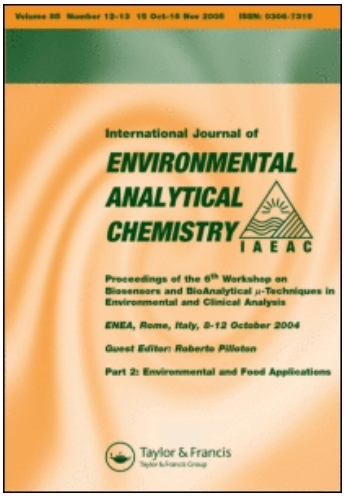

International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

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To cite this Article Krzymien, M. E.(1985) 'Dual Adsorber-Capillary Column System for Gas Chromatographic Analysis of Air Samples', International Journal of Environmental Analytical Chemistry, 21: 1, 43 — 62
To link to this Article: DOI: 10.1080/03067318508078370
URL: http://dx.doi.org/10.1080/03067318508078370

Intern. J. Environ. Anal. Chem., 1985, Vol. 21, pp. 43–62
0306-7319/85/2102-0043 $18.50/0

Dual Adsorber–Capillary Column System for Gas Chromatographic Analysis of Air Samples

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(*Received December 7*, 1984; *in final form February 14, 1985*)

An interface which allows thermal desorption and subsequent capillary gas chromatographic analysis of air samples is described. A small solid-sorbent trap is positioned between the sampling tube and capillary column. A sample thermally released from the sampling tube is transferred by a carrier gas at high flow rate to the trap and retained. From there it is again thermally released and transferred to the capillary column by carrier gas at a low flow rate, as required by capillary GC. The transfer and injection steps are effected by means of externally placed solenoid valves. The performance of the system depends on the desorption temperature and time allowed for transfer of the sample between the two adsorbers and the column. These parameters are programmable and can be changed to suit the requirements of a particular analysis. The system allows the analysis of sub-parts-per-billion concentrations of organic compounds in a comparatively simple and reproducible manner. Operation of the system does not require cryogenic cooling of either the trap or the GC oven. Chromatograms of a variety of air samples are presented and discussed.

KEY WORDS: Capillary GC, organics in air, solid sorbent trapping, thermal desorption.

1. INTRODUCTION

Preconcentration of air contaminants on solid sorbents has gained wide recognition and has been applied to the analysis of a great number of air pollutants.

When air passes through a tube filled with a sorbent, contaminants are retained until a breakthrough volume is exceeded. The trapped analytes are recovered from the sorbent either by extraction with a solvent or by thermal desorption.

Solvent extraction does not necessitate modification of an instrument, since the sample is introduced as a solution by syringe injection. The main disadvantage of this technique is dilution of the analytes, usually by a factor of $10^2 - 10^3$, a serious drawback in trace analysis.

Thermal desorption on the other hand allows introduction of the entire collected sample into the gas chromatograph without dilution. The main disadvantages of the method are:

1) inability to repeat the analysis of the same sample, and
2) necessity of hardware changes to the instrument.

Its advantages are speed of analysis and high sensitivity.

Sorbent trapping followed by thermal desorption is used mostly in gas chromatography with packed columns. The technique has been applied in this laboratory for analysis of explosives,[1] pesticides[2,3] and formaldehyde.[4] A two-stage adsorber configuration with packed column has been employed in a portable gas chromatographic explosives detector[5] and for analysis of insecticides and herbicides.[6]

The application of thermal desorption to capillary gas chromatography is more complicated owing to the low carrier gas flow required for the capillary column which conflicts with the much higher flow required to transfer desorbed analytes from a sampling tube to the column. This seemingly inherent problem may be solved by introducing a trap in front of the column. The trap might be either a small packed or capillary precolumn or head of a capillary column cooled to sub-ambient temperature.[7–17] Kebbekus and Bozzelli[15] reported an interesting technique where the thermally desorbed sample is transferred to a pressure vessel and from there via a sampling loop to the GC. It enables the analysis of the aliquots of the same sample to be made. Unfortunately, this technique is limited to rather volatile compounds, since low vapour pressure substances would require excessive heating of the vessel to avoid adsorption on the walls. In addition sensitivity is lost due to dilution of the sample with carrier gas in the pressure vessel.

There also exists a commercial instrument (Perkin–Elmer ATD 50)

which desorbs analytes automatically from fifty sampling tubes and transfers them to a gas chromatograph with either packed or capillary columns. The instrument, however, uses only special metal sampling tubes which may not be suitable for labile compounds.

The device described in this paper was developed as a modification of the Varian 4600 or 6000 gas chromatograph operated by the Vista 401 or Vista 402 Data System, and relies on the automation capabilities of the Vista System. It can, however, be applied to any gas chromatograph. Operation of the device does not require cryogenic cooling of either the trap or the GC oven. Sub-ambient oven temperatures might however be helpful in the analysis of volatile compounds.

The basic elements of the device are a small packed precolumn and carrier gas switching valves which allow the low flow through the capillary column and high flow to transfer the desorbed analytes from the sampling tube to the precolumn.

