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A Method for Sampling Halothane and Enflurane Present in Trace Amounts in Ambient Air

Anton G. Burm, M.Sc.,* and Johan Spierdijk, M.D., Ph.D.†

A method for the sampling of small amounts of halothane and enflurane in ambient air is described. Sampling is performed by drawing air through a sampling tube packed with Porapak Q,® which absorbs the anesthetic agent. The amount absorbed is determined by gas chromatography after thermal desorption. This method can be used for "spot" or personal sampling or for determining mean whole-room concentrations over relatively long periods (several hours). (Key words: Anesthetics, volatile: halothane; enflurane; trace concentrations. Measurement techniques: chromatography.)

Because of the possible risks of chronic exposure to anesthetic gases and vapors, regulations to protect operating room personnel have been established in several countries. It is recommended that the exposure to nitrous oxide be decreased to at most 30 ppm (vol/vol) and halothane to 2 ppm (vol/vol) as a time-weighted average.[1] To determine whether such strict criteria are met in practice requires straightforward and reliable methods for measurements. Analytic devices used include an infrared gas analyzer, a leak meter, a mass spectrometer, or most often, a gas chromatograph. The main problem with gas chromatography is not with analysis but with the method for sampling of air. The concentration of an anesthetic agent in an operating room often fluctuates appreciably around a certain mean value which is dependent on the amount of escaping anesthetic gas or vapor, the ventilation in the room, the air-current pattern, and the place in the room where samples are obtained.[2] The mean concentrations are not always constant at a given location either. If the concentrations fluctuate widely and sampling is performed rapidly, as is usually the case for the commonly used technique of syringe sampling, a large number of samples must be taken and analyzed to obtain a reliable picture of the actual exposure of personnel.

Therefore, we developed a time-saving method to determine average concentrations of volatile anesthetics over a longer period (as long as several hours). For this purpose, a pump was used to draw air through a sampling tube partially filled with Porapak Q®,‡ which absorbs organic anesthetic agents such as halothane and enflurane.

Methods and Materials

Porapak Q (50–80 mesh) was chosen as packing material for the sampling tubes because anesthetic agents such as halothane and enflurane are strongly absorbed by it at room temperature and desorption can be performed at reasonable temperatures (*e.g.*, 160 C). Before being introduced into the sampling tubes the Porapak Q was packed in a stainless steel tube 1 m long (internal diameter: 4.3 mm) and heated at 230 C for 20 hours while helium was passed through the tube at a rate of 50 ml/min.

Sampling tubes were prepared as follows: segments 7.5 cm long were cut from a piece of stainless steel tubing (internal diameter: 4.3 mm; external diameter: 6.35 mm). These segments were packed with 200-mg pretreated Porapak Q, which was held in place with plugs of silanized glass wool and small perforated screws (fig. 1). For sampling, the tubes were placed in a holder. After the sample had been collected, the tubes were sealed with brass Swagelok®§ fittings and vespel ferrules and stored until the analysis could be performed. Vespel ferrules were used because they can be removed from the tube easily after the analysis and reused. The sampling tubes were discarded after 10 to 20 analyses.

When air containing an organic anesthetic agent is drawn continuously through a sampling tube the anesthetic agent is retarded according to its affinity for the Porapak Q packing material. The affinity determines the maximum volume of air that can be drawn through the tube before noticeable amounts of anesthetic appear in the air passed through the tube, and therefore determines the maximum sampling volume (breakthrough volume). Because the concentration in the outflowing air increases slowly

* Research Chemist.
† Professor and Chairman of Anesthesia.
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Address reprint requests to Mr. Burm.

‡ Waters Associates, Inc., Maple Street, Milford, Massachusetts 01757.
§ Crawford Fitting Co., 29500 Solon Road, Solon, Ohio 44139.

