Quantitative PCB Standards for Electron Capture Gas Chromatography

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Abstract

The weight of PCB represented by each electron capture gas chromatographic (EC-GC) peak in solutions of Aroclors 1221-1260 has been determined. The Aroclor samples from which these solutions were prepared are proposed as quantitative PCB standards. Their compositions were determined by elemental analysis, GC with a Coulson conductivity detector, and combined GC/MS. Retention times relative to p,p′-DDE are recommended to designate individual GC peaks of PCB's. A table is given for each Aroclor showing the weight percent of each EC-GC peak in the mixture. A procedure using Aroclors 1242, 1254, and 1260 is recommended for analyzing environmental samples containing more than one Aroclor mixture. Stock solutions of the Aroclors in isooctane are stable except when directly exposed to sunlight. Ampoules of the Aroclor solutions are offered.

Introduction

Quantitation of polychlorinated biphenyls (PCB's) from electron capture (EC) chromatograms is complicated because the EC detector responds differently to each PCB isomer (1,2). Quantitation by direct comparison of an unknown EC chromatogram with those of Aroclor standards is difficult because individual peaks in environmental samples are sometimes obscured by pesticide residues, are completely missing, or have considerably different relative intensities.

To avoid these difficulties, Berg and co-workers (3) have proposed that the PCB's in a sample be converted to decachlorobiphenyl, and the EC signal from this derivative be compared with that from conversion of a known amount of Aroclor 1254 to decachlorobiphenyl. This method appears to be ideally suited to a monitoring program designed for rapid and sensitive measurement of the total quantity of PCB's without regard to composition. In many cases, this derivative approach is undesirable because an extra analytical step is required and because any evidence of metabolism or degradation of the sample is destroyed. The disadvantages of both the direct comparison method and the derivative method can be largely overcome by using Aroclor standards in which the quantitative composition of each EC-GC peak is known. We have prepared these standards and recommend procedures for their use.

Experimental

The Monsanto Company* provided the Aroclor samples, which were not marked with lot numbers. Elemental analysis by Galbraith Laboratories, Knoxville, Tennessee, showed the following percent compositions (average of triplicate analyses):

Aroclor 1221	C, 72.94; H, 4.45; Cl, 22.74
1232	C, 64.44; H, 3.46; Cl, 31.88
1242	C, 54.64; H, 2.70; Cl, 42.86
1248	C, 49.50; H, 2.17; Cl, 48.64
1254	C, 44.10; H, 1.61; Cl, 54.30
1260	C, 38.18; H, 0.94; Cl, 60.87

The Food and Drug Administration provided the primary standard p,p′-DDE, which was used as a retention time standard and calibration standard for the conductivity detector.

A Microtek 220 gas chromatograph was equipped with a Coulson conductivity detector. The column was a 6 ft x 1/4 in., o.d., glass U-shaped tube packed with 3% SE-30 on 80/100 Gas Chrom Q. The carrier gas was helium at a flow rate of 60 ml/min. All Aroclors were chromatographed isothermally; Aroclors 1221 and 1232 at 175°C; 1242, 1248 and 1254 at 185°C; and 1260 at 190°C. The chromatograms were quantitated by measuring peak areas with either a planimeter or disc integrator.

A Microtek 220 gas chromatograph with Ni-63 EC detector was operated at 15-30 V (DC) and 275°C. The column was a 6 ft x 1/4 in., o.d., glass U-shaped tube packed with 3% SE-30 on 80/100 mesh Gas Chrom Q. The carrier gas was nitrogen at 90 ml/min. Aroclors 1221 through 1254 were chromatographed isothermally at 200°C and Aroclor 1260 at 215°C.

An F&M 700 gas chromatograph with tritium EC detector at 205°C was operated at a pulse interval of 15 microseconds. The coiled glass column was 6 ft x 1/4 in., o.d., packed with 5% SE-30 on 80/100 Gas Chrom Q. The carrier gas was 95% argon and 5% methane at 80-100 ml/min. All samples were chromatographed isothermally at 195°C.

Mass spectra (70 eV) were obtained on a Finnigan 1015-C quadrupole mass spectrometer interfaced with a Gohlke separator to a modified Varian 1400 GC. GC conditions were set to produce chromatograms equivalent to those from EC-GC. The spectrometer was controlled by a DEC PDP-8 computer, and spectra were collected on magnetic tape and printed or plotted under computer control.