2. EXPERIMENTAL

2.1 Construction of a dual adsorber system

The system consists of two basic units connected in series with a capillary column. The first unit (Part A, Fig. 1) is a sampling tube 6.4 mm O.D. × 76 mm packed with a solid sorbent held in place by two glass wool plugs. For analysis it is inserted into the injector, modified as shown in Figure 1. The second unit or the second adsorber (Part B, Fig. 1) is connected to the injector with a $\frac{1}{4}$-to-$\frac{1}{8}$ reducing graphite-filled Vespel ferrule and a Swagelok fitting. It is a 3.2 mm O.D. (2.4 mm I.D.) × 63.5 mm nickel tube with a $\frac{1}{16}$ Swagelok union silver-soldered to the lower end. The tube is clustered by two 4.8 mm I.D. × 38 mm and one 2.5 mm I.D. × 38 mm thin wall stainless steel tubes which are silver-soldered together. The two larger tubes house two 30 W cartridge heaters and the smaller one contains a 100 ohm platinum temperature sensor. The cluster of tubes is wrapped in one layer of glass fibre insulating tape. The cartridge heaters ballistically heat the adsorber from 40° to 220° in about a minute. The second adsorber tube is packed with Tenax-GC 80/100 mesh held in place by two glass wool plugs. Above and below the glass wool plugs there are two silver-soldered gas lines which are

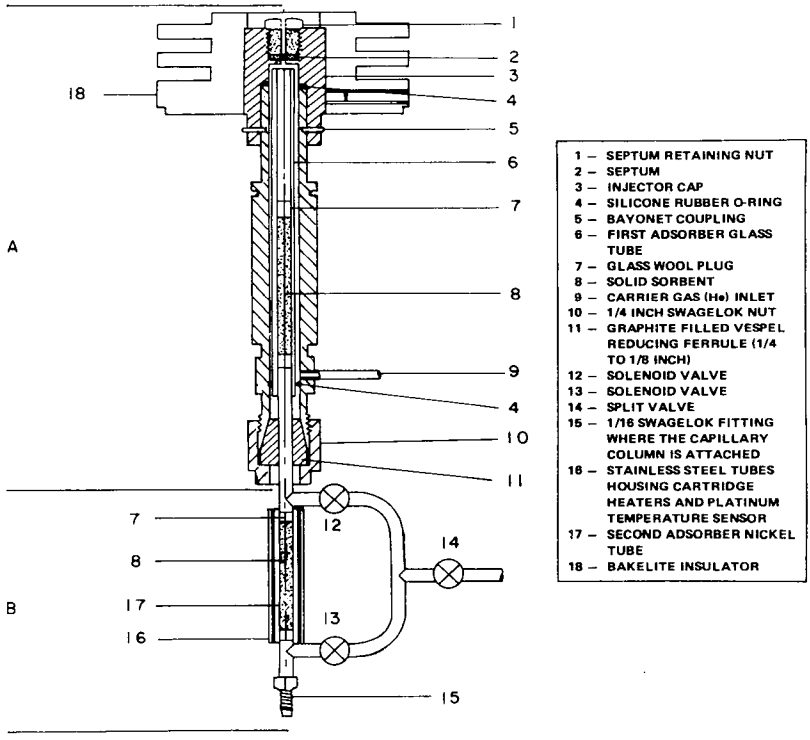

FIGURE 1 Dual trap.

connected to solenoid valves, and the valve outlets are further connected to a microneedle valve which provides a desirable split ratio. The second adsorber (Part B, Fig. 1) is placed inside the GC oven. The solenoid valves and the split valve are in a separate pneumatics compartment outside the oven.

2.2 Operation

When the sampling tube (first adsorber) is inserted into the hot injector, the GC oven and the second adsorber are kept at an initial temperature which is low enough to trap the sample. At this stage valve 12 (Figure 1) is closed and valve 13 remains open, so the carrier gas flows through the sampling tube at a rate of 80 cm^3/min

and transfers thermally released sample to the second adsorber (cool) where it is trapped while carrier gas passes through valve 13 and vents to the ambient through the split valve 14. A fraction of the carrier gas (depending on the split ratio) flows into the capillary column. Usually the flow velocity through the column is 50 cm/sec. Upon completion of the transfer (transfer time is adjustable from 0.1 to 99 minutes) valve 12 opens, valve 13 closes, and a relay connects power from the temperature controller to the cartridge heaters. At this moment the oven temperature might start rising or might remain at some initial level for a time, as required by the analysis.

When the analysis is completed the system automatically returns to the initial stage and is ready for the next analysis.

2.3 Chromatographic analysis

All the work was done on a Vista 44 Automated Gas Chromatograph. Typical chromatographic conditions were as follows: column—15 m × 0.2 mm I.D. fused silica coated with SE-30 (J&W Scientific); carrier gas—helium at 80 cm^3/min through first and second adsorbers during sample transfer to the second adsorber and 50 cm/sec through the column, 1 cm^3/min through second adsorber during analysis. Make-up carrier 25 cm^3/min. Detector—FID, air at 300 cm^3/min, hydrogen at 25 cm^3/min; range 10^{-12} A. Temperatures: injector—variable up to 280°; second adsorber—initial same as oven, final up to 260°; oven—initial 35° held for 3 min, final 160° at 10° per min and held for 2 min; detector—220°. Transfer time—3 min. Second adsorber heater on from 3.25 min to 15.5 min.

Injector, second adsorber and initial temperatures as well as initial temperature hold time and relays adtivation times were varied to test the effect of these parameters on the system performance.

2.4 Test mixtures

Performance of the dual trap system was evaluated primarily by analysis of a mixture of four hydrocarbons: *n*–dodecane (C_{12})(24.62%), *n*-tridecane (C_{13})(24.86%), *n*-tetradecane (C_{14})(25.09%) and *n*-hexadecane (C_{16})(25.43%) (Mixture 21A supplied by PolyScience, Niles, Ill.) with boiling temperatures ranging from 214.5° to 287.5°. To broaden the boiling point range a 50–50 mixture of *n*-octane (b.p. 125.8°) and *n*-pentadecane (b.p. 270.5°) was analysed. The

hydrocarbons were dissolved in methylene chloride. Samples (5–10 ng per component) were either injected via the septum directly to the column, injected and trapped on the second adsorber or deposited on the Tenax bed of the first adsorber.

