Journal of Chromatography, 176 (1979) 291–303

CHROM. 11,939

LARGE-BORE COATED COLUMNS FOR SAMPLING AND CONCENTRATION OF ORGANIC VOLATILES IN AIR, HEADSPACE AND WATER ANALYSIS

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(Received February 21st, 1979)

SUMMARY

Large-bore coated (LBC) columns were used as sampling and concentrating traps in analyses for traces of organic volatiles in air and water. This simple technique utilizes long metal columns thinly coated with SE-30 for direct trapping of the organics. The sample is simply passed through the LBC column; the trapped organics are then thermally desorbed onto a conventional porous polymer pre-column or onto a second LBC column. If desired, this can be shorter or narrower bore than the initial LBC sampling column. The sample is finally desorbed onto the gas chromatographic column for analysis. Multiple transfers between LBC columns are possible, with increased concentration at each transfer, resulting in a "concentration pump" effect. The technique offers the advantages of great simplicity, efficiency and ease of sample transfer. Samples are obtained with low back-pressure and minimal interfering artifacts. The system shows almost complete imperturbability to moisture.

Indifference to moisture and the low back-pressure enable direct sampling of very large volumes of air and even breath. Direct sampling of aqueous systems was also possible. The latter area was not fully investigated but offers potential for water pollution analysis and in direct examination of biological fluids and aqueous flavor extracts where heat sensitivity is a problem. With LBC columns the sampling and concentration sequence exposes the substances sought to no more drastic conditions than those they will be subjected to in the process of gas chromatographic analysis.

INTRODUCTION

An increasing interest has been shown within the past few years in headspace and air analyses. The interest in the field is amply demonstrated by the large numbers of publications and symposia, and the numerous techniques devised for sampling, concentration, and analysis of volatiles.

The advent of gas chromatography (GC) has rendered headspace analysis a valuable technique in determination of volatiles at the sub-threshold levels.

* Presented at the fifth annual FACSS meeting on November 1, 1978, in Boston, Mass., U.S.A.

The techniques employed in sampling and concentrating of volatiles in the air and headspace varied with ingenuity of the researchers. The methods included direct sampling and injection on the column of up to 10 ml (refs. 1, 2), concentrating the volatiles on either pre-cooled open tubing[3], or short pre-columns packed with porous polymers[4–9], or with very small amounts of activated charcoal[10,11].

The introduction of porous polymers as trapping devices has facilitated the sampling for volatiles and enhanced the interest in headspace analysis. Environmental awareness and demands have also played a significant role.

In spite of the wide acceptance and use of porous polymers, they are not of absolute efficiency and have certain disadvantages, depending on the amount and type of polymer used, such as artifact peak formation and loss in the more volatile components especially when large volumes are sampled on short pre-columns. Butler and Burke[12] reported on the capacity and efficiency of the various polymers and concluded that each has its own merits, depending on the problem at hand, and none has universal applicability. Mackay and Hussein[13] reported briefly on some of the problems encountered in sampling on porous polymers.

This work deals with a novel technique which involves sampling of air and headspace volatiles on large-bore coated (LBC) columns. The column is thinly coated with SE-30. Other coating materials are possible.

The use of wide bore columns for sampling was suggested[14] in 1977 by using Tygon tubing to sample breath for sulfur-containing onion compounds. The results obtained from the Tygon tubing, although not spectacular, led to the idea that coated metal columns can achieve good results because of the better control in their coating and their less impermeable nature. The use of LBC metal columns was recently reported by the same authors[13]. They illustrated the advantages of sampling on LBC columns and compared them to porous polymer columns.

The predominant uses of coated open-tubular columns are in analysis rather than sampling. These, of course, are the well known Golay columns and their modifications, support-coated open-tubular (SCOT) capillary metal and glass columns. The work on the subject is too extensive to cite. However, the books of Jennings[15] and Ettre[16] give excellent background and coverage. Self *et al.*[17] utilized a few inches of coated capillary Nylon tubing as an enrichment trap for organic volatiles from headspace prior to their analysis on a coated Nylon capillary column. However, the trap was cooled, during sampling, by immersion in liquid oxygen. The flow-rate (<1 ml/min) and the amount of sample were low. Larger volume samples were possible if the concentration of water vapor was low.

Success in GC headspace analysis of very diluted mixtures depends on optimal concentration and transfer of the trace volatiles onto the analytical column. This requires that all components are at maximum concentration in a short single band at the head of the analytical column. This concentration effect is achieved after sampling on LBC column by transfer of the trapped volatiles to a short Tenax-GC pre-column. The use of Tenax-GC pre-column, however, can be avoided by using another shorter, narrower bore LBC column than the sampling column for the transfer trap. The sample is then eluted thermally off the transfer trap onto the analytical column, and the analysis is initiated concurrently with the final desorption.

Due to ease of elution, a single transfer of the trapped sample off the LBC column onto the receiving trap is usually sufficient to achieve the narrow concentra-

tion band on the analytical column. However, multiple transfers which give a "concentration pump" effect can be done, giving a concentration factor of about 10 times at each transfer.

