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RETENTION BEHAVIOR OF CHLORINATED BENZENES, CHLORINATED PHENOLS, ALDRIN, AND AROCLOR 1242 ON TENAX-GC, CHROMOSORB 101, FLORISIL, AND CARBOPACK C HT

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SUMMARY

Four sorbent materials, Tenax-GC, Chromosorb 101, Florisil, and Carbopack C HT were characterized using elution analysis by gas chromatography for chlorinated benzenes, chlorinated phenols, Aldrin and Aroclor 1242. Plots of log V_n *versus* $°K^{-1}$ were linear with correlation coefficients of 0.992–1.000. Chlorinated phenols, Aldrin and Aroclor 1242 were not eluted on a Florisil column at temperatures as high as 325–375°C. Specific retention volumes, which were calculated for 20°C using results from linear regression analysis, generally followed the order: Tenax-GC > Chromosorb 101 > Carbopack C HT. At 250°C, measured specific retention volumes generally followed the order: Chromosorb 101 > Carbopack C HT > Tenax-GC. These results show that analytical procedures based on thermal desorption techniques may be developed for the determination of these chlorinated organic compounds. Tenax-GC is the best sorbent material based on retention properties.

INTRODUCTION

Various chlorinated organic compounds including pesticides, chlorinated phenols and polychlorinated biphenyls (PCBs) have been released into the atmosphere globally[1,3]. As chlorinated aromatic compounds are relatively unreactive and biologically non-degradable, these toxic compounds may remain in the environment for weeks or months and may bioaccumulate in fish and animals[4–6]. Furthermore, such compounds may migrate in aerosol or vapor phase over extended distances and may be deposited by natural processes such as rain, snow or gravity[7,8].

Numerous analytical methods are available for the determination of organic compounds in the vapor phase; however, one technique which has grown in popularity is sorbent trapping. In this technique vapor samples are passed through a tube which contains sorbent material on which the organic compounds are retained. As atmospheric gases are not retained, enrichment factors of 100–10,000 may be obtained, if breakthrough volumes for the components are not exceeded. Organic compounds

may later be removed using solvent stripping or thermal desorption for identification and quantitation by gas chromatography (GC) and GC–mass spectrometry (MS).

Although these techniques have been successfully applied in the analysis of different vapor samples for many types of organic compound[9-15], the suitability of sorbent collection with thermal desorption has not been determined in vapor-phase analysis of air samples for the higher molecular weight chlorinated organic compounds. In this paper, retention behavior is examined for selected chlorinated organic compounds on four commercially available sorbent materials using elution analysis by GC[16,17]. Results from these studies may be used in the design and operation of sorbent collection/thermal desorption techniques for the determination of these compounds in vapor-phase samples.

EXPERIMENTAL

Chemicals

All chemicals, sorbent materials and solvents were used as received from the manufacturer or supplier. Sorbent materials included: Tenax-GC from Applied Science Labs., State College, PA, U.S.A.; Chromosorb 101 from Johns-Manville, Denver, CO, U.S.A.; Carbopack C HT from Supelco, Bellefonte, PA, U.S.A.; and Florisil from Applied Science Labs. Chlorinated organic compounds were: monochlorobenzenes and *p*-dichlorobenzene from Fisher Scientific, Fair Lawn, NJ, U.S.A.; *m*-chlorophenol and 1,2,4-trichlorobenzene from Eastman Organic Chemicals, Rochester, NY, U.S.A.; 2,4,6-trichlorophenol from Matheson Coleman and Bell, East Rutherford, NJ, U.S.A.; Aldrin from Varian, Palo Alto, CA, U.S.A.; and Aroclor 1242 from Monsanto, Rexdale, Canada. The solvent was distilled in glass-grade methylene chloride from Burdick and Jackson, Muskegon, MI, U.S.A.

Gas chromatograph

A Hewlett-Packard Model 5830A gas chromatograph was equipped with flame ionization detectors (FIDs). Isothermal conditions were used with column temperatures set at 25°C intervals between 75 and 375°C. Other conditions were: injection port temperature, 250°C; FID temperature, 275°C; slope sensitivity, 0.1 mV/min; area reject, 1000; attenuation, 32 or 64; carrier gas, helium; carrier gas flow-rate, 29 or 30 ml/min; and injection volume 0.8–1.5 μl. Septums (HT-9, Applied Science Labs.) which are stable at temperatures between 250 and 275°C were replaced after every 6–10 injections.