*Mention of products or companies does not imply endorsement by the Environmental Protection Agency.

1. Gregory, N. L., J. Chem. Soc. (B), 1968, 295 (1968).
2. Zitko, V., Hutzinger, O., and Safe, S., Bull. Environ. Contam. Toxicol. 6, 160 (1971).
3. Berg, O. W., Diosady, P. L., and Rees, G. A. V., Bull. Environ. Contam. Toxicol. 7, 338 (1972).

Results and Discussion

The weight of PCB present in each GC peak of a given Aroclor can be calculated from two pieces of information:

1) the empirical formula of the compound represented by the peak, and
2) the absolute amount of chlorine represented by the peak.

Combined GC/MS determines the first, and a GC equipped with an electrolytic conductivity detector can determine the second.

GC/MS examination of Aroclors showed that several peaks were mixtures of PCB's with different numbers of chlorines. To estimate the composition of a GC peak containing PCB's with different numbers of chlorines, the following observation was used: equal weights of two PCB's that differ only by one chlorine give the same sum, within 20%, when the intensities of all the signals from the molecular ion, or parent, cluster, the parent-minus-one-chlorine cluster and parent-minus-two-chlorine cluster of each PCB are added. This rule was derived from a limited study of quadrupole mass spectra of a series of synthetic PCB's (4) and may not hold for other types of spectrometers.

Figure 1 is the mass spectrum from an Aroclor 1248 GC peak that is a mixture of one or more trichlorobiphenyls and one or more tetrachlorobiphenyls. The molecular ion pattern at m/e 290-298 is typical of four-chlorine molecules. These molecules lose one chlorine, producing a three-chlorine fragment pattern (parent-minus-one-chlorine) at m/e 255, 257, 259, and 261. However, between these signals, there is also a strong three-chlorine pattern at m/e 253, 255, 259, and 261; this is the parent ion cluster of the trichlorobiphenyl(s). The signals at m/e 220-226 are seen after loss of two chlorines from the tetrachlorobiphenyl (the trichlorobiphenyl(s) does not show a significant signal for loss of one chlorine), and the signals at m/e 186 and 188 are seen after loss of two chlorines from trichlorobiphenyl.

In Figure 1, the tetrachlorobiphenyl intensities (peak heights) totaled 612 mm and the trichlorobiphenyl 804 mm. Tetrachlorobiphenyl is thus about two-

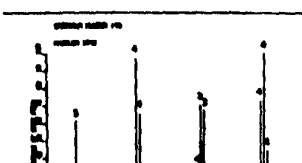

Figure 1. A limited portion of the mass spectrum of a mixture of tetrachlorobiphenyl(s) and trichlorobiphenyl(s) from GC peak 91 of Aroclor 1248 (See Figure 2). The abscissa is marked in atomic mass units (m/e).

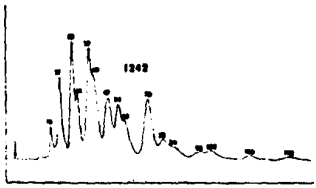

Figure 2. Gas chromatogram of Aroclor 1242 on SE-30 with an electrolytic conductivity detector. The peak identification numbers correspond to the retention time relative to p,p'-DDE=100. From injection, at the arrow, to peak 148 was about 20 min.

thirds of the mixture. These data were used to calculate the average molecular weight of the material in the GC peak.

The Coulson conductivity detector responds linearly to chlorine. Linear response to PCB's was shown with individual PCB isomers (4) containing one to six chlorines, and the detector response was checked for reproducibility several times each day with p,p'-DDE. A typical Coulson chromatogram for 1242 is shown in Figure 2. Peak resolution was not completely optimized here or in the GC-MS work so that these separations would be typical of those in the pesticide literature (5-10). Other studies (4, 11, 12) have show that the Aroclors are much more complicated than shown here, but this resolution is adequate for routine analysis.

