Journal of Chromatography, 257 (1983) 255–266
Elsevier Scientific Publishing Company, Amsterdam — Printed in The Netherlands

CHROM. 15,484

COMPARISON OF METHODS OF ANALYSIS OF POLYCHLORINATED BIPHENYLS IN OILS

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(Received November 1st, 1982)

SUMMARY

In order to standardize and validate methods for the analysis of polychlorinated biphenyls in transformer oil, waste oil, hydraulic fluid and capacitor oil in the concentration range 30–500 ppm, a study was performed which included the variables of capillary/packed column separation, Hall conductivity/electron capture detection and autoquant/area/height peak quantification. Statistical analysis of the results shows that there is no significant overall advantage in any combination of these procedures. In addition, certain statistically significant factors specific to a given oil/analysis method combination have been identified.

INTRODUCTION

There is at present no single generally accepted official method for determining polychlorinated biphenyls (PCBs) in oils. Gas chromatographic (GC) methods have been proposed for PCB analysis[1–4] that have been improved in recent studies[5–9]. These methods have been applied to the monitoring of PCBs in a variety of matrices[10–12]. Reviews of the literature relating to various aspects of the instrumentation used for these analyses have also been published[13,14]. In order to standardize and validate methods for the analysis of PCBs in transformer oil, waste oil, hydraulic fluid and capacitor oil, the U.S. EPA instituted a study entitled "Validation of Procedures for PCBs in Oils"[15]. The objectives of this study included the comparison of results obtained in using electron-capture (ECD) and Hall conductivity (HCD) detectors for the analysis of PCB-containing extracts from the above oils. In an extension of that effort, this study was designed to compare the results of analysis using packed column isothermal and fused-silica capillary column temperature-programmed techniques, as

0021-9673/83/0000–0000/$03.00

TABLE I

PCB MATERIALS AND CLEANUP TECHNIQUES

Each set contains six samples as three Youden pairs (listed in the text as samples A–F).

PCB (Aroclor)	*Matrix*	*Cleanup technique*
1016	Capacitor oil	Alumina column
1242	Hydraulic fluid	Alumina column
1254	Waste (road) oil	Florisil column
1260	Transformer oil	Acid extraction

well as the ECD and HCD detectors. In addition, several methods of peak quantitation were compared.

EXPERIMENTAL

Experimental design

The matrix of PCBs, oil type, and cleanup techniques used, as defined by the EPA contract[15], are given in Table I. Six Aroclor samples in the concentration range 30–500 ppm (w/w) as three Youden pairs were analyzed in each set. Each extract was analyzed using the method given in Fig. 1.

Quality control

Standardization of PCB recovery values and instrument calibration was performed using U.S. EPA-EMSL primary standards[15]. A PCB spike recovery of 80 ± 20% was maintained throughout the study. Each batch of twelve samples was extracted and quantitated together with two duplicates, two method standards (spikes) and one reagent blank. Values of alumina and Florisil activities were verified for each batch of adsorbents. These procedures, as well as the lack of duplicate analyses of each sample extract, were dictated by the contract protocol.

Cleanup procedure

Details of the cleanup procedure can be obtained from the US EPA contractor[15]. A summary is given below.

For alumina column cleanup (used for capacitor oil and hydraulic fluid), 3% deactivated alumina plus anhydrous sodium sulfate were used to remove interferences. Elution with hexane was followed by GC analysis.

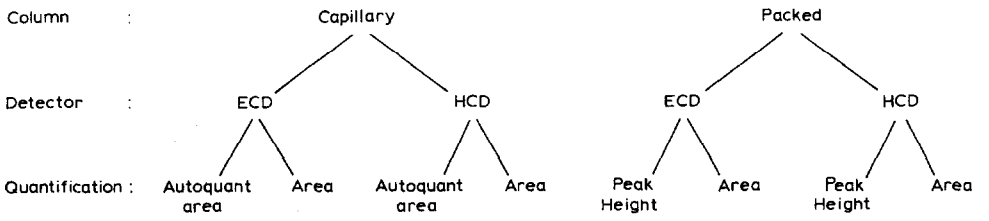

Fig. 1. PCB analysis flowchart (as applied to each PCB sample).

