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The detection limit, defined as two times the reagent blank, is 1 pg of Se injected or 5 ng of Se/L seawater, using 200-mL samples. The latter limit could conceivably be lowered by using larger sample volumes.

As discussed previously, Se(VI) does not coprecipitate with hydrous iron oxide to any significant extent. This would rule out simultaneous determination of selenium(VI)—the other major selenium species in seawater—with selenium(IV). The remaining alternative is to perform a Se(VI) reduction prior to preconcentration. However, all existing reduction reactions involve addition of large quantities of acids (3), which is not compatible with the conditions required in the coprecipitation. Work is now progressing toward developing a reduction scheme suitable for coprecipitation with iron(III) oxide.

ACKNOWLEDGMENT

We thank H. A. van der Sloot, The Netherlands Energy Research Foundation, and R. E. Sturgeon and S. N. Willie of this laboratory for the analyses of NASS-1.

Registry No. Se, 7782-49-2; water, 7732-18-5.

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RECEIVED for review February 13, 1984. Accepted April 23, 1984.

Qualitative and Quantitative Analyses of Commercial Polychlorinated Biphenyl Formulation Mixtures by Single Ion Monitoring Gas-Liquid Chromatography/Mass Spectrometry and Multiple Regression

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Polychlorinated biphenyl containing samples are analyzed by a capillary gas-liquid chromatograph/mass spectrometer/data system. Single ion peak area data from test samples and reference Aroclors are analyzed by a multiple regression procedure. Regression parameters obtained are used to determine the Aroclor constituents in test samples as well as quantitative compositional information. For samples which are prepared from the same batches of Aroclors used as references, the coefficients of determination resulting from the adopted regression model are always higher than 99%; total Aroclor concentrations calculated are within 10% of the expected values. A sample prepared from different batches of Aroclors gives a lower (94.7%) coefficient of determination. Extracts from two "weathered" field sediment samples yield coefficients of determination in the range of 87% as a result of differential degradation of PCB components.

Polychlorinated biphenyls (PCBs), produced by the chlorination of biphenyl, are a class of compounds containing 209

possible congeners. In most cases, PCBs are obtained in the form of commercial chlorination products, typical of which are Aroclor 1242, 1248, 1254, and 1260, having 42%, 48%, 54%, and 60% chlorine content, respectively. Three decades of heavy usage of these formulations (banned in 1979) have resulted in the widespread contamination of water and land. Since PCBs exhibit a high degree of biological and chemical stability and lipid solubility, they tend to accumulate in food chains and have even been found in human adipose tissue (1). Since they are a health concern to the public, reliable methods are needed to identify and quantitate these compounds when encountered in the environment.

Structural identifications of PCBs can be done with mass spectrometric (2, 3), nuclear magnetic resonance (2, 3), and infrared (4) spectrometric methods. For the quantitation of PCBs, the current AOAC method, 29.018 (5), recommends the comparison of either *total* or *individual* PCB peak size (electron capture detector response peak area or peak height) of test samples with that of reference Aroclor(s). The comparison of individual peaks provides better results but requires the establishment of the weight percent of each chromato-

