distilled water and the direct analysis of the aqueous solution by gas chromatography. This method represents an improvement in sensitivity, reproducibility, time per analysis and convenience over methods found in the literature. The impinger/GC method was found to be useful in determining nicotine concentrations in both low and high nicotine environments. Direct injection of the sample into the gas chromatograph equipped with a glass column permits the analysis of nicotine in the sample at levels as low as 1.0 µg/ml for a 3 µl injection. This corresponds, given a sampling rate of two liters per minute and a sampling time of one hour, to a lower limit of detection of ambient nicotine of 40 µg/m³. The current U.S. Occupational Safety and Health Administration (OSHA) maximum allowable 8-hour average exposure is 500 µg/m³.

INTRODUCTION

Numerous analytical methods have been published for the analysis of airborne nicotine, but they have not provided us with the specificity, simplicity or the concentration range we were seeking (1 — 6). A recent method published by the U.S. National Institute of Occupational Safety & Health (NIOSH), No. S293, was investigated and found to be inadequate for our purposes because the method was restricted to low nicotine environments, lacked reproducibility and required specialized instrumentation. We wish to report the development of a new sampling and gas chromatographic method for the determination of ambient nicotine.

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EXPERIMENTAL

Sampling

The sample collection device used in our studies is illustrated in Figure 1. The unit consists of two microimpinger tubes connected in series with tube B acting as a trap for tube A. Previous studies using a third impinger tube indicate that any residual nicotine from tube A is quantitatively captured by tube B thus eliminating the need for more than two traps in the system. An example of a micro-

Figure 1. Ambient nicotine sampling device.

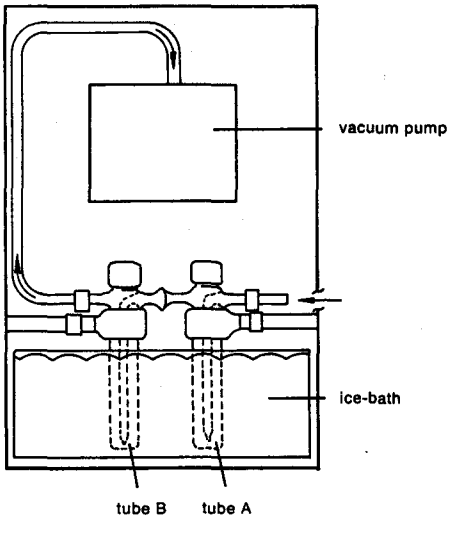
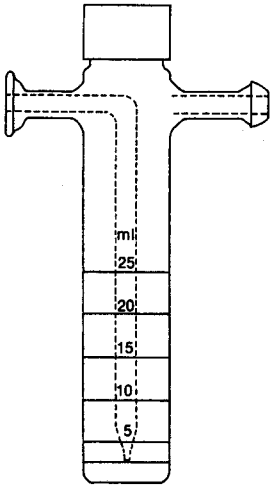


Figure 2. Diagram of a microimpinger tube.



impinger tube is given in Figure 2. A calibrated variable speed battery-powered pump is connected to the secondary impinger tube B. Five milliliters of distilled water is pipetted into each tube. The pump draws sample air through the collection system at a predetermined rate. The impinger tubes are immersed in an ice-water bath to increase nicotine solubility and decrease solvent volatility. The proper operating procedure involves keeping the impinger tubes chilled and maintaining a constant air flow rate. The sampler can operate for as little as one minute or for a maximum time of two hours. After two hours minor fluctuations can occur in the sampler flow rate which can be traced to the capacity of the pump batteries. The optimal flow rate range is from 0.54—2.5 l/min. Flow rates exceeding 2.5 l/min cause excessive frothing of the impinger solvent. A 25-minute trial run is used to estimate the amount of nicotine in the sample air. The optimum sampling time and flow rate can be determined from these preliminary studies. After sampling, the contents of the two impinger tubes are transferred to two 5 ml volumetric flasks. Occasionally in a humid atmosphere, the final volume of each impinger tube might exceed 5 ml. In this case, the contents of the impinger tubes are transferred to appropriately larger volumetric flasks, and the volume is adjusted. The final flow rate is recorded, and the average flow rate calculated. The samples are then analyzed directly by gas chromatography to calculate the concentration of nicotine.

An experiment was performed to determine the amount of nicotine that migrates from tube A to tube B under chilled and non-chilled conditions during a normal sampling run. Five milliliters of a 1 mg/ml nicotine solution was placed in tube A and five milliliters of distilled water was placed in tube B. Nicotine-free air was pulled through the system at a flow rate of 1.9 liters/minute for 30 minutes, 60 minutes and 130 minutes, respectively. Table 1 shows the advantage of using the ice-water cooled impinger system. After all three time increments, no migration of nicotine occurred from A to B within experimental error. Under ambient conditions, tube A showed no migration after 30 minutes, however after 60 minutes, 0.02% of the nicotine in tube A had migrated to tube B and after 130 minutes 0.5% had migrated. Final nicotine concentrations shown in Table 1 were higher under non-chilled conditions due to evaporation and subsequent contractions of the solution in tube A. Under chilled conditions, this concentration effect was less pronounced.

Our studies have revealed the capture efficiency of the sampling system to be 96%—100%. An experiment was performed to determine the net capture efficiency of the ice-water cooled sampling system. This was determined by drawing air samples which had been enriched in nicotine. A nicotine atmosphere was generated by placing a known amount of nicotine in a vaporization bulb. A known volume of dry air was passed through the generator which was connected to the chilled impinger tubes. The nicotine capture was measured by weighing the vaporization bulb at frequent intervals on an analytical balance and determining the net loss. The contents of each impinger tube were analyzed for nicotine and the net capture was calculated. In each case, no migration of nicotine from A to B was observed. The data are given in Table 2.