This work demonstrates the applicability of LBC columns to sampling of organic volatiles. The factors affecting the trapping efficiency of LBC columns, such as column length, thickness of coating, sample volume, and sampling flow-rate are investigated. The use of stationary phases other than SE-30 as wall coating materials will be explored in future publications, and also the effect of greatly increased diameter.

Although the main utility of LBC columns is in sampling for and concentration of organic volatiles, some separation can be achieved if used as GC columns. A 50 ft. $\times$ $\frac{1}{4}$ in. O.D. (0.186 in. I.D.) with 0.014 mm SE-30 wall coating has approximately 4000 theoretical plates.

The use of LBC columns offers the advantages of efficiency, low back-pressure, minimal interfering artifacts, ease of sample elution, and indifference to moisture.

The low back-pressure and indifference to moisture are very useful features in breath analysis whereby large samples can be collected by directly blowing on the LBC column with minimal precaution to prevent introduction of saliva onto the column. To minimize moisture condensation, the column was maintained at 50° while sampling.

As a demonstration of the effectiveness of LBC columns in entrapment of organic compounds, flavorants were successfully analyzed for by direct sampling of their extremely dilute aqueous solutions on the LBC column. Flavor profiles were also obtained for soft beverages and beers.

Liquid sampling on LBC columns has a potential in many areas especially in analysis for water pollutants, fingerprinting of oil spills for identification of the source, and in quality control of beverages where a fingerprint of the flavor in the finished product can be easily obtained. This topic is currently being pursued and will be detailed further.

LBC columns are extremely efficient in retention of organic volatiles. However, losses are encountered with the very volatile components. The extent of losses depends on the sampling volume, sampling flow-rate, length and diameter of LBC column, and amount of wall coating.

The utility of LBC columns in sampling for organic volatiles in gaseous media and the factors affecting the efficiency of trapping are demonstrated in this work by a simulated headspace system giving consistently identical samples. A 0.1-μl volume of peppermint oil was deposited in the inlet side of a cold finger-condenser which was maintained at 40°. The effluent side of the condenser was connected to the LBC column with a short PTFE tube. The inlet side of the condenser was connected to a high-purity nitrogen tank. Sampling was initiated by starting the flow of nitrogen, which was previously set at 100 ml/min, and continued until the desired volume was reached.

Upon collection of the sample, the LBC column is heated to desorb the sample onto a short Tenax-GC pre-column or onto another LBC column, which is in turn thermally eluted for analysis onto the analytical GC column.

EXPERIMENTAL

Gas chromatographic conditions

A Perkin-Elmer Model 3920 gas chromatograph with flame-ionization detector was used. The column used was a single aluminum 8 ft. × ¼ in. O.D. (0.186 in. I.D.) packed with 10% Carbowax 20M on Chromosorb W 80–100 mesh (acid washed and DMCS treated). The carrier gas was nitrogen at a flow-rate of 50 ml/min, measured at ambient temperature. The column oven temperature was programmed at 4°/min from 70 to 230°. Injector and interface temperatures were 250°. Recorder: 5 mV at 0.5 in./min chart speed. The integrator used was a Vidar Model 6230.

Materials

Pre-column packing and LBC coating materials were (a) Tenax-GC (60–80 mesh; Applied Science Labs., State College, Pa., U.S.A.) and (b) SE-30 silicon rubber (100% methyl) (Analabs, North Haven, Conn., U.S.A.).

The pre-columns used were short lengths of Pyrex glass tubing 15 cm × 0.6 cm O.D. (0.38 cm I.D.). The material was packed between two glass wool plugs in the pre-column so that the packing is within the hot zone of the tube heating oven which is used for sample desorption.

Large-bore coated columns

Large-bore aluminum (¼ in. O.D.; 0.186 in. I.D.) and stainless-steel (⅛ in. O.D.; 0.08 in. I.D.) tubings of the desired length were coiled to fit in the oven of a gas chromatograph which was used for desorption of the sample. The column was cleaned by repetitive aspiration of benzene and dried. Coating of the column with SE-30 was done following the procedure previously described[13]. The weight of coating in a 50 ft. × 0.186 in. I.D. column ranged from 1.8 to 4.5 g. Thicker coating may be achieved by repetitive application of the SE-30 solution and drying. Thinner coating may also be achieved by quick passage of the solution through the column. Each gram of SE-30 coating in a 50 ft. × 0.186 in. I.D. results in a coating thickness of 0.0046 mm, on the assumption of a uniform coating.

Sampling

A 0.1-μl volume of peppermint oil was deposited onto a small glass wool plug placed in the inlet of a cold-finger condenser. A 0.1-μl syringe fitted with a Chaney adaptor was used to minimize variation in injection size. The condenser, which was maintained at 40° in a water bath prior to deposition of the sample, was connected with a PTFE tube to a purified nitrogen cylinder. The sampling column was connected to the effluent side of the sampling system and the nitrogen flow, which was previously adjusted to the desired rate, was initiated and continued until the desired sampling volume was attained.