Air samples were collected by drawing air, usually at a rate of 200 cc/min, through the sampling tube with a diaphragm pump.

3. RESULTS AND DISCUSSION

Retention times of Mixture 21A components remained essentially constant regardless of changes of the second adsorber temperature (Table I). An initial oven temperature of 35° to 50° was sufficiently low to trap the hydrocarbons at the head of the column. The same occurs in the case of *n*-octane, although the latter elutes very closely to the solvent. For compounds with a boiling point below 130° a sub-ambient initial temperature should be used.

Separation number and resolution. It was thought initially that unavoidable dead volumes in the connections between segments of the system might adversely affect the overall performance. The chromatograms shown in Figure 2, however, demonstrate that the system performs relatively well. The separation number (TZ) calculated for C_{12} and C_{13} equals about 12, and the resolution $R = 16$

TABLE I

Retention time reproducibility

II trap temperature °C	Retention time (min.)			
	C_{12}	C_{13}	C_{14}	C_{16}
160	No peak	No peak	No peak	No peak
170	No peak	No peak	No peak	No peak
180	9.20	10.5	11.9	No peak
200	9.18	10.5	11.8	14.2
220	9.17	10.5	11.9	14.2
240	9.20	10.5	11.8	14.2
	9.18	10.5	11.8	14.2
	9.19	10.5	11.8	14.2
250	9.18	10.5	11.8	14.2
260	9.19	10.5	11.8	14.2

FIGURE 2 Separation of mixture 21A. First adsorber—220°, second—240°, transfer time 3 minutes.

which is about ten times the value usually considered adequate for complete separation (6 or $R = 1.5$). The separation power of the capillary column thus has not been lost by introducing the dual trapping system in front of the column. The peaks do show some tailing which, however, is not severe and does not significantly affect the performance of the system. The tailing could probably be reduced by using subambient initial temperatures which would allow the sample to be trapped at the head of the column in a narrower plug.

Sample discrimination was carefully investigated as an important aspect of system performance. Lighter components of a sample might not be trapped efficiently and heavier ones might not be released quantitatively. An apparent change of sample composition would be observed.

The discrimination may occur in the first as well as the second trap. Each one, therefore, was investigated separately. First, the system was operated with only the second trap in place. The sample was injected via the septum into the space normally occupied by an adsorber tube which, for this test, was replaced by an injection port insert to reduce the injector volume. The sample was retained on the second trap and then thermally desorbed for analysis. Maximum temperature of the second trap (auxiliary temperature) was varied from 180° to 260°. The initial oven temperature was maintained for an additional 15 seconds from the moment when power was applied to the second trap heaters.

As evident from Table II, the peak area of the light component, C_{12}, is constant from 200°, while that of the heaviest component, C_{16}, increases with temperature. At the maximum temperature employed, 260°, the ratio C_{12}/C_{16} is close to the theoretical value of unity.

For evaluation of the dual adsorber system the hydrocarbon mixture was deposited on the Tenax bed with a syringe. The adsorber was then inserted into the hot injector with the sample end toward the second trap (back flush) or away from the trap so the sample had to pass through the entire bed of Tenax. The injector temperature was varied from 140° to 220°. The second trap was heated to 260° for desorption.

At 140° the hydrocarbons were released from the first adsorber only in the back flush position (Table III) when the sample was in contact with a very small amount of Tenax. No hydrocarbons were released at this temperature if the sample was forced to pass through the entire length of Tenax adsorber. At low adsorber temperatures only a fraction of the deposited sample was recovered, with a strong bias against heavy components.

As indicated in Table III, the sample was completely recovered at 200–220°. At this temperature measured peak area ratios are close to theoretical ones. In the back flush position some discrimination against C_{12} and C_{13} occurs, probably because part of the hydrocarbons is lost during insertion of the adsorber tube into the hot injector. There is little or no loss in the "normal" position because the top of the tube (where the sample is deposited) heats up more slowly and reaches desorption temperature after the injector is closed. This, therefore, is the preferred way of inserting the adsorber tube.

TABLE II
Second adsorber discrimination

Temperature °C	Areas				Ratios		
	C_{12}	C_{13}	C_{14}	C_{16}	C_{12}/C_{13}	C_{12}/C_{14}	C_{12}/C_{16}
160	Peak unidentifiable						
180	83997	52244	29234	31109[a]	1.61	2.87	2.70
	75035	79144	29784	32289	0.95	2.52	2.32
	87315	46644	30176	52012	1.87	2.89	1.68
				Average	1.48	2.76	2.23
				S.D.	0.47	0.21	0.52
200	108335	106830	101350	78793	1.01	1.07	1.37
	105291	103817	98640	78326	1.01	1.07	1.34
	102457	100936	95303	75644	1.02	1.08	1.35
				Average	1.01	1.07	1.35
				S.D.	0.01	0.01	0.02
220	104458	102605	96927	77028	1.02	1.08	1.36
	105891	104300	102034	78622	1.02	1.04	1.35
	110416	109422	104598	85861	1.01	1.06	1.29
				Average	1.02	1.06	1.33
				S.D.	0.01	0.02	0.04
240	105494	105696	104948	89881	1.00	1.01	1.17
	106525	105964	102483	88479	1.01	1.04	1.20
	103284	102908	102504	87940	1.00	1.01	1.17
				Average	1.00	1.02	1.18
				S.D.	0.01	0.02	0.02
260	108629	109281	106264	94209	0.99	1.02	1.15
	110251	113356	109823	98216	0.97	1.00	1.12
	107149	107641	108321	94379	1.00	0.99	1.14
				Average	0.99	1.00	1.14
				S.D.	0.02	0.02	0.02

[a]Peak not well shaped. Sometimes doubled or tripled owing to temperature overshots and retrapping.