Preparation of columns

Known amounts of sorbent materials were packed inside 30-cm sections of 3 mm O.D. × 2 mm I.D. nickel tubing using occasional tapping or vibration. Exact weights of sorbent materials which were used in the columns were: Tenax-GC, 0.2118 g; Chromosorb 101, 0.3515 g; Carbopack C HT, 0.8527 g; and Florisil, 0.5839 g. Columns were conditioned in the chromatograph with helium carrier gas and temperatures between 250 and 375°C until bleed levels became constant and below 10% full scale at attenuation 64. Maximum temperatures which were used with each sorbent were: Tenax-GC, 250°C; Chromosorb 101, 275°C, Carbopack C HT, 300°C; and Florisil, 375°C.

Preparation of solutions

Standard solutions of each compound were prepared at concentration levels near 500 to 1000 ng/μl in methylene chloride solvent. Solutions were prepared by weighing exact amounts or measuring known volumes of each compound into 400–600-μl reacti-vials (Chromatographic Specialties, Brockville, Canada. Additional dilutions were made when necessary and final concentrations were: monochlorobenzene, 370 ng/μl; *p*-dichlorobenzene, 420 ng/μl; *m*-chlorophenol, 460 ng/μl; 1,2,4-trichlorobenzene, 480 ng/μl; 2,4,6-trichlorophenol, 1800 ng/μl; Aldrin, 1100 ng/μl; and Aroclor 1242, 1 μl in 500 μl.

Determination of retention volumes

Measurements were made and calculations based on descriptions by Dal Nogare and Juvet[16], and Vidal-Madjar *et al.*[17]. Constant volumes of solutions (1 μl) were injected at each temperature with a Model 701 N microliter syringe (Hamilton, Reno, NV, U.S.A.), and inlet pressure measurements were made with a needle gauge (Chromatographic Specialties), which was inserted through the septum. At temperatures between 75 and 375°C, methylene chloride was not noticeably retained on any sorbent column and was used to calculate void volumes. Void volumes never exceeded 1.5 ml.

The adjusted retention volume, V_R', of a solute in a column is the total retention volume, V_R, which has been corrected for the void volume, V_a:

$$V_R' = V_R - V_a$$

When expressed in terms of the carrier gas flow-rate, F_c, in the column, the retention time of the unretained solute, t_a, and the retention time of the solute, t_R, this gives

$$V_R' = (t_R - t_a) F_c$$

The net retention volume, V_n, is corrected for the effect of gas compressibility by a factor j

$$V_n = jV_R' = j(t_R - t_a) F_c$$

where

$$j = 1.5 \frac{(p_i/p_o)^2 - 1}{(p_i/p_o)^3 - 1}$$

and P_i is the pressure at the column inlet and P_o is the pressure at the column outlet. The flow-rate was measured with a soap-bubble flow meter and corrected for vapor pressure of water and column temperature:

$$F_c = F_a (T_c/T_a) [1 - (p_w/p_a)]$$

where T_c is the column temperature in °K; T_a is the ambient temperature in °K; p_w is the vapor pressure of water at 25°C; p_a is the atmospheric pressure; and F_a is the measured flow-rate at the column outlet.

RESULTS AND DISCUSSION

Results from the characterization of retention properties using GC elution analysis for four sorbent materials with selected chlorinated benzenes, chlorinated phenols, and Aldrin are given in Table I and Figs. 1–4. In Table I values for log V_n are listed for each compound with each sorbent material at several temperatures between 100 and 375°C. Plots of log V_n *versus* °K^{-1} are shown in Figs. 1–4, and linear regression analyses of these plots show curves are linear with correlation coefficients (r) between 0.992 and 1.000. Values for the slopes, r^2 values, and y-intercepts for each figure are listed in Table II. The slope in these plots may be related to the differential heat of adsorption $\Delta \bar{H}_A$, as in eqn. 1:

$$\Delta \bar{H}_A = -2.303R \frac{\mathrm{dlog}\ V_n}{\mathrm{d}(°\mathrm{K})^{-1}} \tag{1}$$