For Florisil cleanup (used for waste oil), activated Florisil was used to remove interferences. This was followed by anhydrous sodium sulfate, which was used to dry the eluate. Elution with hexane was followed by GC analysis.

For acid extraction (used for transformer oils), hexane-diluted samples were extracted once with concentrated sulfuric acid. The extracted samples were analyzed by GC.

Gas chromatography

Details of the GC procedure (packed column only) can be obtained from the contractor[15]. A summary is given below.

Packed column. A glass column, 180 cm × 2 mm I.D., packed with Gas-Chrom Q (100–120 mesh) coated with 3% OV-1 was used. Separation was achieved with the column oven set between 160 and 205°C (isothermal) for the four Aroclors. On-column injection was used.

Capillary column. A 30-m long fused-silica capillary column coated with SE-54 was used. Injection was performed using an automated Grob injector. Temperature programming in the range 50–250°C at 10°C/min was used for the Aroclors.

Instruments. A Tracor 560 gas chromatograph equipped with a Tracor/Varian 770 autoinjector was used for all packed column chromatography. This instrument was also equipped with a Tracor 700A HCD operated in the halogen mode[9], and a Tracor ECD.

A Hewlett-Packard 5880 A gas chromatograph equipped with a Hewlett-Packard 7835 autoinjector was used for all capillary column chromatography. This instrument was also equipped with a Hewlett-Packard Tracor 700A HCD operated in the halogen mode, and Hewlett-Packard ECD.

Quantification. Details of the quantification procedure can be obtained from the contractor[15]. A summary is given below.

Packed column data. All peak area integration was performed using a Hewlett-Packard 3390 A integrator. Peak height measurements were performed manually or, in a few instances, using peak height information provided by the integrator.

Capillary column data. All peak area integration was performed using a Hewlett-Packard 5880 A Level 4 data system. Data listed as "Computer Autoquant" were picked by the 5880 A data system using an algorithm designed to find peaks characteristic of the PCB pattern within defined retention windows[16].

RESULTS AND DISCUSSION

Results obtained from this study are valid only for the four PCB–matrix–cleanup procedures defined in Table I. Although conclusions drawn from these data *may* be applicable to other PCB–matrix–cleanup procedure combinations, this is not necessarily so. Within these limitations, these data should allow conclusions to be drawn as to which of the analytical procedure combinations given in Fig. 1 yield the most accurate data.

A full compilation of all of the data is beyond the scope of this paper[15], but the following is a summary of the most important points of comparison.

Analysis of variance model

Fig. 1 illustrates the experimental design for the data with respect to three factors: type of detector, type of column and method of quantification. The analysis of variance model is a nested factorial model[17]. As the two methods of quantification in a capillary column are different from the two methods in the packed column, the factor "method of quantification" is nested within the factor "column". However, both detectors occur with each type of column. Thus, the two factors, type of detector and type of column, are crossed.

Statistical analyses were performed using two different models: model 1 used analysis of variance with the relative absolute value as the dependent variable and model 2 used analysis of covariance with the measured concentration as the dependent variable and the actual concentration as the covariate. Both models included the nested factorial structure.

Statistical model 1

In the ideal situation, the measured concentration, y, would be equal to the actual concentration, x. In statistical model 1, the dependent variable that was used is $R = |y - x|/x$. The actual concentration was used as a factor in addition to the three factors mentioned under *Analysis of variance model*. The six actual concentrations were coded by rank from lowest to highest as concentrations 1–6. Table II gives a separate analysis of variance table for each of the four Aroclors. All computations were carried out with the statistical package SAS[18]. The actual concentration is a random effects factor and the three factors, type of detector, type of column and method of quantification, are all fixed effects factors. Thus, the model is a mixed effects nested factorial model with R as the dependent variable.

The analysis of variance (ANOVA) table for Aroclor 1 is given in Table II(a). It can be concluded that only two terms indicate significant differences in the dependent variable: the three-way interaction between column, detector and concentration is significant, which means that the interactions between type of column and type of detector are not the same at each level of concentration.

The other significant factor is the type of detector: the HCD has a smaller mean R value than the ECD. This implies that the mean R is smaller regardless of column type, method of quantification or concentration. However, the column by detector by concentration interaction is significant. Thus, there are differences between the mean R values for the two types of detectors, but the differences are dependent on the type of column and the concentration level.