Duration of transfer of a sample from the first to the second adsorber also influences the performance of the system (Table IV). Time must be allowed for the adsorber to reach desorption temperature and then for a sample to pass through the Tenax column (first adsorber) and connecting lines to the second trap. However, if the transfer time is excessively long the sample breaks through the

TABLE III

Two adsorber discrimination.

I adsorber temperature[a] °C	Back flash position							Normal position						
	Areas				Ratios			Areas				Ratios		
	C_{12}	C_{13}	C_{14}	C_{16}	C_{12}/C_{13}	C_{12}/C_{14}	C_{12}/C_{16}	C_{12}	C_{13}	C_{14}	C_{16}	C_{12}/C_{13}	C_{12}/C_{14}	C_{12}/C_{16}
140	66931	67299	66016	55344	0.99	1.01	1.21			No peaks				
160	72342	77745	76852	65125	0.93	0.94	1.11	25457	6725	No peak	No peak	3.79	—	—
								9063	2927	No peak	No peak	3.10	—	—
								30724	10316	No peak	No peak	2.98	—	—
											Average	3.29		
											S.D.	0.44		
180	71555	72640	72576	70148	0.99	0.99	1.02	69136	62807	45090	8856	1.10	1.53	8.32
	63136	63608	66235	67164	0.99	0.95	0.94	62926	58806	44661	8310	1.07	1.41	7.57
								77251	65279	55429	9140	1.18	1.39	8.45
				Average	0.99	0.97	0.98					1.12	1.44	8.11
				S.D.	0.00	0.03	0.06					0.06	0.08	0.48
200	68309	68098	70857	75584	1.00	0.96	0.90	71459	72024	72526	61933	0.99	0.99	1.15
	67994	70794	75583	83817	0.96	0.90	0.81	62451	63074	61213	49404	0.99	1.02	1.26
								63213	65009	64424	56532	0.97	0.98	1.12
				Average	0.98	0.93	0.86					0.98	1.00	1.19
				S.D.	0.03	0.04	0.06					0.01	0.02	0.10
220	61075	60815	65443	78990	1.00	0.93	0.77	64687	67410	66610	57882	0.96	0.97	1.12
	64821	62783	63682	79343	1.03	1.02	0.82	65669	65788	65263	58717	1.00	1.01	1.12
	64768	63984	68773	81121	1.01	0.94	0.80	65821	67044	66464	57051	0.98	0.99	1.15
				Average	1.01	0.96	0.80					0.98	0.99	1.13
				S.D.	0.02	0.05	0.03					0.02	0.02	0.02

[a]Second adsorber temperature 260°. Transfer time 3 minutes.

TABLE IV
Transfer time

Transfer time min.	Areas				Ratios		
	C_{12}	C_{13}	C_{14}	C_{16}	C_{12}/C_{13}	C_{12}/C_{14}	C_{12}/C_{16}
1	65402	65153	57782	37573	1.00	1.13	1.74
	64170	62336	56680	37966	1.03	1.13	1.69
	63308	61805	57781	35589	1.02	1.10	1.78
				Average	1.02	1.12	1.74
				R.S.D.%	1.50	1.55	2.60
				S.D.	0.02	0.02	0.05
2	63214	64928	67766	54781	0.97	0.93	1.15
	64130	63468	66625	52502	1.01	0.96	1.22
	65636	65277	64144	59127	1.01	1.02	1.11
				Average	1.00	0.97	1.16
				R.S.D.%	2.32	4.72	4.80
				S.D.	0.02	0.05	0.06
3	64687	67410	66610	57882	0.96	0.97	1.12
	65669	65788	65263	58717	1.00	1.01	1.12
	65821	67044	66464	57051	0.98	0.99	1.15
				Average	0.98	0.99	1.13
				R.S.D.%	2.04	2.02	1.72
				S.D.	0.02	0.02	0.02
4	79153	84736	83799	70558	0.93	0.94	1.12
	89352	81439	87855	75626	1.10	1.02	1.18
	66568	69403	71842	64801	0.96	0.93	1.03
				Average	1.00	0.96	1.11
				R.S.D.%	9.10	5.12	6.80
				S.D.	0.09	0.05	0.08
5	68340	69965	73893	70526	0.98	0.92	0.97
	76585	72210	77383	66121	1.06	0.99	1.16
	75190	76228	77252	75350	0.99	0.97	1.00
				Average	1.01	0.96	1.04
				R.S.D.%	4.32	3.76	9.79
				S.D.	0.04	0.04	0.10
6	78188	73563	80225	73098	1.06	0.97	1.07
	72161	74962	75817	70468	0.96	0.95	1.02
	68062	73919	72767	70394	0.92	0.94	0.97
				Average	0.98	0.95	1.02
				R.S.D.%	7.36	1.60	4.90
				S.D.	0.07	0.02	0.05

TABLE IV (continued)

Transfer time min.	Areas				Ratios		
	C_{12}	C_{13}	C_{14}	C_{16}	C_{12}/C_{13}	C_{12}/C_{14}	C_{12}/C_{16}
7	76369	77703	73558	70358	0.98	1.04	1.09
	76635	81764	80101	83400	0.94	0.96	0.92
	63709	64573	63171	60115	0.99	1.01	1.06
				Average	0.97	1.00	1.02
				R.S.D.%	2.73	4.03	8.87
				S.D.	0.03	0.04	0.09
8	65633	67625	72681	72941	0.97	0.90	0.94
	68291	64397	71247	69894	1.06	0.96	0.98
	79748	83612	83738	74216	0.95	0.95	1.07
				Average	0.99	0.94	1.00
				R.S.D.%	5.90	3.43	6.68
				S.D.	0.06	0.03	0.07

I—Adsorber temperature 220° II—Adsorber temperature 260°.

second adsorber and is partly lost or its composition is altered owing to greater loss of lighter components. Extent of the loss depends also on the second adsorber temperature: the lower the temperature the smaller the loss. It is therefore advantageous to allow a longer transfer time and maintain the second adsorber at a low temperature. This is particularly true for samples containing components with a wide range of volatility. As shown in Table IV for the test sample at two minute transfer time, the determined sample composition was close to the theoretical value with a slight bias against *n*-hexadecane. At five minutes the ratios almost perfectly match theoretical values and remain virtually unchanged up to eight minutes transfer time indicating that the light components did not break through the second trap and that the entire sample is quantitatively adsorbed and consequently desorbed from the second adsorber.