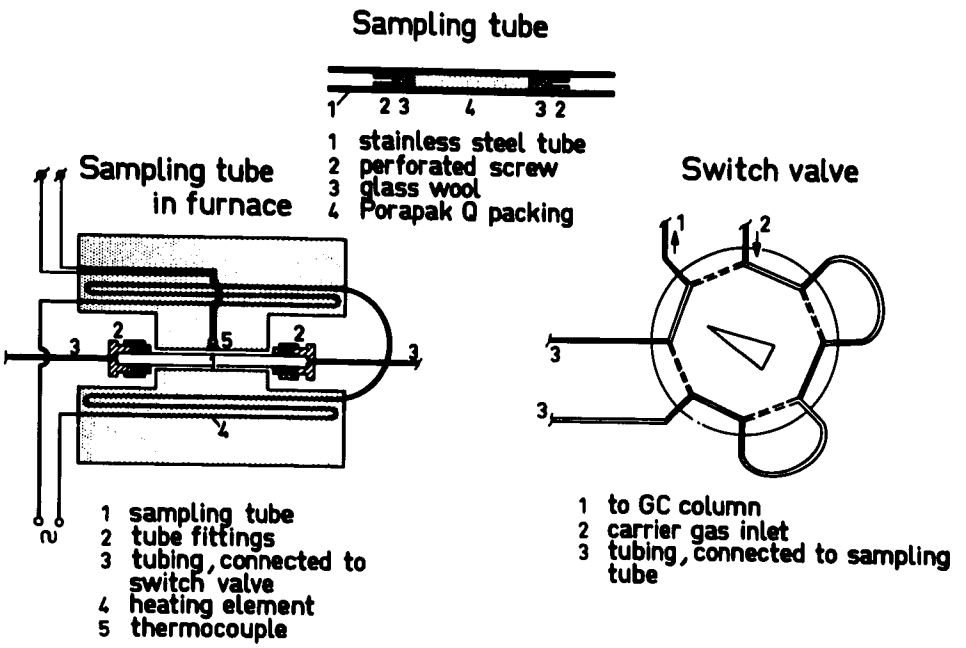

FIG. 1. Sampling tube, furnace and switch valve arrangement.

at low flow rates there is actually no sharp breakthrough point. For practical purpose the breakthrough point was defined as that moment when the concentration of anaesthetic at the outlet of the sampling tube equalled 1 per cent of the concentration at the inlet of the tube. Up to that moment the anesthetic agent is trapped nearly quantitatively (more than 99.9 per cent).

Breakthrough points for the sampling tubes were determined by leading air containing halothane or enflurane through the tubes and analyzing the air leaving the tubes by gas chromatography. Mixtures of air and anesthetic agent were drawn from a cylinder or were prepared using a diffusion cell.[3] The sampling tubes were held in a temperature-controlled water bath. The breakthrough time and the amount of air that had passed through the tube until that time (breakthrough volume) were recorded.

For sampling operating room air a battery-powered Sipin SP-2 air-sampling pump,[¶] from which the stroke counter had been removed, was used. The flow rate can be varied from about 5 ml/min to 100 ml/min. The pump weighs about 320 g and can be carried conveniently in a pocket or worn on a belt. The flow rate applied for sampling is chosen on the basis of the desired duration of the sampling period, which can vary from a few minutes to a few hours, and the desired sample volume. The sample volume was chosen such that a total of 0.5 to 50 μg halothane or enflurane was absorbed, of course taking into account the breakthrough volume. Typical sampling conditions used in our department were a sampling time of one hour and an air-flow rate of 15 ml/min. Before and after each sample was taken, the flow rate was measured using a soap-bubble meter.

A home-made oven was used for the desorption of the absorbed pollutants. The sampling tubes were connected by Swagelok fittings and stainless-steel tubes to an eight-port switch valve (fig. 1). Normally a valve with four ports will suffice. The sampling tubes were warmed in the oven for 2½ min, after which the valve was switched and the desorbed anesthetic agents were carried through the gas chromatographic column.

A Hewlett-Packard 5750 gas chromatograph equipped with a flame ionization detector was used for the analysis. The separation of anesthetic agents and any other impurities takes place in a stainless steel column 120 cm long (internal diameter: 2.1 mm) packed with Chromosorb 102® (80–100 mesh). The column temperature and the carrier gas (helium) flow rate were 115 C and 62 ml/min, respectively.

The gas chromatograph was calibrated with sampling tubes loaded with known quantities of the anesthetic agents.

Results

Breakthrough volume was mainly dependent upon the temperature in the sampling tube (table 1). At higher concentrations the breakthrough volumes will be smaller because of the increasing nonlinearity of the absorption isotherms.

[¶] Anatole J. Sipin Company, Inc., 425 Park Avenue South, New York, New York 10016.