The area of each Aroclor peak was determined and the weights (nanograms) of chlorine and PCB present were calculated using the response of p,p'-DDE as follows:

$$\frac{\text{ng DDE injected}}{\text{DDE peak area (cm}^2\text{)}} \times \frac{4 \times \text{at. wt. Cl}}{\text{mol. wt. DDE}} = \frac{\text{ng Cl}}{\text{cm}^2}$$

$$\frac{\text{ng Cl}}{\text{cm}^2} \times \text{PCB peak area (cm}^2\text{)} = \text{ng Cl}$$

$$\text{ng Cl} \times \frac{\text{Gram molecular weight}}{\text{No. of chlorines in molecule} \times 35.46\ \text{g}} = \text{ng PCB}$$

Tables I-VI present these results as the percent of

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Table I. Composition of Aroclor 1221

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	No. of Chlorines[c]	
11	31.0	15.0	1	
14	19.9	9.1	1	
16	10.1	9.7	2	
19	2.9	9.7	2	
21	20.0	9.0	2	
28	5.4	13.9	2	95%
			3	5%
33	1.4	30.1	3	10%
			4	90%
37	1.7	48.8	3	
40			3	
Total	93.3			

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent. Overlapping peaks that are quantitated as one peak are bracketed.

[b]Standard deviation of seventeen results as a percentage of the mean of the results.

[c]From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

Table II. Composition of Aroclor 1232

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	No. of Chlorines[c]	
11	16.2	3.4	1	
14	9.9	2.5	1	
16	7.1	0.5	2	
20	17.0	2.4	2	
21			2	
28	8.6	3.4	2	40%
			3	60%
32	3.0	4.7	3	
37	6.3	2.5	3	
40	6.4	2.7	3	
47	4.3	4.1	4	
54	3.4	3.4	3	33%
			4	67%
58	2.0	2.7	4	
70	4.6	3.1	4	90%
			5	10%
76	1.7	7.5	4	
Total	94.3			

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent. Overlapping peaks that are quantitated as one peak are bracketed.

[b]Standard deviation of four results as a means of the results.

[c]From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

total Aroclor weight represented by each GC peak. The peaks are identified by their retention times relative to p,p'-DDE. We recommend that this be adopted as a standard method for designating individual PCB GC peaks. The separate percentages given for overlapping peaks were obtained by dividing the area with a perpendicular to the baseline from the minimum point between the two peaks. The accuracy of the Coulson determinations was checked by comparing each Aroclor's calculated percent chlorine with its elemental analysis. The amount found by Coulson GC was 98-103% of the elemental analyses except for Aroclor 1221.

Seventeen analyses with 1221 were performed and the average of the data was used to prepare Table I. Willis and Addison (13) have recently reported semi-quantitative values for the composition of Aroclor 1221. Their analysis was based on EC-GC and flame ionization GC. They found 12.7% biphenyl present and about the same amounts of other materials as in Table I. Willis and Addison accounted for 92.9% of the weight of materials in 1221. If their 12.7% biphenyl

Table III. Composition of Aroclor 1242

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	No. of Chlorines[c]	
11	1.1	38.7	1	
19	2.9	4.2	2	
21	11.5	3.9	2	
28	11.0	5.8	2	25%
			3	75%
32	6.1	4.7	3	
37	11.5	5.7	3	
40	11.1	6.2	3	
47	8.5	4.8	4	
54	6.8	2.9	3	33%
			4	67%
58	5.6	5.3	4	
70	10.3	2.8	4	90%
			5	10%
76	3.0	4.3	4	
84	2.7	9.7	5	
90	1.8	9.4	5	
104	2.3	16.4	5	
125	1.9	20.4	5	90%
			6	10%
146	1.9	10.9	5	75%
			6	25%
Total	96.3			

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.

[b]Standard deviation of six results as a percentage of the mean of the results.

[c]From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

13. Willis, D. E., and Addison, R. F., *J. Fisheries Res. Board Can.* **29**, 203 (1972).

is added to the PCB values in Table I, the weight percent Aroclor is 103.0 A mixture composed of the PCB's that Wilks and Addison quantitated would contain 19.6% by weight chlorine; the Coulson determinations gave 22.9%; elemental analysis gave 22.7%.

When the data in Tables I-VI are compared to Aroclor chromatograms from an EC detector operated in the DC mode (Figures 3-8), peak size obviously is not a valid indication of concentration. For example, in Aroclor 1242 (Figure 6a) peaks 21, 28, 37, and 40 each represent about 11% of the mixture (See Table III), but their areas differ by as much as 65%. There are also major differences in peak ratios when the Aroclors are measured with a detector operated in the pulsed mode as shown in Figure 5b.

