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GAS CHROMATOGRAPHIC DATA FOR POLYCHLORINATED BIPHENYL COMPONENTS IN SIX AROCLORS®

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SUMMARY

Illustrations of gas chromatographic curves, relative retention times, and response data from 10% DC-200 and 1:1 15% QF-1/10% DC-200 on 80–100 mesh Chromosorb W HP columns with electron capture detection are compiled for six Aroclors®. Aroclors® are commercial mixtures of polychlorinated biphenyls commonly used as analytical references for polychlorinated biphenyl residue determination.

INTRODUCTION

The industrial chemicals known as polychlorinated biphenyls (PCB), which have become widespread environmental contaminants[1–9], are generally determined by essentially the same techniques used for organochlorine pesticides[4] and may act as interferences in the gas-liquid chromatographic (GLC) determination of pesticide residues. Because there is no standard PCB and individual chlorinated biphenyl compounds are not readily available, it is necessary to rely on Aroclors® as analytical references for PCB residue determinations.

Aroclor® is the general tradename for commercial mixtures of PCB manufactured in the United States by Monsanto Company. Each Aroclor is a mixture of chlorinated biphenyls (1200 series), chlorinated terphenyls (5400 series) or a combination of chlorinated biphenyls and terphenyls (4400 series). The last two digits of the identifying number indicate the percentage weight of chlorine, *e.g.*, Aroclors 1254 and 1260 are biphenyls containing 54 and 60% chlorine, respectively[4]. GLC patterns of PCB residues in environmental samples have generally resembled Aroclors 1254 and 1260, although the possibility exists that residues may derive from any of the Aroclors.

The GLC retention times, relative peak sizes, peak shapes, and overall peak pattern of PCB residues must be carefully compared to the corresponding data for the Aroclors in order to determine the Aroclor(s) that most closely resemble the residue. Because the magnitude of total area varies significantly for the same weight of different Aroclors, the quantitative value determined for the PCB residue depends

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upon which Aroclor(s) is chosen for quantitation reference. The interpretation and evaluation of GLC chromatograms in PCB determinations is augmented by ready access to relative retention times, response data, and reference chromatograms of the various Aroclors.

Data for these GLC characteristics of six Aroclors, obtained by using two GLC columns regularly employed in our laboratories[8], are presented here. Data are not presented for Aroclors 1232, 1268, 5442, 5460, and 4465. Aroclor 1232 has been commercially prepared[7] by blending appropriate quantities of Aroclors 1221 and 1242 to obtain 32% total chlorine; chromatograms of material obtained from different lots revealed substantial differences between some lots. Aroclor 1268 was not available at the time of this work. GLC at the described conditions of Aroclors 5442 and 5460 (chlorinated terphenyls) and 4465 (mixture of PCB and chlorinated terphenyls) resulted in multicomponent chromatograms with peaks emerging at retention times extending to several hours; several hundred nanograms were required for detection of late-eluting constituents. These GLC parameters are considered unsuitable for detection and measurement of the polychlorinated terphenyls. A system utilizing parallel columns with liquid phases of 1% OV-101 and 3% Dexsil 300 operated at 240° has been devised for GLC of both the polychlorinated terphenyls and PCB[8].

EXPERIMENTAL

GLC data were obtained with a gas chromatograph equipped with an electron capture detector and 4-mm × 6-ft. glass columns, packed both with 10% DC-200 on 80–100 mesh Chromosorb W HP and a 1:1 mixture of 15% QF-1 plus 10% DC-200 on 80–100 mesh Chromosorb W HP[9]. Operating conditions: nitrogen, 120 ml/min; column and detector temperature, 200°; injection temperature, 225°. The concentric design electron capture detector was operated at a d.c. voltage to produce half full scale recorder deflection for 1 ng heptachlor epoxide when full scale deflection is 1×10^{-9} A. Recorder speed was $\frac{1}{4}$ in./min.