Applicability of the system has been demonstrated by chromatograms of head space samples of several oils and air samples. Since some of the samples were multicomponent mixtures, the separation capability of the system was confirmed. Chromatograms are presented in Figures 3 through 9.

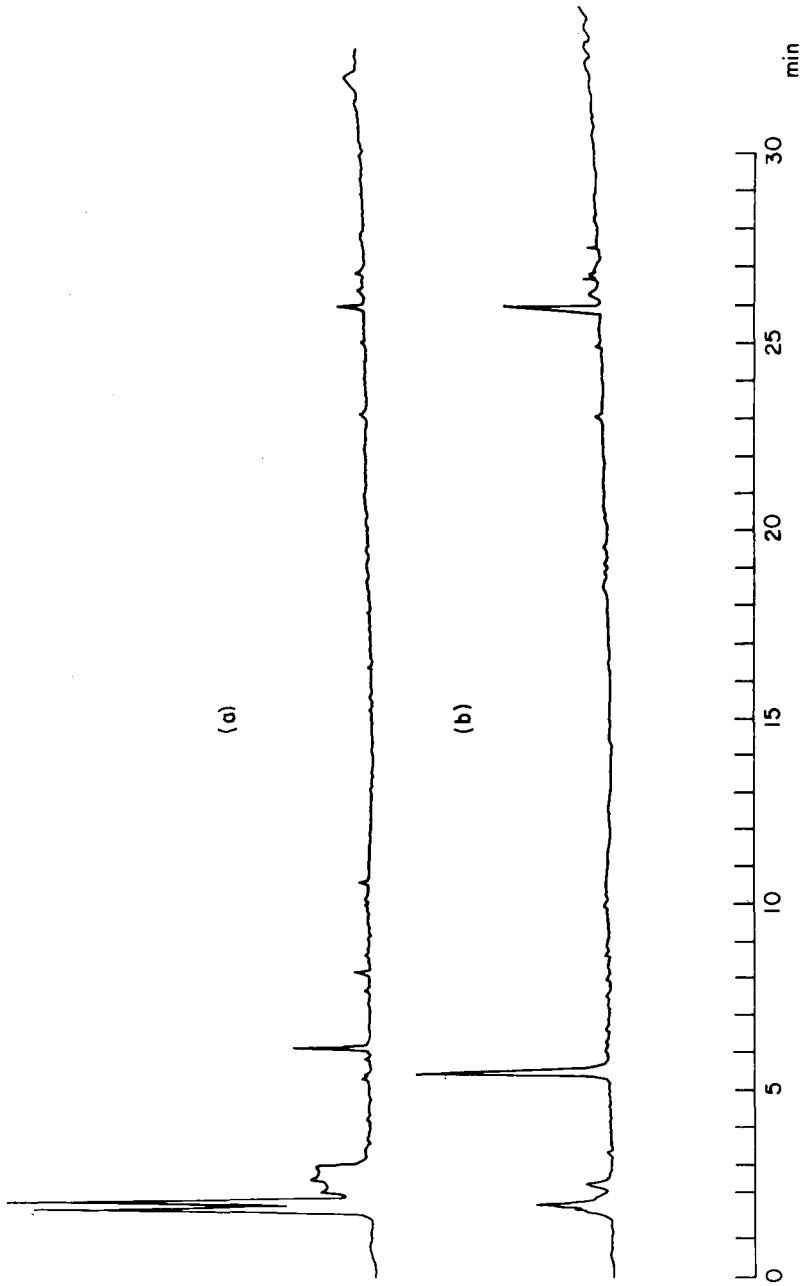

FIGURE 3 (a) Outside air, one liter sample, (b) laboratory air, one liter sample.

Figure 3 compares chromatograms of laboratory air (A) and air samples on the roof of the laboratory building (B). They are distinctly different. Laboratory air contains a significant amount of two components, probably hexane and acetone used as solvents in the laboratory. These components are much less abundant in outside air. Each sample contains a characteristic component: in outside air it is a compound with a retention time of 5.5 minutes, and in laboratory air a compound with a retention time of 6 minutes. On each chromatogram a heavy component appears with a retention time of about 26 minutes. The concentration of these components might be roughly estimated as 10–50 parts in 10^9.