The method previously described by Hussein and Mackay[13] was followed in collection of actual air and/or headspace samples.

Sample desorption

The sample was eluted off the sampling LBC column onto a short Tenax-GC

pre-column, or onto a shorter, narrower bore LBC column than the one used for sampling. The sampling column was heated at 250° for 12 min while 50 ml/min nitrogen was flowing through it. The receiving pre-column or short LBC column was outside the heating oven and connected to the sampling column with a short 1/16-in. O.D. stainless-steel tube. The receiving pre-column or LBC column was at ambient temperature.

The sample was eluted for analysis off the receiving Tenax-GC pre-column onto the analytical column as described in the previous work[13,14]. The receiving LBC column was heated to 225° for 6 min, with a heating tape controlled with a thermostat, for desorption of the sample. The nitrogen flow of 50 ml/min of the analytical column was redirected through the LBC column during elution with a Toggle valve.

The LBC elution process was repeated when multiple sample transfer (concentration pump) was desired.

Liquid sampling

A 20-ml aliquot was poured through a 25 ft. × 0.25 in. O.D. (0.186 in. I.D.) LBC column (0.019-mm SE-30 wall coating). The effluent sample was collected in a beaker and recirculated 4 times through the LBC column. The LBC column was rinsed with 2 × 20 ml distilled water to eliminate or minimize any non-volatile residuals remaining on the column.

The column was then purged with nitrogen at 100 ml/min for 5 min to eliminate the bulk of the moisture. The sample was eluted off the LBC column by heating at 250° for 12 min with a 50 ml/min nitrogen flow and trapped onto a Tenax-GC pre-column connected (if desired) to a back-up charcoal pre-column containing 5 mg charcoal, 90–100 mesh. Both pre-columns were maintained at ambient temperature. The use of the charcoal is to trap escaping volatiles off the Tenax-GC pre-column.

The extent of moisture on the pre-column depends on the nature of the sample. If the moisture is excessive, the pre-columns, in tandem, are purged with nitrogen at 100 ml/min until no visible moisture is noted. The direction of nitrogen flow should be the same as that of sample elution onto the pre-columns. The time required varied from 15 to 50 min.

An alternative procedure for minimizing moisture condensation on the pre-column is to maintain their temperature at 50° during elution of the sample off the LBC column. An infrared lamp can be used for this procedure.

The final analyses were performed by desorption of the volatiles at 250° in the case of the Tenax-GC pre-column, and at 300° for the charcoal pre-column.

RESULTS AND DISCUSSION

Peppermint oil was chosen for the simulated headspace sampling method because of its complexity and the diversity in volatility of its constituents, and because it is widely used and identities of its components are well established.

The simulated headspace sampling method is similar to real situations of volatile stripping. However, continuation of sampling beyond 3 l of the stripping gas, when the sample has already been effectively transferred onto the LBC column, is a severe test of the trapping efficiency of the sampling device, since, contrary to usual headspace sampling, the organic volatiles introduced into the column in the first few

TABLE I

COMPARISON OF SAMPLING EFFICIENCY OF IDENTICAL AMOUNT OF PEPPERMINT OIL USING VARYING SAMPLING VOLUMES ON LBC COLUMN AND A SHORT TENAX-GC PRE-COLUMN

LBC column: 50 ft. × 0.186 in. I.D.; 0.014 mm coating. Tenax-GC pre-column: 200 mg Tenax.

Compound	*Amount in 0.1-μl sample (μg)*	*Sampling efficiency (%)* *			
		LBC column			*Tenax-GC pre-column; 3 l*
		3 l	*24 l*	*100 l*	
α-Pinene	0.9	101.9	8.2	0	18.1
β-Pinene	1.4	98.2	19.7	0	32.6
Limonene	1.4	108.3	149.7	147.0	109.1
Eucalyptol	5.3	95.7	25.1	14.4	47.4
Menthone	18.9	91.8	84.1	69.4	91.5
Menthofuran	3.9	101.3	87.7	73.8	86.1
Isomenthone	3.1	106.4	217.8	207.4	83.5
Menthyl acetate	5.0	94.3	92.7	76.1	98.4
Neomenthol	4.8	96.7	76.0	38.0	91.8
Menthol	41.3	96.1	88.5	62.3	96.3

* Expressed as percentage of amount found in direct injection of same amount of oil.

liters, are now followed by very large volumes of pure carrier gas which tends to the volatiles right through the trap.