Table 1. Effectiveness of the cooling bath in limiting solvent evaporation and nicotine loss.

Impinger tube**		Nicotine added (mg/ml)	Ice-bath	Sampling time	Nicotine recovered (mg/ml)
1	A	1.00	yes	30 min	1.07
	B	0			b.l.d.*
2	A	1.00	no	30 min	1.21
	B	0			b.l.d.
3	A	1.00	yes	60 min	1.05
	B	0			0.0002
4	A	1.00	no	60 min	1.20
	B	0			0.002
5	A	1.00	yes	130 min	0.95
	B	0			0.005
6	A	1.00	no	130 min	1.44
	B	0			0.006

* Below limits of detection, i.e. less than 0.1 μg/ml.
** Pump rate 1.9 l/m.

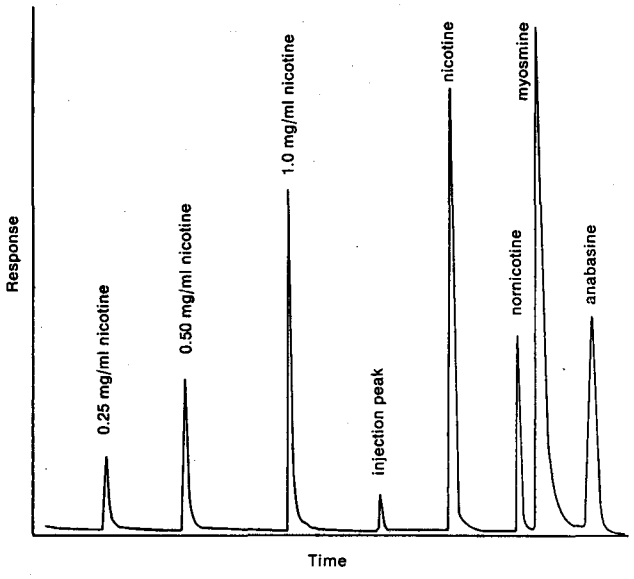
Table 2. Nicotine capture efficiency of impinger system at pump flow of 2 liters/minute.

Sampling time	Amount vaporized	Amount in A (average)	Capture (%)
15 min	140 μg	135 μg	96
30 min	250 μg	245 μg	98
60 min	740 μg	729 μg	99
120 min	1 mg	960 μg	96

Gas Chromatographic Analysis

Nicotine is satisfactorily chromatographed with a Hewlett-Packard 5750 equipped with flame ionization detector. The following conditions and materials were used: a 58-inch × 3 mm outside diameter glass column packed with 5% PPE (polyphenylether, 6-ring) on 110/120 mesh

Figure 3. Nicotine standards and samples.



Anakrom ABS. The oven temperature, injection port temperature and detector temperature are 190 °C, 210 °C and 285 °C, respectively. The helium carrier gas flow is 45 ml/minute at 40 p.s.i. This GC column was found to be stable to aqueous nicotine solutions for a period of two years during routine sample analysis. The polyphenylether column was selected after an extensive search for the best column adapted to aqueous injections of nicotine solutions.

The standards are prepared from freshly vacuum-distilled Eastman practical grade nicotine. The purified nicotine is diluted with distilled water to provide a concentration range of 10—500 µg/ml for air quality samples. The lowest detectable concentration was found to be one µg/ml in a 3 µl injection. The retention time of nicotine is 0.44 minutes while the retention times of nornicotine, myosmine and anabasine are 0.86, 0.98 and 1.09 minutes, respectively. We found that the minor airborne alkaloid components did not interfere with the gas chromatographic analysis. We did not investigate other interfering substances because, from the chromatograms, these do not appear to be a significant problem. The standard curves are linear over the range 10—550 µg/ml with a relative standard deviation of 9.5% for 10—50 µg/ml based on 30 replicates and 4.7% for 100—550 µg/ml based on 20 replicates. An example of a typical nicotine analysis is illustrated in Figure 3.

Table 3. Nicotine profile of a cigarette manufacturing plant.

Site	Volume of air sampled (m^3)	Concentration (µg/m^3)
Blending department (one meter from dryer)	0.1	150
Cutting department (between tobacco separator)	0.09	340
Reprocessed leaf department (exit end of dryer)	0.35	51
Tobacco dryer exhaust stack	0.05	600

EXPERIMENTAL RESULTS

The results listed in Table 3 represent the concentrations of nicotine determined by this sampling procedure at various locations in a cigarette manufacturing plant.

SUMMARY

The impinger tube/gas chromatograph sampling technique has distinct advantages in specificity, simplicity and accuracy over the older literature methods for airborne nicotine sampling and analysis. The impinger/GC method was found to be useful in determining nicotine concentrations in both low and high nicotine environments.

ZUSAMMENFASSUNG

Bei der Bestimmung des Nikotingehaltes der Umgebungsluft erwies sich ein Verfahren, bei dem die Luft durch zwei mit destilliertem Wasser gefüllte Fallensysteme geleitet und die wäßrige Lösung anschließend gaschromatographisch analysiert wird, hinsichtlich Spezifität, Einfachheit und Genauigkeit als den bisher in der Literatur beschriebenen Verfahren deutlich überlegen. Es eignet sich für Luftproben mit niedrigem und mit hohem Nikotingehalt.

RÉSUMÉ

La technique d'échantillonnage utilisant deux pièges à eau, suivie de chromatographie en phase gazeuse de la solution aqueuse, présente des avantages certains sur les méthodes publiées précédemment pour le piégeage et la détermination de la nicotine dans l'air ambiant, particulièrement en ce qui concerne la spécificité, la simplicité et l'exactitude de la méthode. Cette méthode est également applicable à des concentrations fortes et faibles de nicotine dans l'air.

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