At the time of conducting these experiments there was considerable interest in the identification of chemicals emitted by urea-formaldehyde foam insulation. The dual trap-capillary column system seemed well suited to sample and separate a complicated mixture of compounds emitted by the foam. Figure 4 presents a chromatogram of a 100 liter sample of air passed through urea-formaldehyde foam maintained at room temperature. It is difficult to

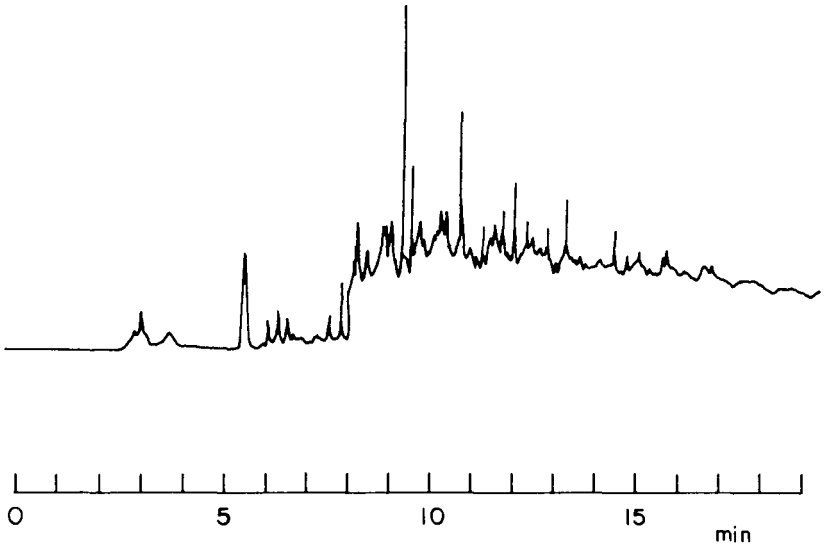

FIGURE 4 Urea-formaldehyde foam effluents, 100 liter sample (250 min sampling at 400 ml/min), flow through the foam—one liter per minute.

estimate the concentration of the components because their breakthrough volumes are not known and consequently trapping efficiency is also unknown. Nonetheless, the separation is adequate enough to attempt identification of the components by mass spectrometry or other methods.

As demonstrated by chromatograms presented in Figures 5 to 9, the system is well suited for finger-printing complicated mixtures by its ability to trap and analyse a wide spectrum of compounds.

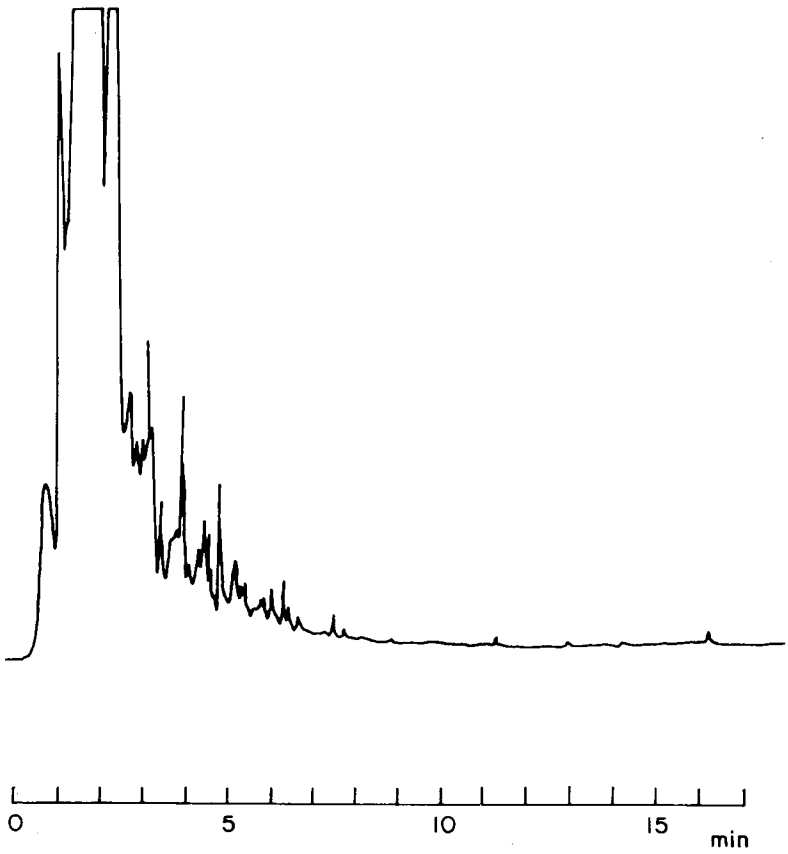

FIGURE 5 Duo seal oil head space, one liter sample.

The head space of Duo-Seal oil (Figure 5), for example, contains large amounts of low molecular weight hydrocarbons, which is not particularly advantageous since the oil is specially formulated for use in vacuum pumps. The head space of 585 oil (fuel oil) (Figure 6) consists of three main components of relatively low molecular weight.

Figure 7 offers an example of mixture identification based on head space analysis. Chromatogram A represents head space analysis of Tellus 33 oil and chromatogram B represents Tellus 69. These are