The data in Table I compare the retention of peppermint oil components on

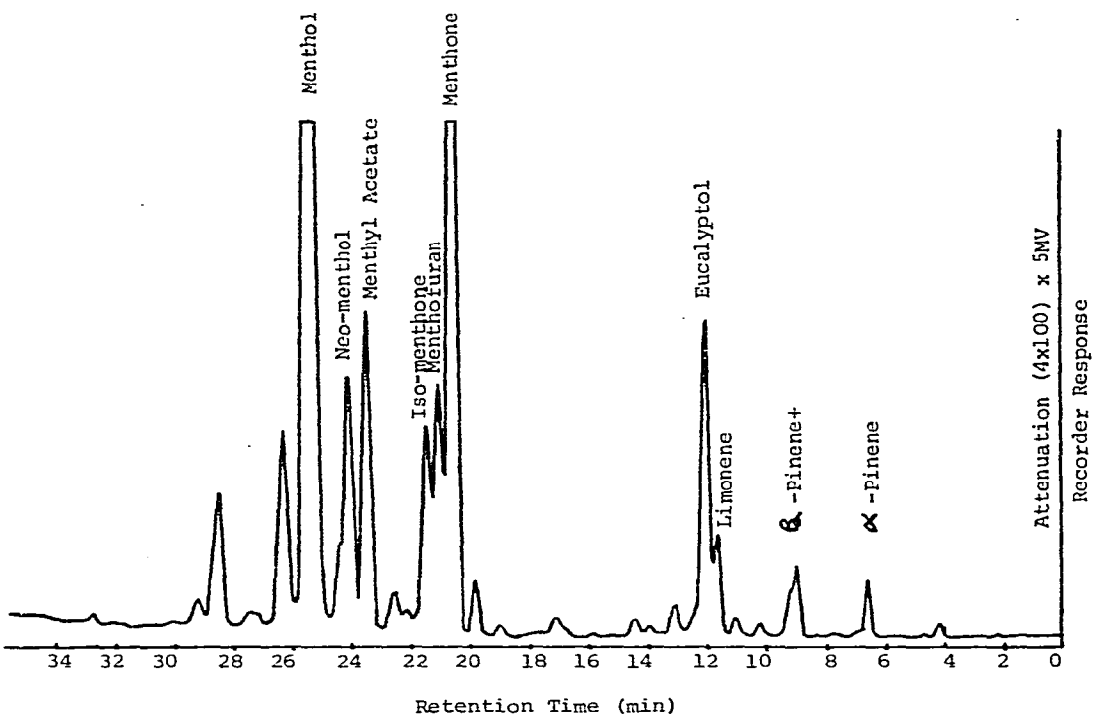

Fig. 1. Analysis of peppermint oil; 0.1-μl sample injected directly into the gas chromatograph.

a 50 ft. × 0.186 in. I.D. LBC column and a wall coating of 0.014 mm SE-30 with various sampling volumes from 3 to 100 l, and retention of the same components on a short Tenax-GC pre-column with 3 l of flushing gas. Retention of the more volatile components, α- and β-pinene, with this sampling volume is better on the LBC column than the Tenax pre-column.

The higher the volume of gas passed through the column, the higher the loss, especially of the more volatile compounds. The high value of limonene can not be explained except for the possibility of an artifact that is more evident with the large flush volume. The high value of isomenthone is also hard to explain except for the possibility of isomerization of some menthone. Such isomerization with heat is possible as has been reported by De Mayo[18].

Figs. 1 and 2 show GC analyses of 0.1 μl peppermint oil by direct injection and by sampling on a 50 ft. × 0.186 in. I.D. LBC column (0.014 mm SE-30 coating) with 3 l nitrogen. The LBC column sample was in turn eluted onto a Tenax-GC pre-column. Aside from the two artifact peaks in the LBC analysis, the chromatograms are similar. The encountered artifacts are generated from the SE-30 coating. The presence of these artifacts will interfere in the analysis only of highly volatile constituents.

The volatile organic compounds are retained by the LBC column due to absorption by the wall coating. The extent of retention of the sample components is, therefore, affected by dimensions of the LBC column, amount of wall coating, and