Different concentrations of Aroclors were injected at the conditions given to determine the quantity necessary for approximately half full scale recorder response or enough response to illustrate all the characteristic constituents of a particular Aroclor. Retention times were measured in millimeters from the leading edge of the response to the solvent and reported relative to the retention time of the pesticide aldrin. These conditions and manner of reporting GLC data for Aroclors were chosen to conform to GLC data compiled in the Food and Drug Administration *Pesticide Analytical Manual*[6] for a large number of pesticides.

RESULTS

The following information has been compiled in the tables and figures to be a source of reference and comparison for the GLC behavior of PCB: retention times, response data and illustrations of chromatograms for six Aroclors from two GLC columns. Table I lists the quantities necessary for approximately half full scale

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TABLE I

QUANTITIES OF AR

Aroclor	Nanog	
	DC-20	
1221	80	
1242	40	
1248	40	
1254	30	
1260	20	
1262	20	

[a] Quantity (1/2 f.s.r.) for in Aroclor constitu
[b] GLC co

TABLE II

GLC RETENTION

Support materia mm I.D. columr ture (Tritium); s Table I); data g

Aroclor 1221	Aro 124
0.24	
0.26	
0.31	
0.36	
0.49	0.3
0.52	0.5
0.59	0.5
0.63	
0.69	0.6
0.75	0.7
0.88	0.8
0.99	0.9
	1.0
1.27	1.2
1.42	1.3
1.52	1.4
1.76	1.7
1.86	1.8
2.08	
2.22	2.2
	2.5
2.63	
3.10	

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TABLE I

QUANTITIES OF AROCLORS NECESSARY FOR APPROXIMATELY HALF FULL SCALE RECORDER RESPONSE[a]

Aroclor	Nanograms for 1/2 f.s.r.	
	DC-200[b]	QF-1/DC-200[b]
1221	80	60
1242	40	40
1248	40	30
1254	30	30
1260	20	20
1262	20	20

[a] Quantities of Aroclor mixtures to produce approximately half full scale recorder response (1/2 f.s.r.) for major Aroclor components or sufficient response to detect all the characteristic Aroclor constituents.

[b] GLC column and detector conditions are given under EXPERIMENTAL.

TABLE II

GLC RETENTION TIME DATA FOR SIX AROCLORS

Support material: Chromosorb W HP, 80–100 mesh; stationary phase: 10% DC-200 (6 ft. × 4 mm I.D. column); carrier gas: nitrogen, 120 ml/min; temperature: 200°; detector: electron capture (tritium); sample size: quantities necessary for approximately half full scale response (see Table I); data given in: retention sequence relative to aldrin from solvent peak.

Aroclor 1221	Aroclor 1242	Aroclor 1248	Aroclor 1254	Aroclor 1260	Aroclor 1262
0.21					
0.26					
0.31					
0.36					
0.39	0.39				
0.52	0.51	0.51			
0.59	0.57	0.57			
0.64					
0.69	0.67	0.67			
0.75	0.72				
		0.80			
0.88	0.86	0.85	0.87		
0.99	0.96	0.96	0.98		
	1.03	1.03	1.05		
1.27	1.22	1.23	1.27	1.28	1.26
1.42	1.39	1.39			
1.52	1.49	1.49	1.52	1.50	1.50
1.76	1.74	1.74	1.78		
1.86	1.83	1.84	1.88	1.86	1.85
2.08				2.07	2.07
2.22	2.20	2.20	2.20	2.21	2.22
	2.56	2.54			
2.65			2.63	2.63	2.61
				2.84	2.82
3.10		3.04	3.08	3.08	3.06
			3.60	3.50	3.50
			4.10	4.10	4.10
			4.30		
			4.90	4.90	4.90
			5.80	5.80	5.80
					6.40
				6.50	6.60
				7.80	7.80
				9.10	9.10

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recorder response or for enough response to illustrate all the characteristic constituents of a particular Aroclor. Tables II and III give the retention times relative to aldrin for all the peaks present in six different Aroclors. These tables are arranged to readily show similarity and differences in the GLC elution pattern of the various Aroclors. Peaks with similar relative retention times in the different Aroclors are not necessarily responses to the same compound. Figs. 1–3 are gas chromatograms of six Aroclors from the 10% DC-200 column; Figs. 4–6 are gas chromatograms of six Aroclors from the 1:1 15% QF-1/10% DC-200 column at the specified conditions.