The ANOVA table for Aroclor 2 is given in Table II(b). The only term that is significant is the detector by concentration interaction. The HCD gives the smaller mean R value for concentrations 2, 3 and 6 and the ECD has the smaller mean R value for concentrations 1, 4 and 5. These are true regardless of the type of column or method of quantification.

The ANOVA table for Aroclor 3 is given in Table II(c). The only factor that is significant is the type of detector. The average R value for the ECD detector is 0.195 and the average R value for the HCD detector is 0.349, regardless of the type of column, method of quantification or actual concentration.

Table II(d) gives the ANOVA table for Aroclor 4. The type of column is the only factor that is significant. The average R values for the capillary column and

packed column are 0.296 and 0.118, respectively, regardless of the type of detector, method of quantification or actual concentration.

Statistical model 2

A second statistical model is an analysis of covariance with the measured concentration as the dependent variable and the actual concentration as the covariate. The analysis of variance structure is the nested factorial model used in model 1. The model equation is

$$y = \beta x + \text{f(COL, D, Q)} + e$$

f(COL, D, Q) is a function of the three factors reflecting the nested structure. The model was originally fit with an intercept, but the intercept was not significantly different from zero. If f(COL, D, Q) were identically zero, *i.e.*, the type of column, detector and method of quantification would have no effect on the measured concentration, then the model would imply that the measured concentration would be zero when the actual concentration is zero. Further, if f( ) $\equiv$ 0 and there is perfect measurement, then $\beta = 1$. If f(COL, D, Q) is not identically zero for all combinations

TABLE II

ANALYSIS OF VARIANCE TABLES FOR THE *R* VALUES

C = True concentration; COL = column; D = detector; Q = method quantification.

Aroclor	*Source of variation*	*Sum of squares*	*Degrees of freedom*	*Mean square*	*F**	*p*
(a) Aroclor 1 (1016)	C	0.0438	5	0.0088 (1)	– (a)	
	COL	0.0890	1	0.0890 (2)	1.57 (b)	0.2659
	COL × C	0.2838	5	0.0568 (3)	– (c)	
	D	0.1664	1	0.1664 (4)	17.15 (d)	0.0090**
	D × C	0.0486	5	0.0097 (5)	0.16 (e)	0.9659
	Q(COL)	0.0519	2	0.0260 (6)	1.65 (f)	0.2419
	Q(COL) × C	0.1583	10	0.0158 (7)	1.23 (g)	0.3736
	COL × D	0.0189	1	0.0189 (8)	0.32 (h)	0.5982
	COL × D × C	0.2983	5	0.0597 (9)	4.66 (i)	0.0188***
	D × Q(COL)	0.0913	2	0.0457 (10)	3.57 (j)	0.0681
	Error	0.1284	10	0.0128 (11)		
(b) Aroclor 2 (1242)	C	0.1394	5	0.0279	– (a)	
	COL	0.0008	1	0.0008	0.09 (b)	0.7744
	COL × C	0.0431	5	0.0086	– (c)	
	D	0.0643	1	0.0643	0.53 (d)	0.5004
	D × C	0.6103	5	0.1221	8.72 (e)	0.0163**
	Q(COL)	0.0691	2	0.0346	2.44 (f)	0.1372
	Q(COL) × C	0.1416	10	0.0142	0.85 (g)	0.6021
	COL × D	0.0043	1	0.0043	0.31 (h)	0.6017
	COL × D × C	0.0698	5	0.0140	0.83 (i)	0.5550
	D × Q(COL)	0.0382	2	0.0191	1.14 (j)	0.3579
	Error	0.1675	10	0.0168		

(Continued on p. 260)