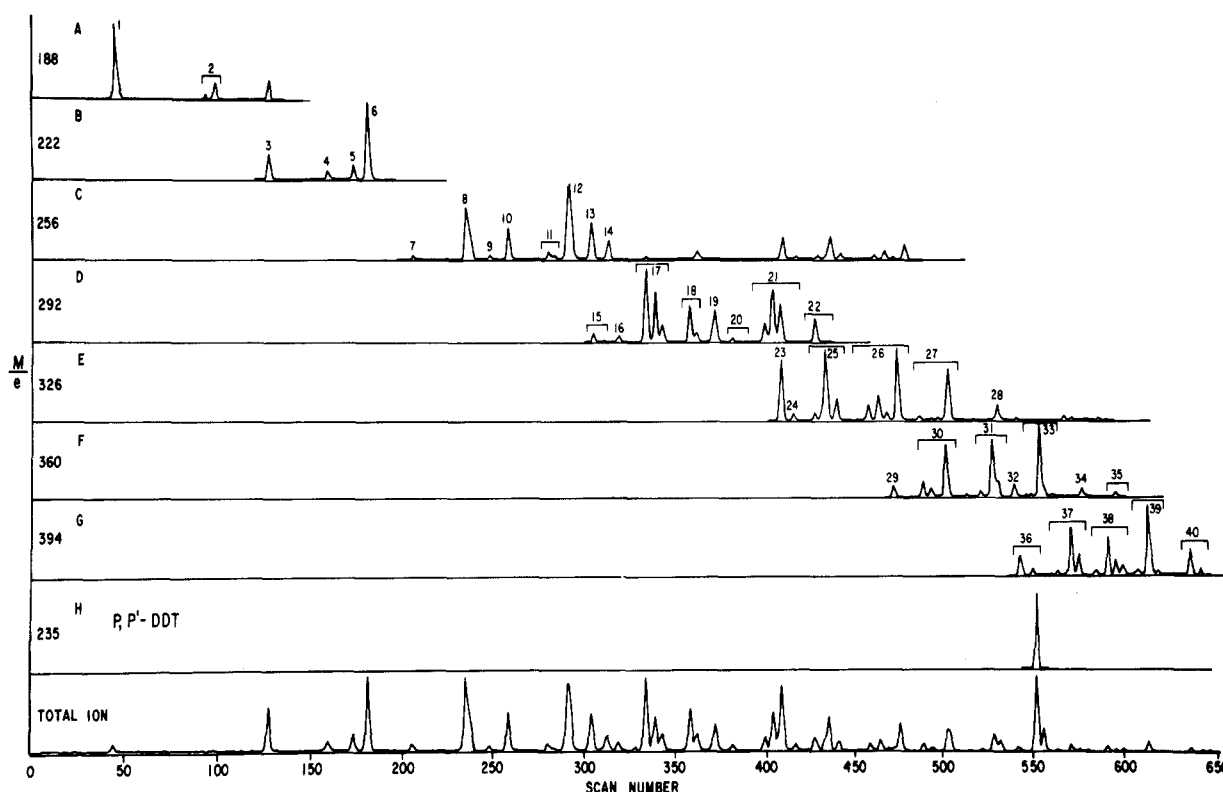


Figure 1. Examples of reconstructed GC/MS ion chromatograms containing Aroclor 1242, 1248, 1254, and 1260.

graphic peak and its response factor in each Aroclor. Accordingly, the analysis of Aroclor mixtures is much more difficult (3) and seldom addressed (6).

Studies reported here use a capillary gas-liquid chromatography/mass spectrometry (GC/MS) approach, with single ion monitoring detection and quantitation, to (1) provide maximum separation and conclusive identification of PCBs and (2) eliminate possible interference arising from pesticides (3, 5, 7). The conventional multiple regression analysis method was used (8) to regress data obtained from test samples on those obtained from Aroclor references. Regression parameters so obtained were used to determine whether the PCBs present in test samples were a nonspecific mixture of PCBs, a specific Aroclor formulation, or a mixture of Aroclors. Furthermore, since weight percents and response factors for individual PCBs are automatically resolved in the regression process, the method also provides quantitative information. This approach provides an easier and more reliable method for the analysis of mixture of Aroclors which are present in most PCB contaminated samples (3). The ability to analyze the presence of specific Aroclor(s) is particularly important when the source of contamination needs to be identified.

This multiple regression analysis approach has been applied to the qualitative analysis of cannabinoids in *Cannabis sativa* L. (9) and the semiquantitative comparison of cannabinoid-containing samples (10). This study extends the approach to the quantitative analysis of Aroclors.

Other multiple regression approaches (8) and pattern recognition methods (11) should be referred for comparison purpose.