TABLE I

VALUES FOR log V_n (ml) FROM GAS CHROMATOGRAPHIC ANALYSES

| Compound	Temperature (°C)											
	100	125	150	175	200	225	250	275	300	325	350	375
Sorbent: Tenax-GC												
Chlorobenzene	3.01	2.46	1.88	1.34	0.84	0	0	—	—			
p-Dichlorobenzene	—	2.73	2.10	1.57	1.13	0.70	0.32	—	—			
m-Chlorophenol	—	—	2.88	2.25	1.66	1.27	0.90	—	—			
1,2,4-Trichlorobenzene	—	—	3.05	2.38	2.01	1.58	1.05	—	—			
2,4,6-Trichlorophenol	—	—	—	2.72	2.19	1.63	1.19	—	—			
Aldrin	—	—	2.88	2.24	1.75	1.34	1.13	—	—			
Sorbent: Chromosorb 101												
Chlorobenzene	2.91	2.33	1.89	1.44	1.11	0.83	0.58	—	—			
p-Dichlorobenzene	—	2.92	2.36	1.90	1.52	1.19	0.92	—	—			
m-Chlorophenol	—	—	2.93	2.39	1.95	1.57	1.27	0.99	—			
1,2,4-Trichlorobenzene	—	—	2.86	2.34	1.91	1.54	1.22	0.96	—			
2,4,6-Trichlorophenol	—	—	—	2.84	2.35	1.92	1.57	1.26	—			
Aldrin	—	—	—	—	—	3.25	2.75	2.31	—			
Sorbent: Carbopack CHT												
Chlorobenzene	2.05	1.63	1.28	0.94	0.68	0	0	—	—			
p-Dichlorobenzene	2.98	2.48	2.05	1.66	1.33	1.03	0.76	—	—			
m-Chlorophenol	—	2.54	2.00	1.57	1.21	0.90	0.67	—	—			
1,2,4-Trichlorobenzene	—	—	2.78	2.33	1.94	1.57	1.28	1.00	—			
2,4,6-Trichlorophenol	—	—	—	2.76	2.34	1.96	1.61	1.33	1.02			
Aldrin	—	—	—	—	2.66	2.21	1.83	1.54	1.26			
Sorbent: Florisil												
Chlorobenzene					2.63	2.07	1.65	1.27	1.09	—	—	—
p-Dichlorobenzene					2.84	2.44	1.97	1.68	1.45	—	—	—
m-Chlorophenol					—	—	—	*	*	*	*	*
1,2,4-Trichlorobenzene					—	2.70	2.25	1.84	1.59	—	—	—
2,4,6-Trichlorophenol					—	—	—	*	*	*	—	—
Aldrin					—	—	—	*	*	*	*	*

* No elution peak detected after injection of 1000 ng.
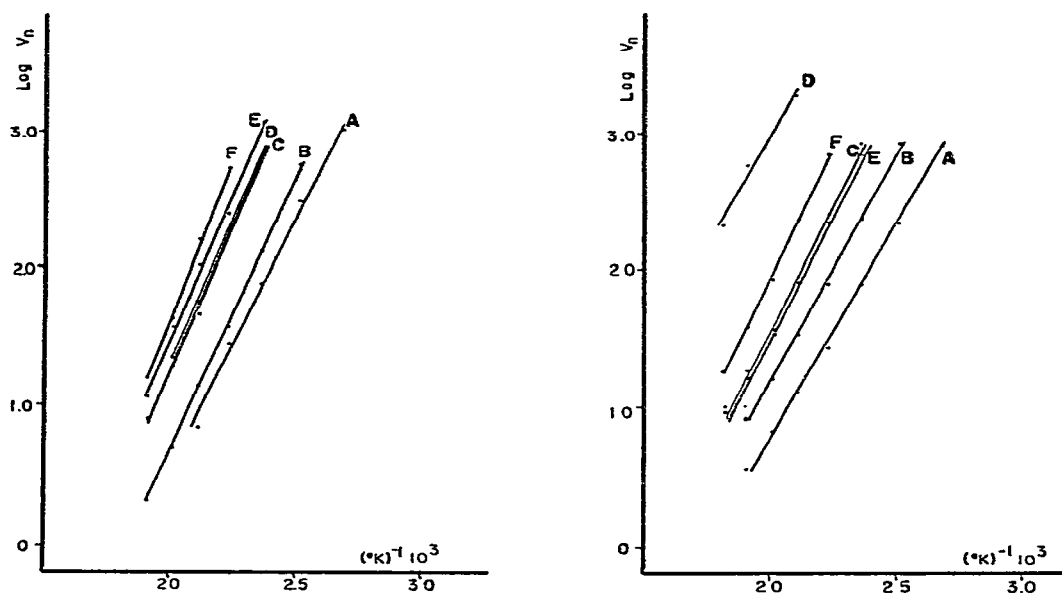
Fig. 1. Plot of log V_n versus $°K^{-1} \times 10^3$ for Tenax-GC. Compounds: A = chlorobenzene; B = p-dichlorobenzene; C = m-chlorophenol; D = Aldrin; E = 1,2,4-trichlorobenzene; F = 2,4,6-trichlorophenol.