A Technique to Quantitate PCB's in Environmental Samples

The chromatograms of PCB's from environmental samples usually show some evidence of degradation or metabolism. A sample may contain a single partially degraded Aroclor or a combination of Aroclors. Such samples can be quantitated by using the standard Aroclors, the data in Tables I-VI, and some simple computation rules. The key principle is that the total amount of PCB present is *the sum of the amounts from all the individual peaks.*

To quantitate PCB's, chromatograph known amounts of the standards. Measure the area for each peak. Using the tables, determine the response factor

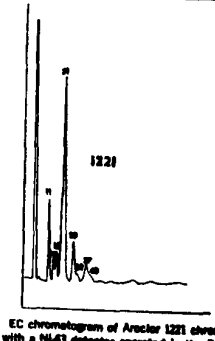

Figure 3. EC chromatogram of Aroclor 1221 chromatographed on SE-30 with a Ni-63 detector operated in the DC mode. The peak identification numbers correspond to the retention time relative to p,p'-DDE=100.

Table IV. Composition of Aroclor 1248

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	No. of Chlorines[c]
21	1.2	29.9	2
28	5.2	5.2	3
32	3.2	3.8	3
37	5.3	2.6	3
40	3.3	3.9	3 / 4 — 85% / 15%
47	15.6	1.1	4
54	9.7	6.0	4 / 5 — 10% / 90%
58	9.3	5.6	4
70	19.0	1.4	4 / 5 — 80% / 20%
78	6.6	2.7	4
84	4.9	2.6	5
98	3.2	3.2	5
104	3.3	3.6	4 / 5 — 10% / 90%
112	1.2	6.6	5
125	2.6	5.9	5 / 6 — 90% / 10%
146	1.5	10.0	5 / 6 — 85% / 15%
Total	106.1		

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.
[b]Standard deviation of six results as a percentage of the mean of the results.
[c]From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

Table V. Composition of Aroclor 1254

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	No. of Chlorines[c]
47	6.2	3.7	4
54	2.9	2.6	4
58	1.6	2.8	4
70	13.2	2.7	4 / 5 — 25% / 75%
84	17.2	1.9	5
98	7.5	5.3	5
104	13.6	3.8	5
125	18.9	2.4	5 / 6 — 79% / 20%
146	10.4	2.7	5 / 6 — 20% / 79%
160	1.3	8.4	6
174	2.4	5.5	6
208	1.3	13.6	6
223	1.0	25.1	7
Total	100.0		

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent.
[b]Standard deviation of six results as a percentage of the mean of the results.
[c]From GC/MS data. Peaks containing mixtures of isomers are bracketed.

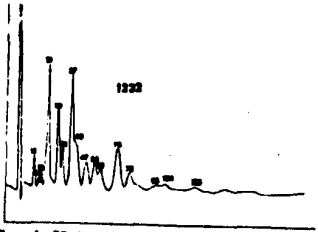

Figure 4. EC chromatogram of Aroclor 1232 chromatographed on SE-30 with a Ni-63 detector operated in the DC mode. The peak identification numbers correspond to the retention time relative to p,p'-DDE=100.

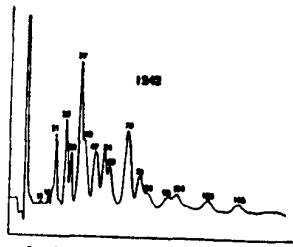

Figure 5a. EC chromatogram of 0.3 ng Aroclor 1242 chromatographed on SE-30 with a Ni-63 detector operated in the DC mode. The peak identification numbers correspond to the retention time relative to p,p'-DDE=100.

Table VI. Composition of Aroclor 1260

RRT[a]	Mean Weight Percent	Relative Std. Dev.[b]	No. of Chlorines[c]
70	2.7	6.8	5
84	4.7	1.6	5
98	3.8	3.8	[d]
104			5 60%
			6 40%
117	3.3	6.7	6
126	12.3	3.3	[e]
			5 15%
			6 85%
146	14.1	3.6	6
160	4.9	2.3	[e]
			6 50%
			7 50%
174	12.4	2.7	6
200	9.3	4.0	[e]
			6 10%
			7 90%
222			[d]
244	9.8	8.4	6 10%
			7 90%
280	11.0	2.4	7
302	4.3	3.0	7
372	4.8	8.6	8
448	.6	33.3	8
502	1.8	10.3	8
Total	99.8		

[a]Retention time relative to p,p'-DDE=100. Measured from first appearance of solvent. Overlapping peaks that are quantitated as one peak are bracketed.