TABLE II *(Continued)*

Aroclor	*Source of variation*	*Sum of squares*	*Degrees of freedom*	*Mean square*	*F**	*p*
(c) Aroclor 3 (1254)	C	0.3230	5	0.0646	– (a)	
	COL	0.0216	1	0.0216	0.99 (b)	0.3652
	COL × C	0.1088	5	0.0218	– (c)	
	D	0.2833	1	0.2833	8.07 (d)	0.0363***
	D × C	0.1757	5	0.0351	0.82 (e)	0.5833
	Q(COL)	0.0355	2	0.0178	0.96 (f)	0.4168
	Q(COL) × C	0.1856	10	0.0186	0.57 (g)	0.8081
	COL × D	0.1949	1	0.1949	4.55 (h)	0.0861
	COL × D × C	0.2142	5	0.0428	1.30 (i)	0.3350
	D × Q(COL)	0.0448	2	0.0224	0.68 (j)	0.5269
	Error	0.3277	10	0.0328		
(d) Aroclor 4 (1260)	C	0.0506	5	0.0101	– (a)	
	COL	0.3817	1	0.3817	22.72 (b)	0.005**
	COL × C	0.0841	5	0.0168	– (c)	
	D	0.0337	1	0.0337	1.78 (d)	0.2400
	D × C	0.0947	5	0.0189	0.66 (e)	0.6713
	Q(COL)	0.0324	2	0.0162	0.35 (f)	0.7144
	Q(COL) × C	0.4658	10	0.0466	2.48 (g)	0.0838
	COL × D	0.0088	1	0.0088	0.31 (b)	0.6037
	COL × D × C	0.1439	5	0.0288	1.53 (i)	0.2636
	D × Q(COL)	0.6477	2	0.0239	1.27 (j)	0.3218
	Error	0.1876	10	0.0188		

* (a), (c), No exact F test available; (b) = (2)/(3); (d) = (4)/(5); (e) = (5)/(9); (f) = (6)/(7); (g) = (7)/(11); (h) (8)/(9); (i) = (9)/(11); (j) = (10)/(11).

** Significant at the 0.01 level.

*** Significant at the 0.05 level.

of COL, D and Q, then the intercept is not zero and is a function of these factors.

The comparison of the levels of column, detector and method of quantification within an analysis of covariance model should be done with adjusted means, in contrast to analysis of variance models that use the unadjusted means. As, in this model, the six values for the actual concentration are the same for each of the eight combinations of analytical procedures, the adjusted and unadjusted means are equal. Consequently, the average measured concentrations can be used for the comparisons in this analysis of covariance model.

Table III(a) gives the results of the analysis of covariance for Aroclor 1, which indicates that there are three significant factors: detector, quantification within columns and the interaction between these two. As the interaction is significant, the means that should be compared are the means for combinations of these factors and not the factors individually. Thus, as detector by quantification(column) is significant, comparisons should be made within the type of column between detectors and methods of quantification. Note that the estimated coefficient of x is 0.77, which is less than 1.0. This implies that on a given combination of type of column, type of detector and method of quantification, for a one unit increase in the actual concentra-

tion the measured concentration increases, on average, by 0.77 ppm.

As the interaction term is significant, the average measured concentration values, shown below in ppm, should be compared by detectors and methods of quantification within columns. Four means must be compared within each column. As the model is balanced, and the four means are to be compared in pairs, the Tukey method[19] of multiple comparisons was used with a 95% confidence level.

Capillary column				*Packed column*			
ECD		*HCD*		*ECD*		*HCD*	
Autoquant	*Area*	*Autoquant*	*Area*	*Area*	*Height*	*Area*	*Height*
140	150	190	160	130	120	240	130

This analysis indicated that there are no differences in the four means for the capillary column and that, within the packed column data, the average measured concentration for the HCD/area combination was significantly higher than the other three means, with no significant difference occurring among the other three means. Table III(b) gives the results of the analysis of covariance for Aroclor 2. The table indicates that none of the factors are significant. As $\hat{\beta} = 0.92$, a one unit increase in the actual concentration will result in the average measured concentration increasing by 0.92 ppm.

The analysis of covariance table for Aroclor 3 is given in Table III(c), in which it is shown that none of the factors are significant. As $\hat{\beta} = 1.03$, it can be concluded that for any column, detector and quantification combination the measured concentration is estimated to increase by 1.03 ppm on average for each one unit increase in the actual concentration.

Table III(d) gives the results of the analysis of covariance for Aroclor 4. The significant terms are actual concentration, the interaction of detector and method of quantification within columns and the type of detector. As the interaction is significant, the average measured concentration values should be compared by detectors and methods of quantification within columns.