EXPERIMENTAL SECTION

Apparatus. A Hewlett-Packard (Palo Alto, CA) HP-5985 GC/MS system equipped with a 30-m (0.25 mm i.d.) J&W Scientific (Orangevale, CA) SE-54 fused silica glass column was used for the separation of PCBs and MS data collection.

Reagents. Aroclor 1242, 1248, 1254, and 1260 were obtained from the US EPA Health Effect Research Laboratory (Research Triangle Park, NC). *p,p'*-DDT was purchased from Supelco (Bellefonte, PA).

Procedure. Standard stock solutions of Aroclor 1242, 1248, 1254, and 1260 were prepared by dissolving appropriate amounts of Aroclor in hexane. The concentration of these stock solutions was around 500 ppm. References and control mixtures of Aroclors were prepared from these standards. The internal standard, *p,p'*-DDT, was prepared as a 1000 ppm stock solution in methylene chloride and added to samples prior to GC/MS analyses. The concentration level of this internal standard in each sample was around 100 ppm.

One-liter of water samples were extracted once with 100 mL of 15:85 (v/v) methylene chloride/hexane and then twice with 60 mL of hexane. Combined extracts were dried with sodium sulfate and evaporated to 1 to 5 mL depending on PCB concentration. Twenty grams of sediment samples was dried and extracted in a Soxhlet apparatus with 200 mL of 1:1 (v/v) acetone/hexane. Extracts were then dried and evaporated to 0.2–5 mL depending on PCB concentration.

A typical GC/MS analysis used 1.25 μ L of sample. GC oven temperature programming was set at 50 $^{\circ}$ C for 1 min, increased 8 $^{\circ}$ C/min for 9 min, and then 6 $^{\circ}$ C/min for 25 min and held at the final temperature (270 $^{\circ}$ C) for 10 min. Helium, linear velocity 40 cm/s, was used as the carrier gas. The mass spectrometer was operated in electron impact mode at 70 eV, with source temperature maintained at 200 $^{\circ}$ C and mass scanning range set from m/z 45 to m/z 450.

Stepwise regression analysis (12) was performed with the SPSS batch system (version H, release 9.1) implemented on an IBM 4341-2 computer, University of Alabama in Birmingham.

RESULTS AND DISCUSSION

Single Ion Chromatograms. Sample single ion and total ion reconstructed chromatograms of a standard mixture of Aroclor 1242, 1248, 1254, and 1260 are shown in Figure 1. Peaks labeled in these chromatograms represent mono- (Figure 1A), di- (1B), tri- (1C), tetra- (1D), penta- (1E), hexa- (1F), and heptachlorinated (1G) biphenyl components adopted for data reduction. Typical mass spectra of these PCB isomers are shown in Figure 2. For those PCB isomers which were not adequately resolved under the adopted chromatographic conditions, peak aggregates were used for area measurements. With the SE-54 column used, some PCB isomers elute earlier than some PCBs having one less chlorine. For example, peak