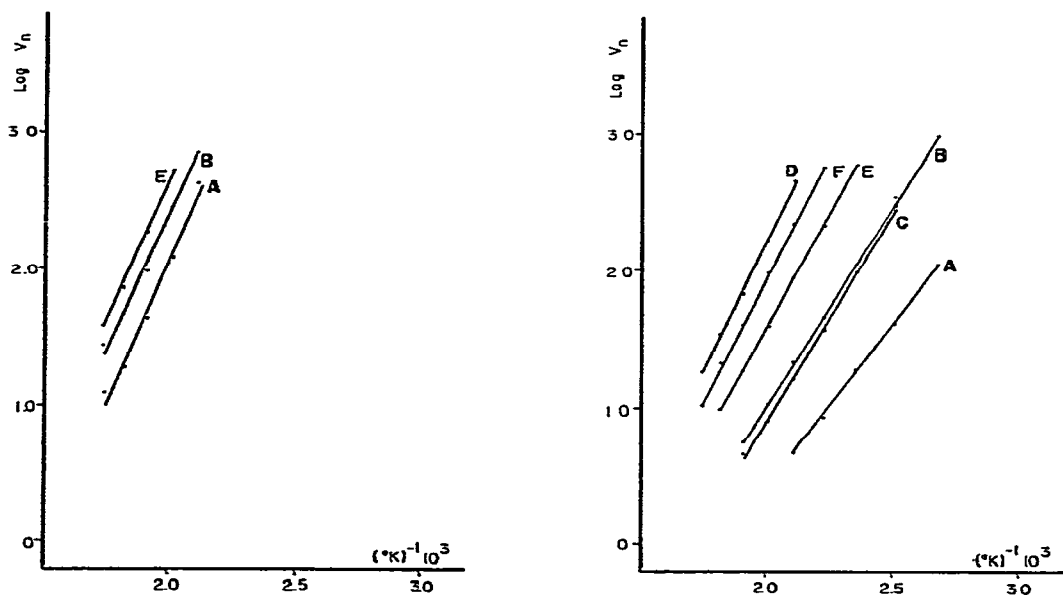Fig. 2. Plot of log V_n versus $°K^{-1} \times 10^3$ for Chromosorb 101. For identification of curves see Fig. 1.

Fig. 3. Plot of log V_n versus $°K^{-1} \times 10^3$ for Florisil. For identification of curves see Fig. 1.

Fig. 4. Plot of log V_n versus $°K^{-1} \times 10^3$ for Carbopack C HT. For identification of curves see Fig. 1.

TABLE II

RESULTS OF LINEAR* REGRESSION ANALYSIS FOR log V_n (ml) *versus* ($°K^{-1} \times 10^3$)

	n^{**}	Slope	y-intercept	rI
Tenax				
Chlorobenzene	5	3.8	−7.2	0.997
p-Dichlorobenzene	6	4.0	−7.3	0.999
m-Chlorophenol	5	4.4	−7.6	0.996
1,2,4-Trichlorobenzene	5	4.3	−7.1	0.993
2,4,6-Trichlorophenol	4	4.9	−8.1	0.998
Aldrin	5	4.0	−6.5	0.985
Chromosorb 101				
Chlorobenzene	7	3.0	−5.3	0.998
p-Dichlorobenzene	6	3.3	−5.5	0.998
m-Chlorophenol	6	3.6	−5.6	0.996
1,2,4-Trichlorobenzene	6	3.5	−5.5	0.997
2,4,6-Trichlorophenol	5	3.9	−5.8	0.998
Aldrin	3	5.0	−6.7	1.000
Carbopack CHT				
Chlorobenzene	5	2.4	−4.4	1.000
p-Dichlorobenzene	7	2.9	−4.8	1.000
m-Chlorophenol	6	3.1	−5.4	0.996
1,2,4-Trichlorobenzene	6	3.3	−5.0	1.000
2,4,6-Trichlorophenol	6	3.6	−5.2	0.999
Aldrin	5	3.8	−5.4	0.997
Florisil				
Chlorobenzene	4	3.8	−5.7	0.992
p-Dichlorobenzene	5	3.9	−5.4	0.995
1,2,4-Trichlorobenzene	4	4.3	−5.0	0.998