[b]Standard deviation of six results as a mean of the results.

[c]From GC/MS data. Peaks containing mixtures of isomers of different chlorine numbers are bracketed.

[d]Composition determined at the center of peak 104.

[e]Composition determined at the center of peak 222.

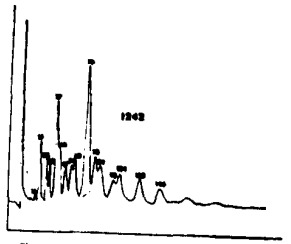

Figure 5b. Pulsed mode EC chromatogram of Aroclor 1242 chromatographed on SE-30 with a tritium foil detector. The peak identification numbers correspond to the retention time relative to p,p'-DDE=100.

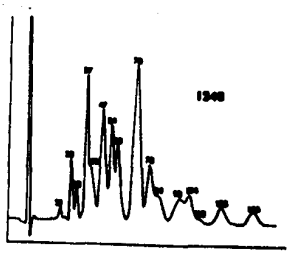

Figure 6. EC chromatogram of Aroclor 1248 chromatographed on SE-30 with a Ni-63 detector operated in the DC mode. The peak identification numbers correspond to the retention time relative to p,p'-DDE=100.

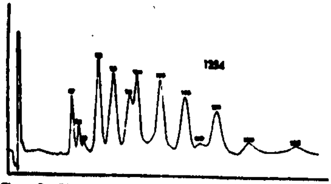

Figure 7. EC chromatogram of Aroclor 1254 chromatographed on SE-30 with a Ni-63 detector operated in the DC mode. The peak identification numbers correspond to the retention time relative to p,p'-DDE = 100.

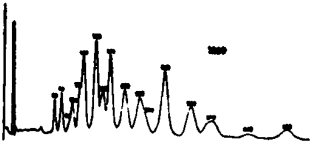

Figure 8. EC chromatogram of Aroclor 1260 chromatographed on SE-30 with a Ni-63 detector operated in the DC mode. The peak identification numbers correspond to the retention time relative to p,p'-DDE = 100.

(ng PCB/cm²) for each peak. Chromatograph the sample and measure the area of each peak. Multiply the area of each peak by the response factor for that peak. Add the nanograms of PCB found in each peak to obtain the total nanograms of PCB present.

Environmental Samples Containing Only One Aroclor

An example of a sample containing a single Aroclor that is partially metabolized or degraded is seen in Figure 9a. This is a chromatogram of fat extract from a turkey that had been fed fishmeal contaminated with Aroclor 1248. Figure 9b is standard Aroclor 1248 run at the same conditions.

Several peaks present in the standard are completely missing in the sample, e.g., those at relative retention times (RRT) 28 and 54. Some quantitation methods do not make an adjustment when a peak is missing from the sample. For example, one method used to quantitate this sample compared the sum of all the major sample peak heights with the sum of all the peak heights in the standard. This approach assumes that all the peaks present in the standard are also present in the sample and that all PCB peaks have the same electron capture response. Neither assumption is valid. Quantitation by calculating the amount of PCB present in each individual peak is the solution of this missing peak problem. Calculation by the sum-of-heights method indicates 0.28 ng of PCB is present; the individual peak method gives 0.18 ng.

The standard Aroclor (Figure 9b) shows the peak at RRT 84 separated only as a barely discernible shoulder on peak 78; in the sample their proportions are different and they elute as two separate peaks. Since individual values are given for these peaks in Table III, their individual values can be calculated. In Figure 9, peaks 37 and 40 elute as a single peak for both the sample and the standard. They are quantitated by combining the values in Table III. The sample peak at RRT 208 was not quantitated because no accurate comparison from the standard was available. Here, fortunately, the peak is only a small portion of the total and can be ignored without seriously biasing the results.