Table I. Single Ion Data

reference Aroclors (ppm)				samples						peak no.	<i>m/z</i>	retention time, ^a min
1242 (178)	1248 (166)	1254 (243)	1260 (190)	I	II	III	IV	V	VI			
364	79	0	0	391	124	308	144	85	50	1	188	16.4
67	12	0	0	74	25	85	11	12	4	2		17.9
1226	140	0	0	1034	331	1051	407	158	120	3	222	18.7
339	28	0	0	254	62	269	108	27	30	4		19.6
744	65	0	0	602	186	639	219	81	70	5		20.0
4109	528	7	28	3490	1101	3543	1234	441	337	6		20.2
164	24	0	0	140	12	153	54	15	6	7	256	21.0
3309	1360	10	15	3597	1472	3493	1247	1225	945	8		21.8
117	4	0	0	100	17	65	16	6	7	9		22.2
1391	519	14	0	1529	568	1447	602	453	357	10		22.5
584	140	0	0	429	132	499	182	118	60	11		23.1
4595	2694	67	24	5626	2370	5620	2061	2189	1880	12		23.4
1832	727	7	6	1916	716	1860	722	565	500	13		23.7
803	396	5	0	851	353	975	315	317	240	14		24.0
225	242	28	0	289	93	265	111	135	83	15	292	23.8
173	175	0	0	211	66	212	71	71	80	16		24.2
1886	3199	1747	48	4829	2888	5228	1381	4102	3304	17		24.7
856	1468	419	0	1886	1149	1901	620	1469	1195	18		25.3
842	1561	154	0	1651	829	1573	551	1094	956	19		25.7
119	138	28	0	115	71	115	26	53	29	20		26.0
1743	3774	1254	18	4925	2696	4745	2176	3794	3172	21		26.7
461	966	73	0	1062	507	1057	461	769	684	22		27.4
109	390	1259	351	1655	1020	1755	266	1456	1246	23	326	26.7
24	93	130	0	145	113	188	43	187	132	24		27.0
199	844	2408	475	3285	2087	3453	842	3016	2557	25		27.5
208	1050	2641	173	3661	2248	3536	1000	3805	3093	26		28.4
75	435	1194	51	1718	1031	1812	788	1700	1518	27		29.3
40	185	322	2	497	310	546	326	522	455	28		30.3
0	4	57	184	196	105	223	45	69	68	29	360	28.6
0	22	553	1279	1512	671	1426	324	654	461	30		29.2
0	36	662	1100	1568	731	1592	403	799	631	31		30.2
0	45	50	168	215	80	203	43	70	52	32		30.5
0	0	746	698	1498	726	1476	364	931	788	33		31.0
0	0	91	3	94	77	97	33	116	110	34		31.7
0	0	41	3	85	51	56	35	33	33	35		32.2
0	0	0	187	159	30	133	33	0	0	36	394	30.7
0	0	14	485	423	117	399	107	17	22	37		31.4
0	0	5	377	355	95	345	76	16	14	38		32.0
0	0	25	517	472	129	473	123	39	25	39		32.6
0	0	7	153	154	50	147	34	24	15	40		33.4
5607 ^b	9000	4644	6424	5000	6116	5792	4611	6292	5388		235	30.9

^a See text for gas-liquid chromatographic conditions. ^b The single ion (*m/z* 235) intensity corresponding to 125 µg of the internal standard, *p,p'*-DDT, in each injection.

no. 15 is a tetrachlorobiphenyl, but it elutes before peak no. 14 which is a trichlorobiphenyl.

Multiple Regression Analysis. Table I lists the areas of the 40 single ion peaks and peak aggregates (Figure 1) obtained from one set of reference Aroclors and test samples. These data were used for multiple regression analysis as described below. It is first assumed that for a specific PCB in a sample, its peak area is the sum contributed by this compound in Aroclor 1242, 1248, 1254, and 1260. Since the concentrations of each PCB in unit concentration of different Aroclor vary, intensities of these 40 peaks in a sample are related to those observed in reference Aroclors as formulated below:

$$\begin{aligned}
 Y_1 &= X_{1,1242}C_{1242} + X_{1,1248}C_{1248} + X_{1,1254}C_{1254} + X_{1,1260}C_{1260} \\
 Y_2 &= X_{2,1242}C_{1242} + X_{2,1248}C_{1248} + X_{2,1254}C_{1254} + X_{2,1260}C_{1260} \\
 &\vdots \\
 Y_{40} &= X_{40,1242}C_{1242} + X_{40,1248}C_{1248} + X_{40,1254}C_{1254} + X_{40,1260}C_{1260}
 \end{aligned}
 \quad (1)$$

where Y_n ($n = 1, 2, 3, \dots, 40$) is the intensity of peak n observed in the test sample, $X_{n,m}$ ($m = 1242, 1248, 1254, \text{ or } 1260$) is the intensity of peak n obtained in unit concentration of reference Aroclor m , and C_m is the concentration of Aroclor m in the test sample.