.* Plots were fitted to equation $y = mx + b$, where m is the slope, b is the y-intercept, and r is the correlation coefficient for a least squares fit.
** n is the number of data points.

where R is the universal gas constant. Inspection of Figs. 1–4 reveals that slopes, absolute positions and relative placement of curves are different in each figure. However, certain aspects of the retention behavior of these compounds on the different sorbents are similar. One aspect is the elution order, which may be roughly correlated to boiling points and which generally follows this trend: chlorobenzene, p-dichlorobenzene, m-chlorophenol, 1,2,4-trichlorobenzene, 2,4,6-trichlorophenol. Other aspects of retention behavior are treated individually for each sorbent material.

Tenax-GC

The plots in Fig. 1 fall into two closely gathered clusters of curves. The first and earliest eluting set consists of chlorobenzene and p-dichlorobenzene, while the second set comprises the remaining compounds. The specific retention volumes (l/g) for these compounds on the four sorbents with column diameters of 2 mm, at 20°C, and average linear velocities of 10–25 cm/sec, were calculated through extrapolation using results from linear regression analysis. These values (Table III) were larger on Tenax-GC than those on Chromosorb 101 and Carbopack C HT but less than retention volumes on Florisil. The differential heats of adsorption (Table IV) also

TABLE III

PREDICTED VALUES FOR SPECIFIC RETENTION VOLUME (l/g) OF CHLORINATED ORGANIC COMPOUNDS ON SORBENTS AT 20°C

	Tenax	Chromosorb 101	Carbopack C HT	Florisil
Chlorobenzene	$2.4 \cdot 10^3$	$2.3 \cdot 10^2$	$6.7 \cdot 10^1$	$2.8 \cdot 10^4$
p-Dichlorobenzene	$9.4 \cdot 10^3$	$1.5 \cdot 10^3$	$1.3 \cdot 10^2$	$1.2 \cdot 10^5$
m-Chlorophenol	$1.1 \cdot 10^5$	$1.2 \cdot 10^4$	$1.6 \cdot 10^2$	*
1,2,4-Trichlorobenzene	$1.6 \cdot 10^5$	$7.1 \cdot 10^3$	$1.9 \cdot 10^3$	$7.1 \cdot 10^5$
2,4,6-Trichlorophenol	$1.7 \cdot 10^6$	$8.2 \cdot 10^4$	$1.3 \cdot 10^4$	*
Aldrin	$5.9 \cdot 10^4$	$5.7 \cdot 10^7$	$3.9 \cdot 10^4$	*

* Compounds not eluted under analytical conditions employed.

TABLE IV

DIFFERENTIAL HEATS OF ADSORPTION (kcal/mol)

	Tenax	Chromosorb 101	Carbopack CHT	Florisil
Chlorobenzene	17	14	11	17
p-Dichlorobenzene	18	21	13	18
m-Chlorophenol	20	17	14	*
1,2,4-Trichlorobenzene	20	16	15	20
2,4,6-Trichlorophenol	22	18	16	*
Aldrin	18	23	17	*

* Compounds not eluted under analytical conditions employed.

show highest values with Tenax-GC in each case, except p-dichlorobenzene and Aldrin with Chromosorb 101. Additionally the placement and slopes for the plots show large retention volumes for these compounds on Tenax-GC at 20°C, but low retention volumes at 250–300°C. Such properties make Tenax-GC an excellent material for sorbent collection and later thermal desorption of these and similar chlorinated organic compounds in vapor-phase samples.