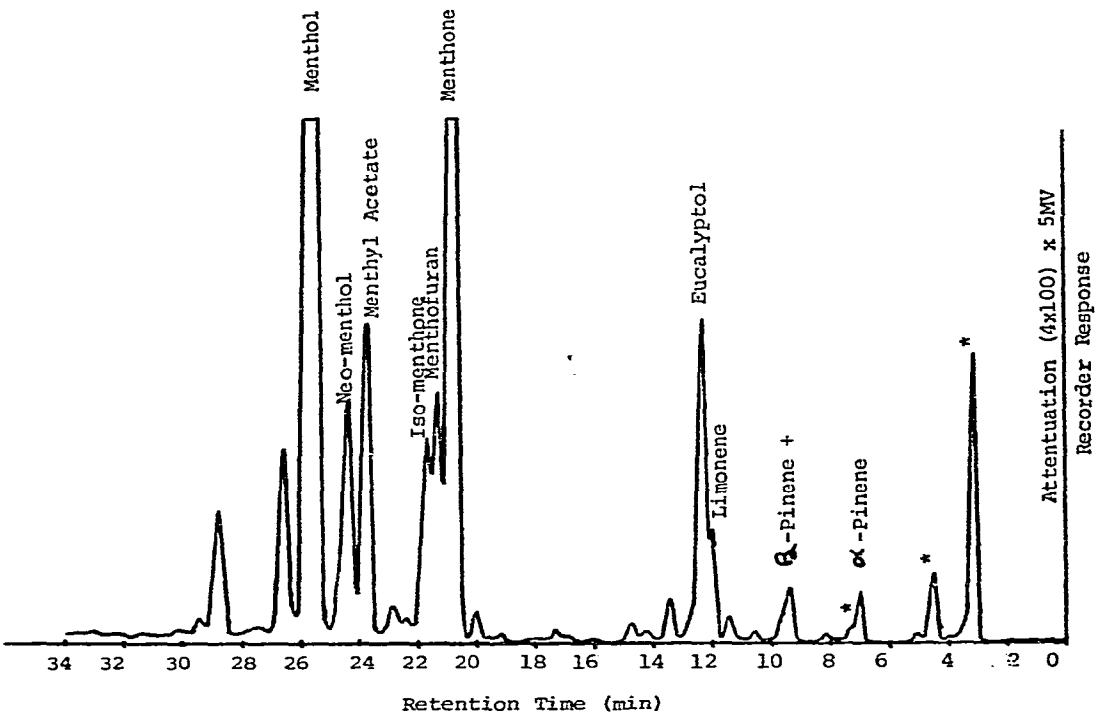

Fig. 2. Analysis of 0.1 μl peppermint oil after sampling on LBC column by sweeping with 3 l nitrogen. * = Artifact from LBC column.

volume of sample or sweep gas. Table II shows differences in retention of components of the identical amount of peppermint oil when sampled with 3 l nitrogen on LBC columns with various wall coating thickness, length, and internal diameter.

TABLE II

COMPARISON OF SAMPLING EFFICIENCY OF LBC COLUMNS OF VARIOUS DIMENSIONS AND WALL COATING WITH IDENTICAL AMOUNTS OF PEPPERMINT OILS AND SAME SWEEP VOLUMES

Column dimensions: A, 5 ft. × 0.186 in. I.D. (wall coating, 0.104 mm); B, 5 ft. × 0.186 in. I.D. (wall coating, 0.025 mm); C, 10 ft. × 0.186 in. I.D. (wall coating, 0.030 mm); D, 3 ft. ×0.085 in. I.D. (wall coating, 0.066 mm); E, 5 ft. × 0.085 in. I.D. (wall coating, 0.055 mm).

Compound	*Sampling efficiency (%)**				
	A	*B*	*C*	*D*	*E*
α-Pinene	120.1**	11.9**	44.1	0	0
β-Pinene	90.9	0.5	84.1	0	0
Limonene	109.9	84.3	126.7	20.2	30.0
Eucalyptol	104.3	33.8	102.8	3.3	4.1
Menthone	99.7	80.9	86.6	81.2	87.6
Menthofuran	101.7	95.6	89.7	86.3	92.0
Isomenthone	99.6	101.7	112.8	129.2	145.7
Menthyl acetate	110.5	83.7	92.3	90.1	99.3
Neomenthol	112.0	91.0	98.8	98.0	105.0
Menthol	102.4	88.3	93.8	92.1	102.3

* Expressed as percentage of amount found in direct injection of same amount of 0.1 μl.
** Some artifact interference.

The effect of the coating thickness is noted in columns A and B of Table II when the same dimension columns were coated with 0.104 and 0.025 mm. The higher thickness resulted in full retention of all components, while appreciable losses are noted in α- and β-pinenes from the lower thickness of wall coating, but when the internal diameters are equivalent, the longer column is more retentive as can be seen from columns B and C of Table II.

The wider bore LBC columns, unexpectedly, are more efficient than the narrower bore ones even if the wall coating is thicker in the latter, as seen from columns B and E of the table.

In all cases the less volatile components, menthol, neomenthol, and menthyl acetate are fully retained.

The flow-rate of sampling onto the LBC column also affects the extent of retention of the organic volatiles by the column. Table III shows a comparison in retention when the identical samples are swept on the column with 12 l nitrogen at 100 and 500 ml/min. While full retention of all components was obtained with the 100 ml/min flow-rate, appreciable losses occurred in the more volatile components (the pinenes, limonene, and eucalyptol) with the high flow-rate.

As an illustration of the utility of LBC columns in real situations, volatile organics were determined in the air of a confectionery plant by sampling 6 l at 100 ml/min on a 50 ft. × 0.186 in. I.D. column coated with a 0.014-mm layer of SE-30. The analysis is shown in Fig. 3. In addition to sampling for volatiles, solids

TABLE III

COMPARISON OF SAMPLING EFFICIENCY OF IDENTICAL AMOUNTS OF PEPPERMINT OIL ON SAME LBC COLUMN USING SAME SAMPLING VOLUME (12 l) BUT VARYING SAMPLING FLOW-RATE

Compound	*Sampling efficiency (%)* *	
	100 ml/min	*500 ml/min*
α-Pinene	96.4	23.0**
β-Pinene	97.7	13.7
Limonene	109.5	86.0
Eucalyptol	104.6	59.7
Menthone	99.7	82.7
Menthofuran	101.8	83.6
Isomenthone	112.6	95.3
Menthyl acetate	87.3	107.2
Neomenthol	107.3	101.0
Menthol	94.8	80.3