For the purpose of this study, eq 1 is set for only four Aroclors, but it can be extended to include other Aroclors.

Table II lists regression analysis results obtained from data listed in Table I and other experiments.

Assignment and Quantitation of Aroclors. Samples I–III, V, and VI were prepared by mixing reference Aroclors used for the establishment of reference Aroclor 1242, 1248, 1254, and 1260 chromatographic patterns, shown in the first four columns in Table I. Test sample IV was prepared by using old stock solutions from different lots of Aroclors. Samples I to IV contain all four Aroclors. Data obtained from samples I and II correlate very well with the reference Aroclors to give high coefficients of determination (higher than 99%) and small constant terms. The concentrations calculated from the data in Table I also agree very well with expected values shown in parentheses. The lower concentrations obtained from sample III indicate either the loss of PCBs or incomplete extraction. The notable deviation of sample IV probably reflects a difference in stock solution preparation. Sample

Table II. Results of Multiple Regression Analysis

sample no.	sample description	Aroclor concn; standard error/ppm					coeff of detm/%	const term ^a
		1242	1248	1254	1260	sum		
I ^b	control mixture	163; 4.5 (178) ^c	173; 9.5 (166)	219; 7.9 (243)	202; 16 (190)	757 (777)	99.5	-7.5
II ^b	control mixture	41.1; 1.7 (44.4)	98.2; 3.6 (104)	121; 3.0 (152)	53.9; 6.1 (47.4)	314 (348)	99.6	-17
III ^b	control mixture (ext. from water)	142; 3.3 (185)	146; 7.0 (173)	200; 5.8 (253)	164; 12 (197)	652 (808)	99.7	-6.7
IV ^d	control mixture	56.4; 5.7 (92.2)	91.9; 12 (86.7)	51.5; 10 (78.1)	43.6; 21 (52.8)	243 (311)	94.7	20
V ^b	control mixture	[11.3] ^e	166; 4.7 (150) (163)	191; 4.8 (199) (238)		357 (360) (357)	99.3 99.5	21 -3.4
VI ^b	control mixture (ext. from water)	[9.44]	161; 45 (147) (163)	185; 4.6 (192) (238)		346 (348) (401)	99.3 99.5	16 -4.2
VII ^b	control mixture		108; 3.1 (110)	183; 3.5 (180)		291 (290)	99.5	11
VIII ^b	control mixture		230; 3.5 (221)	91.0; 3.2 (86.4)		321 (307)	99.7	-17
IX ^d	field sediment (extract)		35.7; 6.2	72.7; 7.1		108	87.8	-0.27
X ^d	field sediment (extract)		76.5; 16	151; 15		228	87.4	18

^a The constant term represents the single ion intensity when no Aroclor is present in the sample. The small values resulting from these analyses indicate they are insignificant. ^b Aroclors in these samples and Aroclors in standards are from the same batches. ^c Numbers inside parentheses are expected values. ^d Aroclors in these samples and Aroclors in standards are from different batches. ^e Numbers in brackets are results obtained from regression on four Aroclors.