The Tukey method of multiple comparisons was used to compare the means within a column. The analysis for the capillary column indicated that the average measured concentration, shown below in ppm, for the autoquant method with the ECD detector is significantly lower than the other combinations used with the capillary column and that there are no significant differences among the other combinations.

Capillary column				*Packed column*			
ECD		*HCD*		*ECD*		*HCD*	
Autoquant	*Area*	*Autoquant*	*Area*	*Area*	*Height*	*Area*	*Height*
100	170	200	170	160	170	140	190

TABLE III

ANALYSIS OF COVARIANCE TABLES FOR THE MEASURED CONCENTRATION

C = True concentration; COL = column; D = detector; Q = method quantification.

Aroclor	*Source of variation*	*Sum of squares*	*Degrees of freedom*	*Mean square*	*F**	*p*
(a) Aroclor 1 (1016)	Actual concentration (covariate)	1,030,222.1415	1	1,030,222.1415	408.36	0.0001**
	COL	595.0208	1	595.0208	0.24	0.6299
	D	23,452.5208	1	23,452.5208	9.30	0.0041**
	Q(COL)	20,997.7083	2	10,498.8542	4.16	0.0230***
	COL × D	3,553.5208	1	3,553.5208	1.41	0.2425
	D × Q(COL)	19,173.5417	2	9,586.7709	3.80	0.0311***
	Error	98,391.0252	39	2,522.8468		
	$R^2 = 0.959$; $\hat{\beta} = 0.77$					
(b) Aroclor 2 (1242)	Actual concentration (covariate)	1,366,889.9732	1	1,366,889.9732	596.72	0.0001**
	COL	652.6875	1	652.6875	0.28	0.5965
	D	2,625.5208	1	2,625.5208	1.15	0.2909
	Q(COL)	3,662.7083	2	1,831.3542	0.80	0.4568
	COL × D	3,350.0208	1	3,350.0208	1.46	0.2338
	D × Q(COL)	4,211.7083	2	2,105.8542	0.92	0.4073
	Error	89,335.8601	39	2,290.6631		
	$R^2 = 0.971$; $\hat{\beta} = 0.92$					

(c) Aroclor 3 (1254)	Actual concentration (covariate)	1,468,334.9511	1	1,468,334.9511	549.27	0.0001**
	COL	6,486.7500	1	6,486.7500	2.56	0.1177
	D	1,408.3333	1	1,408.3333	0.56	0.4605
	Q(COL)	6,470.8333	2	3,235.4167	1.28	0.2904
	COL × D	4,720.3333	1	4,720.3333	1.86	0.1802
	D × Q(COL)	4,977.3333	2	2,488.6667	0.98	0.3837
	Error	98,856.7155	39	2,534.7876		
	$R^2 = 0.965; \hat{\beta} = 1.03$					
(d) Aroclor 4 (1260)	Actual concentration (covariate)	1,037,727.0051	1	1,037,727.0051	567.43	0.0001**
	COL	216.7500	1	216.7500	0.12	0.7325
	D	9,520.3333	1	9,520.3333	5.21	0.0281***
	Q(COL)	7,282.0833	2	3,641.0417	1.99	0.1502
	COL × D	7,056.7500	1	7,056.7500	3.86	0.0566
	D × Q(COL)	18,642.4167	2	9,321.2084	5.10	0.0108***
	Error	71,324.3282	39	1,828.8289		
	$R^2 = 0.971; \hat{\beta} = 1.00$					

* All F values are obtained by dividing the row mean square by the mean square for error.
** Significant at the 0.01 level.
*** Significant at the 0.05 level.

There is no obvious, physical explanation for this difference. There are no significant differences among the average measured concentrations for the four means associated with the packed column. For this Aroclor, $\beta' = 1.00$. Thus, for any given column, detector and method of quantification combination, the measured concentration will increase by 1.00 ppm for each one unit increase in the actual concentration. For Aroclors 2, 3 and 4, the values range from 0.92 to 1.03, which are not significantly different from 1.0, given the normal range of variation encountered in this complex analytical procedure.