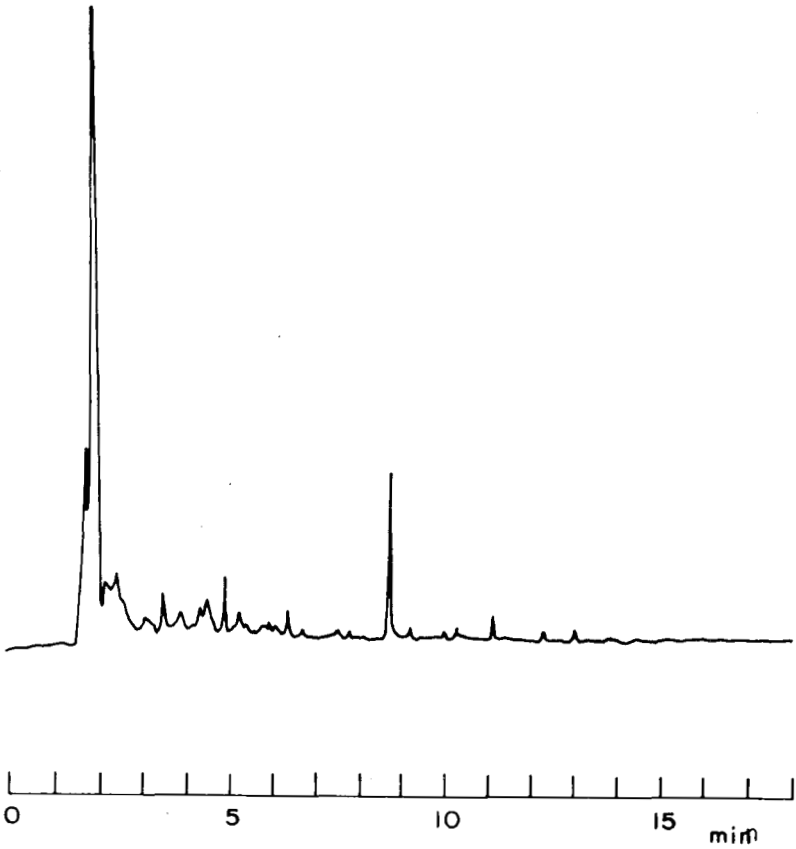

FIGURE 6 585 fuel oil head space, one liter sample.

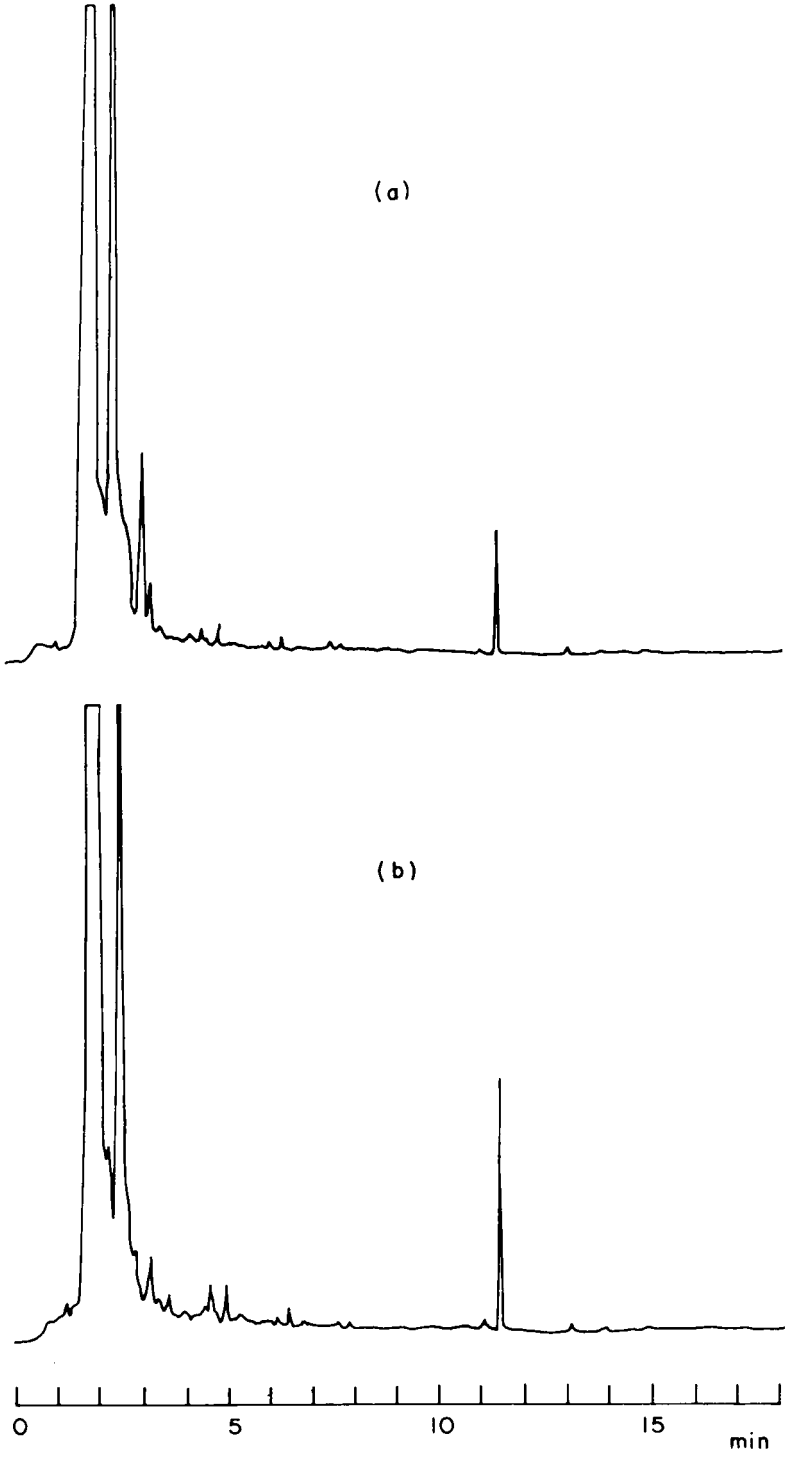

FIGURE 7 (a) Tellus 33 head space, one liter sample, (b) Tellus 69 head space, one liter sample.

very similar chromatograms; however, the ratio of areas of a peak with a retention time of about 3 minutes (peak 1) and a peak with a retention time of about 11.5 minutes (peak 2) differ significantly in each chromatogram. The ratio can thus be used as a basis for identification of the oil.