* Expressed as percentage of amount found in direct injection of same amount of oil.
** Some artifact interference.

were also sampled and analyzed for simultaneously in the same sample by attaching a short pyrex glass tubing containing a small wad of pre-cleaned glass wool to the inlet of the LBC column. The volatile organics were trapped on the LBC column while the solids (sucrose and glucose) were retained by the glass wool pre-filter. The glass-wool filter was extracted with 10 ml distilled water and sucrose and glucose were determined as silyl ether derivatives by GC on an OV-17 column[19]. The amounts of sucrose and glucose were 12.4 and 0.6 μg/l air, respectively. The total organic volatiles in the air sample was 1.58 mg, *i.e.* $2.6 \cdot 10^{-4}$ g/l.

Identities of the components indicate that peppermint, spearmint, and wintergreen oils are in the sampled air. Candies were being manufactured with the three products at the time.

In a previous work by the authors[13] comparisons of sampling large air volumes on LBC and long Tenax-GC columns were shown. Sampling on the latter was reported to have the disadvantage of large interfering artifacts. However, retention of the extremely volatile components, excluding alcohols, was found to be better on the long Tenax column. The ideal system is simultaneous sampling on both LBC and long Tenax columns. The sample from the Tenax column is utilized for analysis of the highly volatile components, using the LBC sample for the remaining components.

Sub-threshold levels of air odorants were previously[13] determined through sampling large air volumes on LBC columns outside a confections manufacturing plant.

Since the artifact interference from the Tenax-GC is not significant when a short pre-column with a small amount of Tenax is used, the final sample elution was made onto this type of pre-column. The short pre-columns were used for convenience. The use of porous polymers can be avoided by carrying out the final elution onto a shorter and narrower LBC column than the one used for sampling. This was successfully carried out with comparable results to those obtained when the Tenax-GC pre-columns were used as the final elution traps.

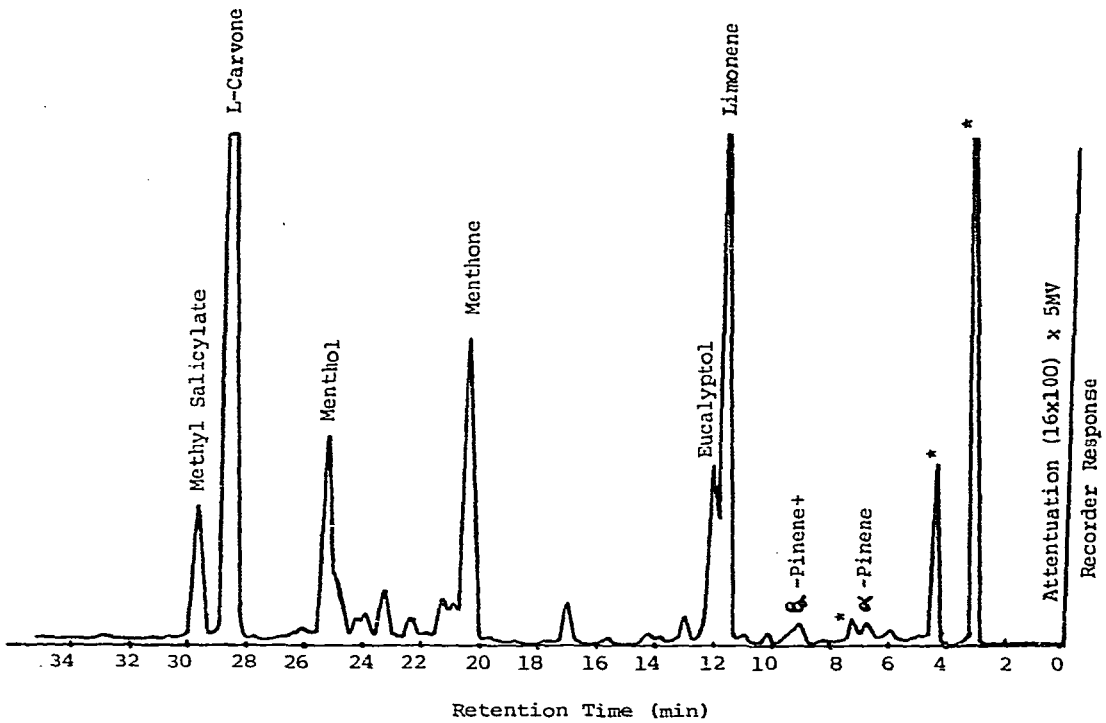

Fig. 3. Analysis of 6 l air in confections plant; sampled directly onto LBC column at a flow-rate of 100 ml/min, desorbed onto a short Tenax-GC pre-column. * = Artifact from LBC column.