The hypothesis testing component of analysis of variance and analysis of covariance procedures is based on the assumption that the variance of the dependent variable is the same at all combinations of the independent variables and/or factors in the model. As there is only one observation for each combination of actual concentration, type of detector, type of column and method of quantification, it is not possible to detect any violation of this assumption. However, if measured concentration values are plotted against actual concentration values, the variation appears to increase as the actual concentration increases. However, residual plots (not shown here) for the four models associated with Table III indicate that the variation is somewhat different at the six actual concentration levels. The differences in the variation of the residuals are related to the fact that there are measured concentrations which are more extreme than the other measurements at that level.

For Aroclor 1 the extreme residuals were at actual concentrations of 441 and 492 ppm. At 441 ppm, the measured value was 540 ppm for the packed/HCD/area combination and 500 ppm for the capillary/HCD/autoquant. At 492 ppm, the measured value was 600 ppm for the packed/HCD/area combination. Note that all three extreme residuals were obtained with the HCD and two of the three were obtained with the packed column.

There was only one extreme residual for Aroclor 2. The measured concentration was 610 ppm for the packed/HCD/area combination while the actual concentration was 402 ppm. The only extreme residual for Aroclor 3 was also associated with the packed/HCD/area combination. The measured concentration was 710 ppm while the actual concentration was 461 ppm.

There were three extreme residuals for Aroclor 4, with two high and one low. The measured concentration was 470 ppm for the capillary/HCD/autoquant combination while the actual concentration was 332 ppm. For the actual concentration of 392 ppm, the measured concentration values for the packed/HCD/height and the capillary/ECD/autoquant combinations were 530 and 210 ppm, respectively.

All but one of these extreme residuals was associated with the HCD. The extreme residuals also were associated with the higher actual concentrations. As the differences in variation in the measured concentrations were associated with outliers, no transformations were used. Further, the extreme residuals were associated with the HCD. Thus, it would be appropriate to investigate possible reasons for this before different statistical models are used.

The most striking feature of the statistical analysis of the data is that commonly held views of which combination of columns, detectors and peak quantitation methods should yield the most accurate results are not supported. For example, the assumption that the use of high-resolution capillary columns should result in more accurate data because there would be more freedom from interference from non-PCB

substances than when packed columns are used is not supported. Further, the assumption that the halogen-specific HCD should yield more accurate results than the ECD (which can respond to many classes of compounds) is not supported. An alternative explanation is that the pre-treatment cleanup procedures (Florisil, alumina, sulfuric acid) have effectively removed all non-PCB species. If that is so, the extra resolution of the capillary column and specificity of the HCD are of no significant consequence. The mechanical complexity of those parts of the gas chromatograph, when compared with the packed column and ECD alternatives, might then lead to less accurate analyses. In addition, the assumption that a computerized data system (using the Autoquant program) should be more capable of yielding accurate results than either a low-cost printer/plotter integrator or a ruler (for peak heights) is not supported.

Within the strict limitations of this study, the conclusion can be drawn that the proper choice of cleanup procedure (Table I)[15] will result in a PCB-containing sample that is sufficiently interference free for the low-resolution, low-specificity packed column-isothermal/ECD–GC procedure to yield accurate results. These results are obtainable at lower instrument, maintenance and analytical cost than would be possible if it were necessary to use capillary-temperature programmed/HCD procedures.

The question of the high extreme residuals observed for all four Aroclors for some of the high-concentration samples when using the HCD may be significant from a legal standpoint. U.S. EPA regulations define a "PCB fluid" as one containing more than 500 ppm and a "PCB-contaminated fluid" as one containing 50–500 ppm of PCBs[20]. It is important that false positives (or negatives) do not occur in this region, as disposal and handling requirements are significantly different depending on these cut-off concentrations.

The statistically significant relationships described in Tables II and III for each PCB are not explainable on a consistent physical basis. However, these individual relationships of factors and cross-terms may be suggestive of further research that is beyond the scope of this effort.

In the above discussion, it is important to note that the instruments were calibrated twice daily, that PCB recoveries were maintained at 80 $\pm$ 20% for each batch, that replicates were maintained within 20% and that primary (EPA-EMSL) standards were used throughout the study. Thus, within the limitations of the experimental design, these conclusions should be valid.

ACKNOWLEDGEMENTS

We thank Paul Flathman, who offered many valuable suggestions during the preparation of this manuscript. We also thank James Dahlgran for his assistance and guidance in the use of the instrumentation. This work was supported by O. H. Materials Co., Findlay, OH, U.S.A.

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