The GLC data for PCB presented here should be a useful reference for evaluation and interpretation of GLC chromatograms for PCB residue determinations.

TABLE III

GLC RETENTION TIME DATA FOR SIX AROCLORS

Support material: Chromosorb W HP, 80–100 mesh; stationary phase: 1:1 15% QF-1/10% DC-200 (6 ft. × 4 mm I.D. column); carrier gas: nitrogen, 120 ml/min; temperature: 200°; detector: electron capture (tritium); sample size: quantities necessary for approximately half full scale recorder response (see Table I); data given in: retention sequence relative to aldrin from solvent peak.

Aroclor 1221	*Aroclor 1242*	*Aroclor 1248*	*Aroclor 1254*	*Aroclor 1260*	*Aroclor 1262*
0.20					
0.29					
0.36					
0.39	0.41				
0.53	0.53	0.53			
0.57	0.61	0.61			
0.71	0.72	0.72			
0.78					
	0.81	0.81			
0.88	0.90	0.90	0.87		
1.02	1.02	1.02	1.02		
1.10	1.09	1.09			
1.31	1.34	1.34	1.32	1.31	1.31
1.53	1.52	1.52	1.52	1.53	1.53
1.82	1.82	1.82	1.82		1.84
				1.86	
1.96	1.97	1.97	1.96		
2.08					2.08
				2.14	
2.26				2.26	2.24
2.34	2.36	2.36	2.34		
2.68			2.68	2.66	2.66
2.84	2.80	2.80	2.80	2.88	2.88
3.22	3.24	3.24	3.20	3.22	3.22
			3.50	3.50	3.50
			3.90	3.90	3.90
			4.20	4.20	4.20
			5.00	5.00	5.00
			6.10	6.10	6.10
				6.50	6.50
					7.40
				8.00	
				9.50	9.30
					11.9

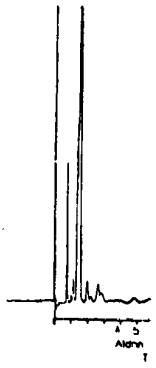


Fig. 1. GLC separation under EXPERIMENTAL.



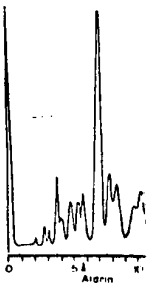


Fig. 2. GLC separation 1248. GLC conditions

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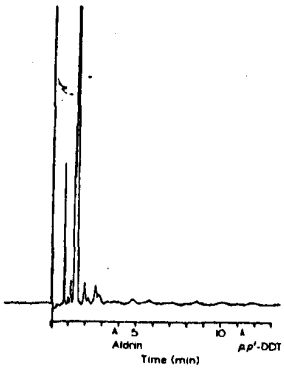


Fig. 1. GLC separation on 10% DC-200 column of 120 ng Aroclor 1221. GLC conditions are given under EXPERIMENTAL.

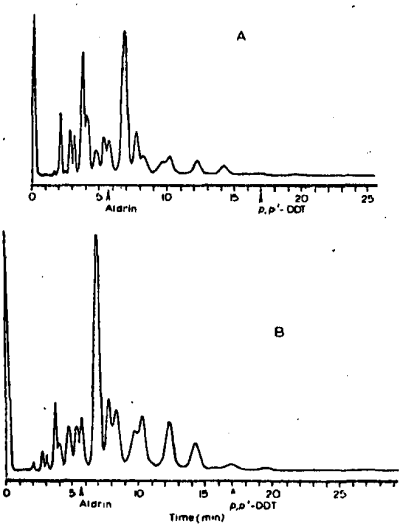


Fig. 2. GLC separation on 10% DC-200 column of (A) 50 ng Aroclor 1242 and (B) 50 ng Aroclor 1248. GLC conditions are given under EXPERIMENTAL.