TABLE IV

COMPARISON OF MULTIPLE SAMPLE TRANSFERS (CONCENTRATION PUMP) BETWEEN TWO LBC COLUMNS

A 0.1-μl volume of peppermint oil was swept onto the first column with 3 l nitrogen.
A, Sample trapped on a 50 ft. × 0.186 in. I.D. LBC column (0.014 mm coating) and transferred onto another 5 ft. × 0.186 in. I.D. column (0.104 mm coating) followed by elution onto Tenax-GC precolumn and final analysis. B, Sampling and transfer columns are same as in A. Sample was cyclically transferred between the two columns 4 times prior to final elution onto Tenax-GC pre-column and analysis.

Component	*Recovery (%)**	
	A, 1 transfer	*B, 4 transfers*
α-Pinene	**	**
β-Pinene	76.1	20.8
Limonene	127.4	67.7
Eucalyptal	111.0	100.5
Menthone	76.2	54.6
Menthofuran	96.7	60.2
Isomenthone	101.7	203.2
Menthyl acetate	133.7	89.1
Neomenthol	109.6	64.3
Menthol	103.8	58.2

* Compared to amount found in direct injection 0.1 μl peppermint oil.
** Not determined due to an interfering artifact.

Multiple transfer of the trapped sample between two LBC columns was carried out to demonstrate its feasibility. However, the ease of sample elution off the LBC column usually renders the process of multiple transfer or "concentration pump" unnecessary except for extremely dilute concentrations.

The data presented in Table IV illustrate the feasibility of the principle with relatively little sample loss. Recovery of trapped components after two and four multiple transfers between the 50-ft. LBC column and another 5-ft. one shows the higher the number of transfers the higher the loss of sample. However, achieving 60% or higher recovery of the sample after four transfers, although not ideal, is satisfactory considering the small sample (90 μg).

Although the concentration ratio between the original sample volume and volume of carrier gas used for the final transfer is 5, the extent of concentration can be much larger depending on the initial sampling volume, the number of transfers, and types of columns.

Indifference of the LBC columns to moisture and their low back-pressure feature enables sampling and analysis of large volumes of breath for organic volatiles. The sample was collected by directly blowing into the column through a PTFE mouthpiece which contained a glass-wool plug to inhibit introduction of saliva onto the column. The column was maintained at 50° during sampling to minimize condensation of moisture. Fig. 4 shows analysis for sulfur components in 5 l of breath after the mouth was rinsed with dilute onion oil solution. The procedure for mouth

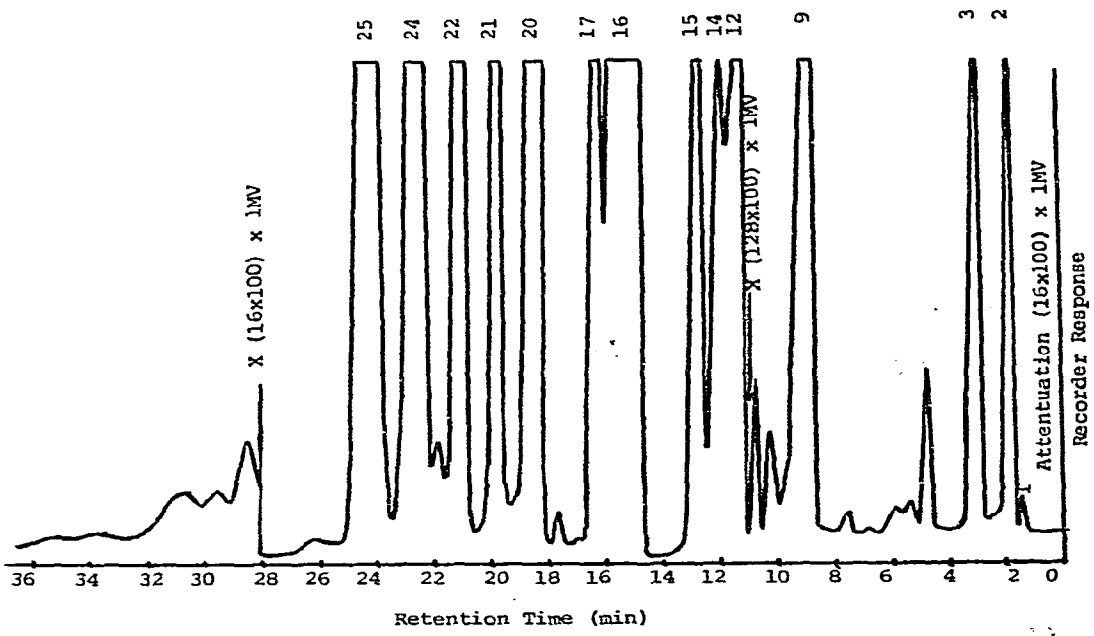

Fig. 4. Analysis for sulfur compounds in breath following mouth rinse with 0.005% onion oil solution; 10 large breaths sampled directly on LBC column (50 ft. × 0.186 in. I.D.; 0.014 mm SE-30 coating). Sample was desorbed onto short Tenax-GC pre-column and eluted on GC column for analysis. FPD detector was used; other GC conditions are same as previously reported[14]. Tentative peak identity: 1 = methyl mercaptan; 2 = dimethyl sulfide; 3 = propyl mercaptan; 9 = dimethyl disulfide; 16 = dipropyl disulfide.

odorization and analysis are the same as reported in the previous work[14], The work should be consulted for further reference on onion breath. Except for a slight methyl mercaptan odor, no onion odor was noted in the LBC column effluent upon sampling of the breath. Analysis of the breath on a LBC column for peppermint oil components after ingestion of peppermint candy was reported in a previous work[13].