Figure 8 shows a chromatogram of an air sample collected from an empty drum which was used as a container for hydraulic oil. There are six peaks with retention times in the range of 1.5 to 3.5 minutes and one large peak with a retention time of about 11.5

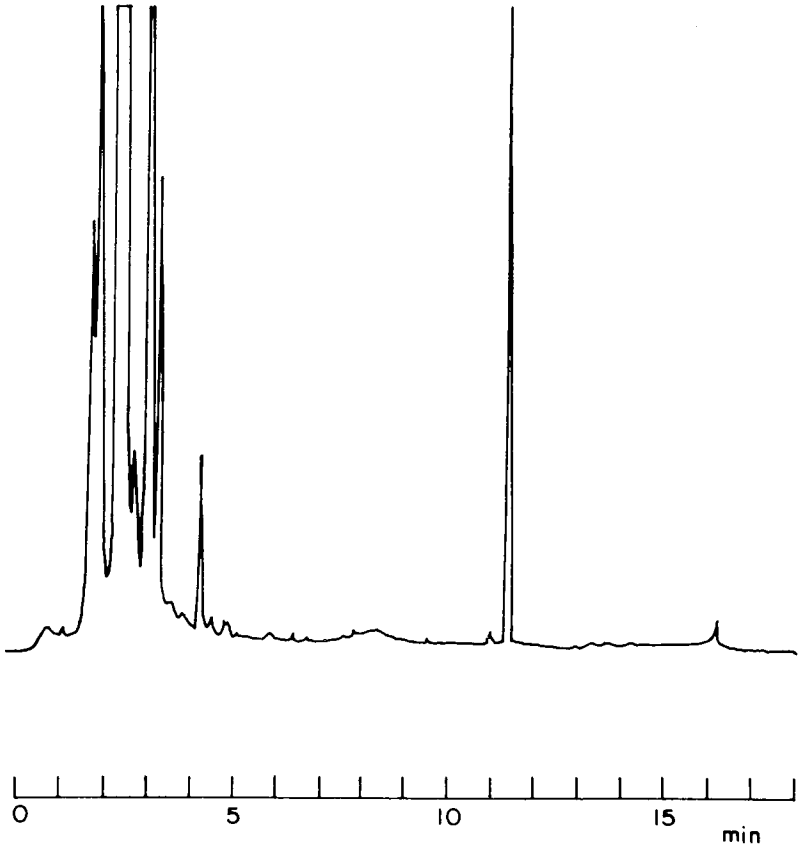

FIGURE 8 Vapour from an empty oil drum, one liter sample.

minutes. The chromatogram is somewhat similar to the chromatograms of Tellus oils (Figure 7), but there is a distinct difference in the size of the peak at 11.5 minutes in Figure 8 and the peak at 11.5 minutes on Figure 7. Tellus oils also contain fewer light hydrocarbons.

Figure 9 illustrates how the composition of a liquid differs from the composition of its vapour. Chromatogram A represents vapour

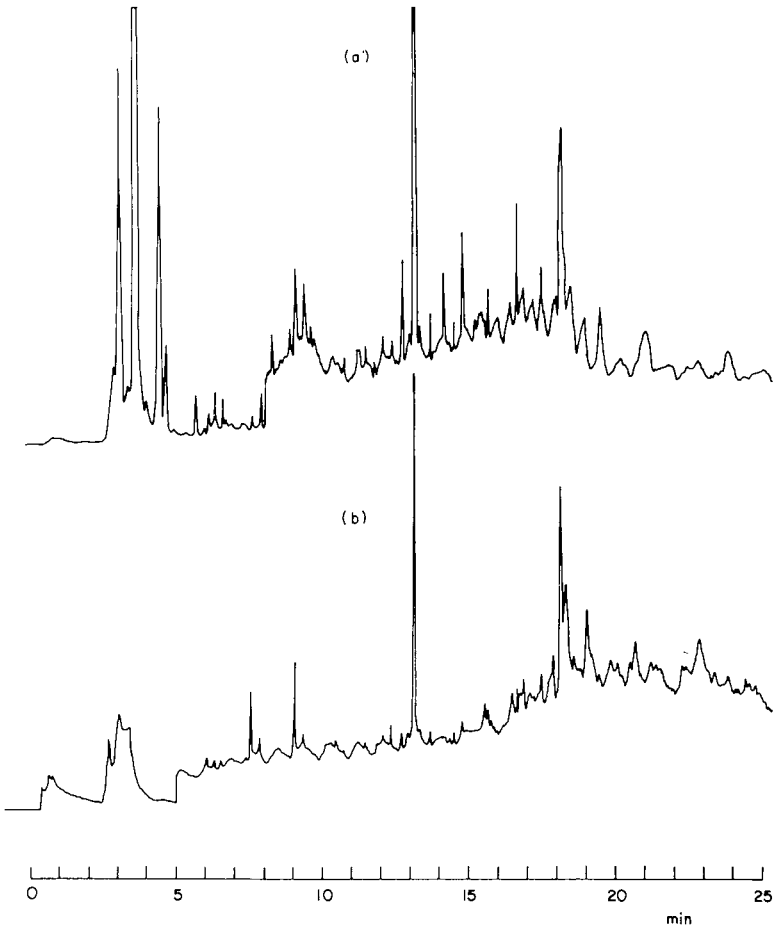

FIGURE 9 (a) Hydroflo 68 head space, one liter sample, (b) Hydroflo 68, 5 μl of CS_2 solution injected on second adsorber.

from Hydroflo 68 oil and chromatogram B represents a solution of the same oil. The head space vapours contain a much higher concentration of light components owing to their greater volatility.

The above observations are presented here to demonstrate that the investigated dual trap-capillary column system is capable of recording the characteristic phenomena, thus it does not alter the composition of a sample and is therefore a useful analytical tool.

The system is applicable to any analysis where trapping of an air sample followed by high-resolution gas chromatography is required.

Acknowledgement

The author sincerely thanks Dr. Lorne Elias for helpful discussions during the preparation of this paper.

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