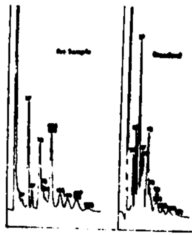

Figure 9a. DC mode EC chromatogram of a fat sample from a turkey that had been fed fishmeal contaminated with Aroclor 1242. The peak identification numbers correspond to the retention time relative to p,p'-DDE = 100.
Figure 9b. Standard Aroclor 1242 run under the same conditions as Figure 9a.

Peaks arising from pesticides can be mistaken for PCB's; DDE and DDT were present in this sample. The PCB peaks eluting in these RRT regions are only about 4% of Aroclor 1242 and can be omitted from the total without causing major error. If there is any question about the presence of DDT, chlorinated naphthalenes, chlorinated terphenyls, or other interferences, then the total PCB residues computed by this method can be confirmed by the derivative technique (8, 14). Methods are also available to separate DDT-type pesticides from PCB's if these results are necessary (8, 15-17).

Environmental Samples Containing More Than One Aroclor

Most PCB contaminated samples of fish, water, and sediment contain residues of several Aroclors. Usually the sample chromatogram can simply be divided into three separate areas and peaks in each area quantitated by using the appropriate Aroclor. Peaks with RRT 11-70 are compared individually to cor-

14. Hutzinger, O., Safe, S., and Zitko, V., Intern. J. Environ. Anal. Chem. 2, 95 (1972).
15. Armour, Judith, and Burke, J., J. Assoc. Offic. Anal. Chemists 53, 761 (1970).
16. Burke, J. A., J. Assoc. Offic. Anal. Chemists 55, [illegible] ([illegible]).
17. Leoni, V., J. Chromatog. 62, 63 (1971).

responding peaks in Aroclor 1242, peaks 84-174 with Aroclor 1254, and peaks with larger RRT with Aroclor 1260. These three Aroclors have been chosen for routine use as standards because they are the PCB's most often found in environmental samples, they were sold in largest quantities (particularly 1242 and 1254), and their chromatograms include all the EC peaks normally found in other Aroclor mixtures. In addition, this system of dividing the chromatograms generally matches the changes in chlorine numbers; i.e., the peaks used from the 1242 standard are the mono-through-tetra chlorobiphenyls, the peaks calculated with 1254 are penta- and hexachlorobiphenyls, and the peaks from the 1260 standard are used to measure the hepta- and octachlorobiphenyls.

Figure 10 is the chromatogram of a composite liver extract of three bluegills from a PCB-contaminated river. This sample obviously contains residues of several Aroclors and was quantitated as described above. However, some sample chromatograms require a more rigorous division. A schematic of this division procedure is shown in Figure 11.

The logic of this division is based on several key facts. Since Aroclor 1254 shows no appreciable peaks (see Figure 7) before RRT 47, any peaks with a lower RRT indicate the presence of some Aroclor of less chlorine content. The most likely compound, from experience and commercial usage, is 1242. Ideally, all peaks through RRT 78 would be calculated against 1242 (though many of them are present in 1254 as well), because peak 78 is unique to 1242 and not ordinarily discernible in 1254 (compare Figures 5 and 6). However, when chromatographic resolution is not optimum, mixtures of the two Aroclors may show peak 78 as only a shoulder on peak 84. Experiments with known mixtures, described below, show that in this case better results are obtained by making the division at RRT 70 and treating peak 78 + 84 as though it were all peak 84 from 1254.

A peak with RRT 117 is present in Aroclor 1260, but absent from 1254. The presence or absence of this peak in the sample chromatogram determines the standard used to quantitate the remaining peaks. Since some columns do not resolve peak 117, the investigator will not always know that 1260 is present. Experiments with mixtures show that calculations based on 1254 are adequate in this case.

All peaks of RRT larger than 174 are calculated with Aroclor 1260 because the relative standard deviations for these peaks are much lower in 1260 (Table VI) than in 1254 (Table V). These rules were tested with known-weight-ratio Aroclor mixtures that were chromatographed through a low resolution column with the GC detector operated in the pulsed mode. Table VII gives the amount of PCB measured as a percentage

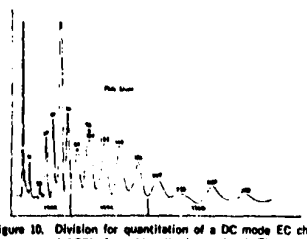

Figure 10. Division for quantitation of a DC mode EC chromatogram of PCB's from bluegill liver extract. The column substrate was SE-30. The large peak at RRT 58 is an artifact.