Because all plots have been corrected to 760 mmHg, retention volumes may not be directly extrapolated for thermal desorption techniques where pressures are 2000 to 3000 mmHg[17]. Other studies[15] have shown that retention properties of Tenax-GC are not greatly influenced by moisture. However, Tenax-GC is not completely inert chemically and may degrade under certain conditions[18]. Also, Tenax-GC has a lower capacity than many porous polymers[14].

Chromosorb 101

Results of elution analysis are shown in Fig. 2 for Chromosorb 101. These plots show that the retention behavior of chlorinated benzenes and phenols was similar to that on Tenax-GC but specific retention volumes at 20°C were lower by factors of 6–22. The plot for Aldrin was displaced well above all the other plots, including that for 2,4,6-trichlorophenol, and the specific retention volume was nearly 1000 times larger than on Tenax-GC. Generally the strong adsorption of these compounds by both Chromosorb 101 and Tenax-GC is not surprising as these material share a similar chemical structure which is highly aromatic in character. This observation is supported by the values for $\Delta \bar{H}_A$ (Table IV). Although $\bar{H}_A$ values on Tenax-GC are

greater for most compounds, both sorbent materials have greater values than Carbopack C HT, where little or no π–π interactions may be expected in sorbent–solute attractions. In sampling procedures with Chromosorb 101 the high capacity is an advantage[14] but the lengthy purification step which is required before determining organic compounds at ng/g concentration levels is a disadvantage.

However, poor recovery of Aldrin, trichlorophenols, and trichlorobenzenes may be expected with thermal desorption techniques at temperatures of 250–300°C and desorption times of 20 min or less. Recovery efficiency may also be decreased by undetermined losses to the column. Such losses were detected through quantitation of peak area from elution analysis with response factors for these compounds. Percentages of components which eluted through the sorbent columns were: for p-dichlorobenzene: Tenax-GC, 89%; Florisil, 51%; and Chromosorb 101, 35%; for 1,2,4-trichlorobenzene: Tenax-GC, 105%; Florisil, 72%; and Chromosorb 101, 43%; and for 2,4,6-trichlorophenol: Tenax-GC, 76%; Florisil, 0%, and Chromosorb 101, 41%. These values show an additional disadvantage in the use of Chromosorb 101 as material for sorbent traps.

Florisil

Results from the characterization of Florisil, which is a synthetic magnesium silicate activated at 650°C, are given in Table I and Fig. 3. For Aldrin and chlorinated phenols, which have a polar hydroxyl group, elution volumes were undetermined even at temperatures of 300–375°C. Elution times were monitored for only 40 min, as beyond this time excessive band broadening made identification of t_R difficult or impossible. Clearly Florisil is unsatisfactory for use in thermal desorption techniques with polar chlorinated organic compounds and poor recovery would be expected for chlorobenzene and polychlorinated benzenes. The $\Delta \bar{H}_A$ values of the chlorobenzenes are as large as those on Tenax-GC. Thus these solute–sorbent interactions are different from but as strong as π–π attractions.

Carbopack C HT

Results from characterization of Carbopack C HT, which is graphitized carbon heat-treated at 1000°C with hydrogen, are given in Table I and Fig. 4. Chlorinated organic compounds had the lowest specific retention volume on Carbopack C HT in comparison to other sorbents. Also the values for $\Delta \bar{H}_A$ were 1–6 kcal/mol lower than for other sorbent materials and thus forces of attraction between solute and sorbent were lower for Carbopack C HT. This conclusion is not unusual because the presumably more saturated carbon structure of Carbopack C HT would have fewer and weaker interactions with aromatic compounds than the mostly aromatic structure of Tenax-GC or Chromosorb 101. Although Carbopack C HT would be satisfactory with thermal desorption methods, early breakthough would be expected for these compounds during sampling because specific retention volumes are very low for chlorobenzene, p-dichlorobenzene and m-chlorophenol.