Effectiveness of the LBC columns in retaining organic volatiles is dramatically illustrated by their use in entrapment and analysis of trace peppermint oil constituents in an aqueous solution containing a trace amount of peppermint oil. Fig. 5 shows analysis for peppermint oil components after a 20-ml aliquot of a 10-ppm solution was recirculated through a 25 ft. × 0.186 in. I.D. LBC column, coated with 2.1 g SE-30 at approximately 0.019 mm thickness.

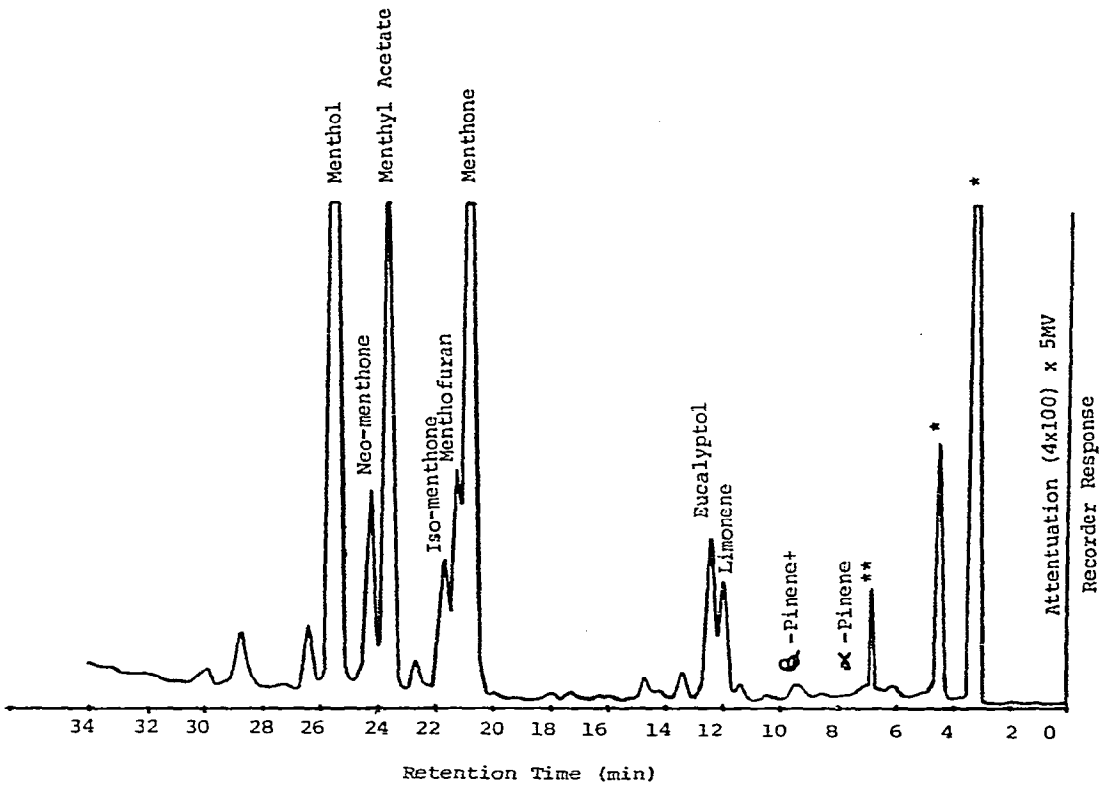

Fig. 5. Analysis of dilute aqueous peppermint oil solution (0.001 % peppermint oil) by direct sampling onto 25 ft. × 0.186 in. I.D. LBC column; 20-ml aliquot was recirculated 5 times through the column. * = Artifact from LBC column; ** = quench effect of residual moisture.

The recirculated aliquot contained 0.2 μl peppermint oil. Recovery of the individual components varied. While 68% of the menthyl acetate and 60% of the menthone in the aliquot was trapped and accounted for, only 23% of menthol was recovered. The wide difference in recoveries from the solution points to preferential partition by the SE-30 coating.

Although the profile of the peppermint oil components recovered by the LBC

column from the aqueous solution differs from that of direct analysis of the oil (Fig. 1), it still is unmistakably that of peppermint.

CONCLUSIONS

The LBC columns have been proven effective in concentration of volatiles from air. The features of low back-pressure and the indifference to moisture render the technique extremely useful in analysis for organic volatiles in the breath and large air samples.

Direct sampling of liquids on LBC columns is a promising area especially for water pollution analysis and quality control of flavors in beverages.

ACKNOWLEDGEMENT

The authors acknowledge the invaluable assistance of A. R. Pidel in the performance of the analyses.

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