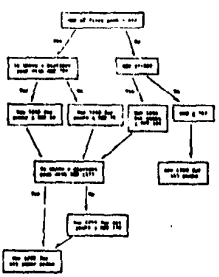

Figure 11. Chromatogram Division Flowchart.

Table VII. Percent Recoveries of Aroclor Mixtures Using Chromatogram Division Rules

Mixture	Weight Ratio	% Recovery 1242[a]	% Recovery 1242[b]	% Recovery 1254[c]	% Recovery 1254[d]
1242	1	98	103		
1254	1				
1242	3	96	100		
1254	1				
1242	4	96	100		
1254	1				
1242	5	99	102		
1254	1				
1242	2	103[e]	107[e]		
1260	1	102[e,e]	104[e,e]		
1254	1.5			109	101
1260	1				
1254	1			103	94
1260	1.5				
1254	1			105	96
1260	4				
1242	4			99[a]	99[b]
1254	1			105[b]	103[b]
1260	2				

[a]Peaks through RRT 70 calculated as 1242 (Table III).
[b]Peaks through RRT 84 calculated as 1242 (Table III).
[c]Peaks through RRT 104 calculated as 1254 (Table V).
[d]Peaks through RRT 174 calculated as 1254 (Table V).
[e]All other peaks calculated as 1260 (Table VI).

of that injected. These results were calculated using peak heights from the chromatograms, Tables III, V, and VI, and the rules given above.

These computation rules and Tables I-VI should also apply to chromatograms run on DC-200, OV-17 and OV-101 columns. However, QF-1 and OV-225 elute PCB's in a different order and the tables do not apply.

Standard Solution Stability

To test the stability to UV light of Aroclor solutions in concentrations typically used in GC analyses (16), several sealed glass ampoules of Aroclors 1242 and 1254 (10 ng/μl isooctane) were prepared. Some samples were stored in the dark, some were continuously exposed two feet from a fluorescent light fixture fitted with a decorative clear plastic shield, and several were stored in a window that received several hours of sunshine each day. No measurable changes in peak ratios were observed by EC-GC in five observations over two months' time. However, after identical samples were exposed to direct sunlight for nine days, some peak ratios changed significantly. For example, in 1254 the apparent weight of material in RRT peak 125 decreased by 50% and RRT peak 104 increased by 15%. Therefore, direct exposure to sunlight should be avoided.

Limited supplies of Aroclors 1242, 1254, and 1260 as dilute isooctane solutions in glass ampoules are available from the authors as reference Aroclor kits. Aroclors 1221, 1232, and 1248 are also available if there is a special need.

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TECHNICAL NOTE

Response of the Flame Ionization Detector to Nitrogen and Oxygen

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Introduction

The FID was reported to be insensitive to fixed gases by earlier workers (1-3). But later, under certain conditions, limited response to the FID was recorded from air (4), from some inorganic compounds of sulphur (5) and carbon (6), and even from oxygen and nitrous oxide (7). During analysis of adsorbed gases of the sedimentary rocks using Perkin Elmer Vapor Fractometer (Model 154 D), fitted with FID, the authors observed positive response from nitrogen and oxygen. Such response was found to depend both on hydrogen concentration in the flame and background current. Conditions were then worked out to maintain the response linear and more sensitive for the purpose of its possible use in analytical work.

Experimental

Separation of nitrogen from oxygen was achieved on 2 m 5-A molecular sieve column at a temperature of 80°C. Helium, supplied by M/S Matheson & Co., U.S.A., was used as the carrier gas at a flow rate of 40 ml min⁻¹. As recommended by the manufacturer of the fractometer, a flow rate of 400 ml min⁻¹ of air was maintained throughout. A critical flow rate of 34 ml min⁻¹ of hydrogen was found to give maximum response to the FID from both nitrogen and oxygen with normal peak shapes on 5 mv potentiometric recorder having a chart speed of 30 in. per hour. A background current of 3×10^{-11}A was observed as against the recommended value of 5×10^{-11}A, under the set of worked out conditions. At the maximum sensitivity adjustment of the FID amplifier, a minimum sample size of 0.02 ml produced measurable peak area. This

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