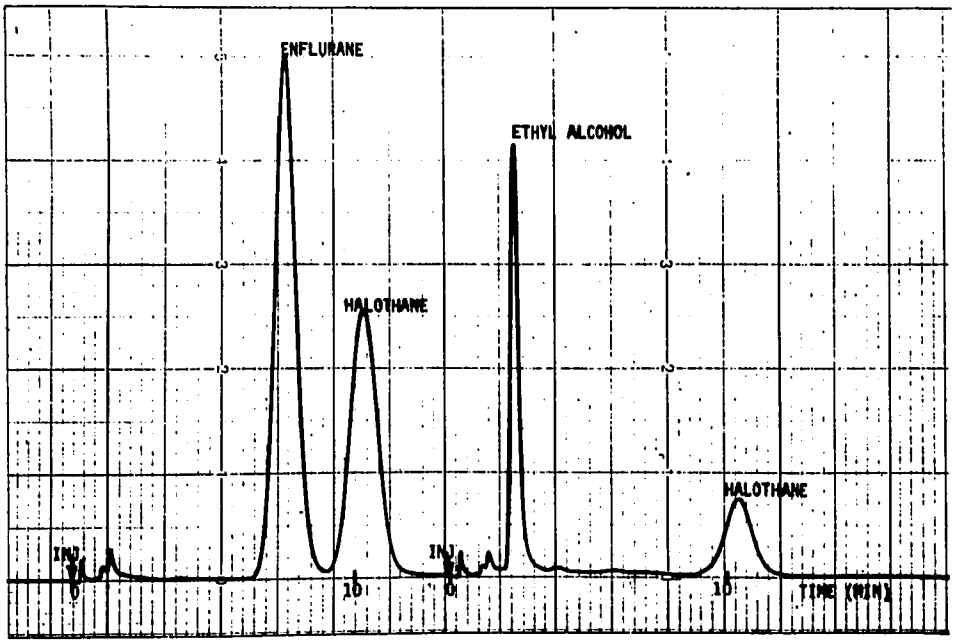

FIG. 2. Chromatograms, obtained from a calibration tube (*left*) and an air sample from an operating room (*right*). The total amount of halothane in the operating room air sample was 2.1 μg.

The optimal desorption temperature proved to be 160 C. At lower temperatures, desorption is too slow, and higher temperatures lead, at high sensitivities, to erroneous peaks in the chromatograms, which are probably attributable to degradation products of Porapak Q. A representative chromatogram obtained for air from an operating room is shown in figure 2. The retention times of halothane and enflurane were 7.5 and 10.3 min, respectively. The coefficient of variation of the peak height in a series of 12 determinations was about 3 per cent for either agent. In the range investigated (amounts of anesthetic agent injected: 1–70 μg) the relationship between amount of the agent and peak height was linear. Injection of larger amounts gave rise to asymmetrical peaks caused by column overloading. The lower limit of detection was about 0.1 μg.

Desorption of halothane and enflurane from the sampling tubes was quantitative. Recovery from samples stored for eight days at room temperature amounted to 98 per cent for both halothane and enflurane.

TABLE 1. Breakthrough (Maximum Sampling) Times for Halothane and Enflurane in Air

	Concentration*		Temperature (C)	Carrier Gas (Air)		Breakthrough	
	mg/cu m	ppm(vol/vol)		Humidity (Per Cent)	Flow (ml/min)	Time (Min)	Volume (l)
Halothane	78	9.7	25	0	25.0	200	5.0
	78	9.7	25	0	9.9	560	5.5
	77	9.7	30	0	9.9	370	3.7
	77	9.7	30	90	9.8	340	3.3
	77	9.7	30	90	24.8	125	3.1
	400	49.5	25	90	10.0	370	3.7
	393	49.5	30	90	12.1	210	2.5
	403	50.8	30	90	10.3	240	2.5
Enflurane	73	9.8	30	90	10.1	320	3.3
	321	43.3	30	90	9.9	295	2.9

* Measured at the inlet of the sampling tube.

Discussion

The method described can be used for both spot sampling and personal sampling. For the latter application the sampling tube is placed in a special holder and attached to, for instance, the collar of an operating gown, and the pump is carried in a pocket or on a belt. This personal sampling is a method for determination of the average concentration of a polluting agent to which a given individual is exposed for a given period. The method has rarely been used for the measurement of trace amounts of anesthetic agents. A personal sampling method in which the sample is collected in an evacuated bottle has been described by Davenport *et al.*[4] Adsorption on activated charcoal followed by extraction with carbon disulfide has also been applied.[1]

Porapak Q had already been used as an absorbent in the measurement of halothane by Celbicova-Ruzickova *et al.*,[5] who employed the chromatographic equilibration procedure, in which air is drawn through the sampling tube until a concentration equilibrium between the gas phase in the tube and the absorbent has been reached. This method requires a certain minimal volume of air to be drawn through the tubes,

whereas the method described here involves a maximum volume.

When the present method is applied with the simultaneous use of several sampling systems, it can provide information about air pollution in several rooms or about the exposure of several individuals, which permits efficient control in large departments. The method also opens possibilities for control in small hospitals, which may not have the equipment necessary for the analysis. The cost of the sampling apparatus is reasonable, the sampling procedure is rather simple, and the samples can be sent to a laboratory that has the necessary equipment. Since the recovery of the anesthetic agents is still almost 100 per cent even several days after sampling, the samples could be shipped by mail if necessary.

The present method was developed primarily for the analysis of halothane and enflurane. Application to other agents is dependent on the extent to which they are absorbed by Porapak Q. Substances less strongly absorbed can be analyzed by using larger amounts of Porapak Q in the sampling tubes. Extremely volatile substances require a stronger absorbent (*e.g.*, activated charcoal). For substances that are very strongly absorbed by Porapak Q, a higher desorption temperature or a less strongly absorbing porous polymer packing must be used.

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