Results from elution analysis with Aroclor 1242

As Aroclor 1242 is a complex mixture of polychlorinated biphenyls[19], results from elution analysis using GC showed only partial resolution on the sorbent columns at various temperatures. These results are presented in Figs. 5–7 as plots of abundance
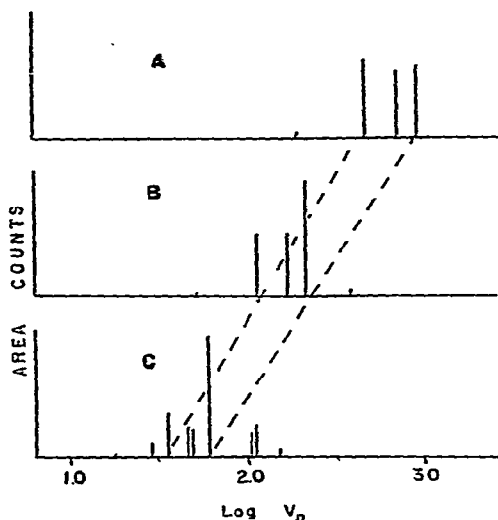
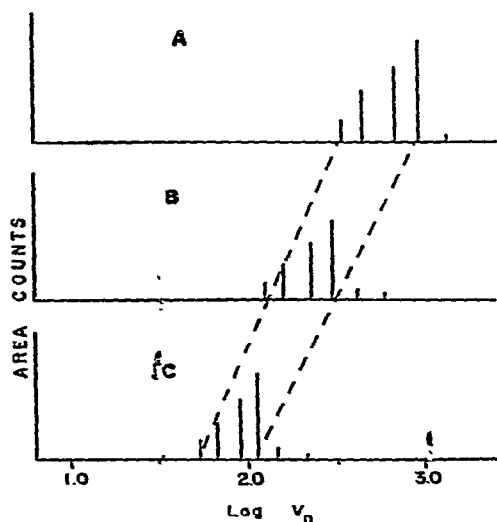Fig. 5. Plots of abundance *versus* log V_n of Aroclor 1242 on Tenax-GC. Temperatures (°C): A, 200; B, 225; C, 250.

Fig. 6. Plots of abundance *versus* log V_n of Aroclor 1242 on Chromosorb 101. Temperatures (°C): A, 225; B, 250; C, 275.

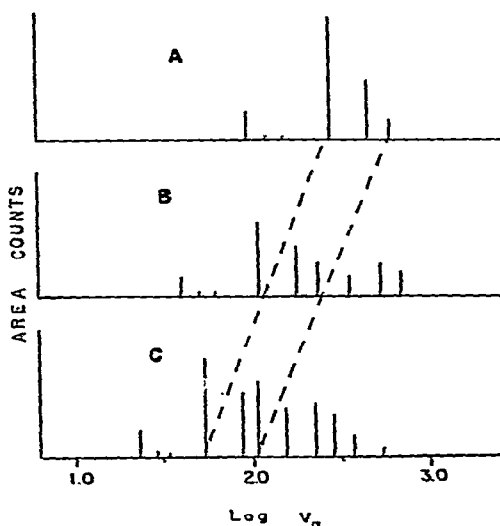Fig. 7. Plots of abundance *versus* log V_n of Aroclor 1242 on Carbopack C HT. Temperatures (°C): A, 200; B, 225; C, 250.

values from integration of chromatograms *versus* log V_n (ml). Although linear regression analysis was not applied to this data, the plots follow certain trends. Each sorbent material showed larger values for log V_n at lower temperatures. Furthermore, differences in log V_n between components in the mixtures are not increased or in-

creased only slightly at lower temperatures. Values for log V_n of the earliest eluting major component in Aroclor 1242 were higher on each sorbent material at 250°C than values of log V_n for 2,4,6-trichlorophenol. Differences in specific retention volumes between the first and last major components were: Carbopack C HT, 6.5 ml; Chromosorb 101, 56 ml; and Tenax-GC 3980 ml. The PCBs in Aroclor 1242 were not successfully eluted on Florisil. These results show that thermal desorption of sorbent traps which contain Tenax-GC, Chromosorb 101 and Carbopack C HT is a possible method for determining PCBs in vapor-phase samples. However, recovery efficiencies will be low unless desorption temperatures are greater than 250°C.

CONCLUSIONS

Results presented here demonstrate that chlorinated aromatic hydrocarbons may be successfully eluted from columns which contain Tenax-GC, Carbopack C HT and Chromosorb 101. Tenax-GC had the best retention properties for use in collection and thermal desorption of these compounds as specific retention volumes were large at ambient temperatures and small at temperatures of 250–300°C. Problems associated with the determination of these compounds using thermal desorption techniques may be reduced or eliminated if transfer lines are reduced in length and heated[20].

ACKNOWLEDGEMENT

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