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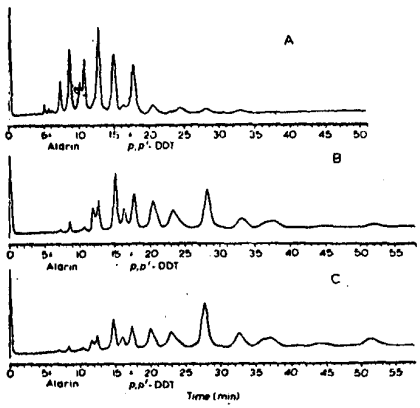


Fig. 3. GLC separation on 10% DC-200 column of (A) 32 ng Aroclor 1254, (B) 20 ng Aroclor 1260 and (C) 20 ng Aroclor 1262. GLC conditions are given under EXPERIMENTAL.

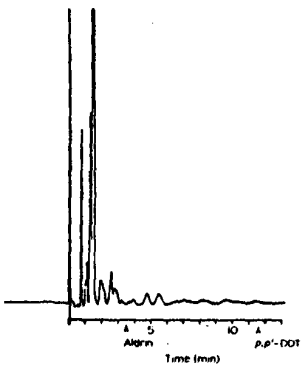


Fig. 4. Gas chromatographic separation on 1:1 25% QF-1/10% DC-200 of 120 ng Aroclor 1221. GLC conditions are given under EXPERIMENTAL.

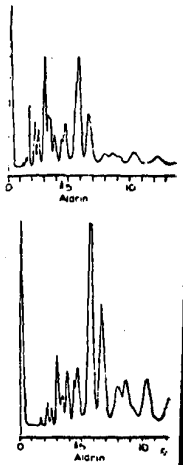


Fig. 5. Gas chromatographs and (B) 50 ng Aroclor 12[illegible]

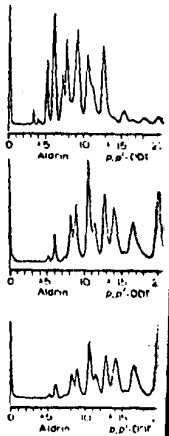


Fig. 6. Gas chromatogra[illegible] (B) 20 ng Aroclor 1[illegible]

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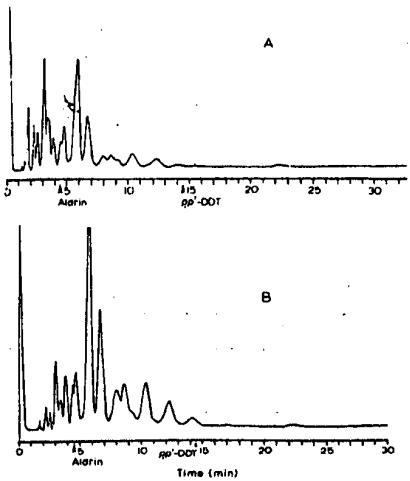


Fig. 5. Gas chromatographic separation on 1:1 15% QF-1/10% DC-200 of (A) 50 ng Aroclor 1242 and (B) 50 ng Aroclor 1248. GLC conditions are given under EXPERIMENTAL.

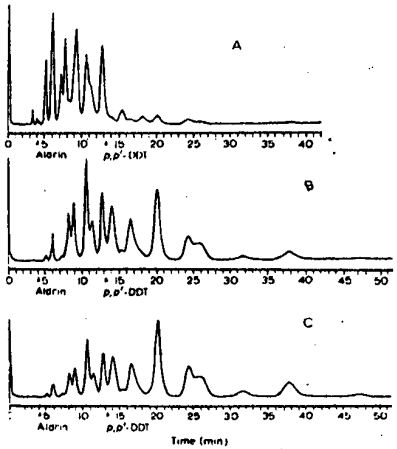


Fig. 6. Gas chromatographic separation on 1:1 15% QF-1/10% DC-200 of (A) 32 ng Aroclor 1254, (B) 20 ng Aroclor 1260, and (C) 20 ng Aroclor 1262. GLC conditions are given under EXPERIMENTAL.

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PROBLEMS OF I
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Department of Chem

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SUMMARY

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INTRODUCTION

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