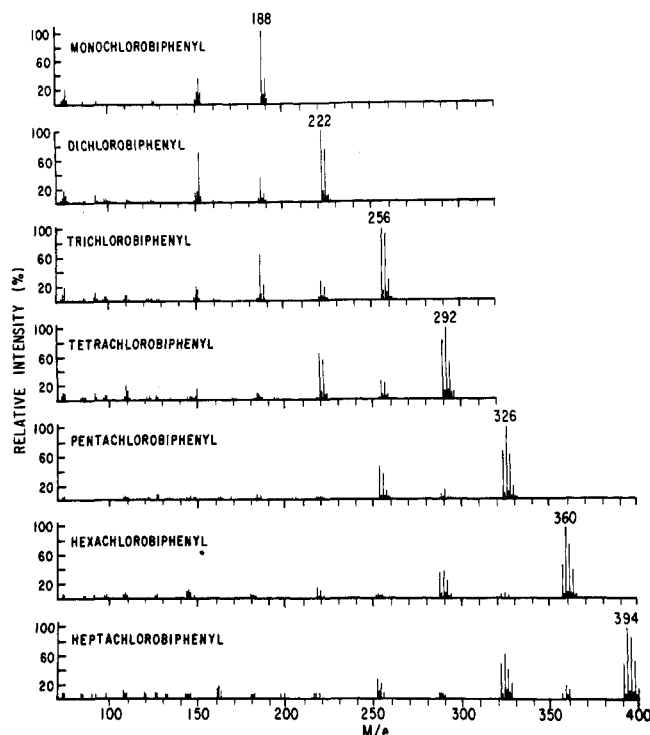


Figure 2. Typical 70-eV EI mass spectra of mono-, di-, tri-, tetra-, penta-, hexa-, and heptachlorinated biphenyls, corresponding to peaks 1, 3, 10, 17 (first peak), 25 (middle peak), 31 (middle peak), and 39 (middle peak), respectively, in Figure 1.

IV was prepared from a set of standards with unknown history.

Samples IV and V were known to contain only Aroclor 1248 and 1254, regression analyses were performed based on only these two Aroclors, and good results were obtained. However, a more objective test of the method would be to perform the regression analysis on these samples using the data of all four Aroclors. In this instance stepwise regression analysis of V and VI indicated that Aroclor 1260 data could not meet the 0.50 significance level for entry into the model. Likewise the

use of Aroclor 1242 data did not improve the "goodness of fit" significantly and resulted in a very low concentration of Aroclor 1242 with respect to the concentrations of Aroclors 1248 and 1254 (shown in brackets). From these results one may conclude that Aroclor 1260 and 1242 are not making significant contributions in these samples. Visual inspection of the chromatographic pattern would not necessarily make this conclusion apparent. It is also interesting to examine the quantitative results obtained from the regression analysis of V and VI. The fact that almost identical sums of PCB concentration are obtained from the use of two (Aroclor 1248 and 1254) or four (Aroclor 1242, 1248, 1254, and 1260) variables certainly will satisfy most analytical situations in which only total PCB concentration is needed.

Replicates and Applications. Samples I and II are duplicates in different concentration levels. Although the concentrations of Aroclor 1242 and 1260 in sample II are about one-fourth of that in sample I and in the standards, results obtained are satisfactory. Extensive linearity study was not conducted, but it is recommended that the final volume of the sample extract should be adjusted to provide comparable concentration levels with those of standards. Samples VII and VIII represent two additional sets of experiments performed at different dates. These are truly independent replicates of sample V in terms of samples and standards. Replicates were conducted in this fashion to obtain maximum statistical significance (13).

Regression analysis on samples VII and VIII was performed with two variables (reference Aroclor 1248 and 1254) only. Good results were obtained for both Aroclor constitutions and total PCB levels. Since reference Aroclor 1242 and 1260 were not analyzed along with samples VII and VIII, regression analysis of these two samples based on four variables was not possible.

Field sediment samples IX and X with unknown history gave lower coefficients of determination. This indicates a deviation of chromatographic patterns from that of Aroclor references, presumably as a result of differential partial degradation of PCBs reported in the literature (3, 14). These data lead to an interesting question—at what coefficient of

determination value should one consider the test sample as a specific combination of Aroclors, rather than a nonspecific mixture of PCBs? Obviously, PCB metabolism and degradation patterns have to be systematically studied before a reliable conclusion can be reached.

Quantitatively speaking, results obtained from field sediment samples IX and X are very comparable with those obtained by EPA procedure (15) using an electron capture detector. With sample weight and dilution factor taken into consideration, the concentrations obtained from this study are equivalent to 3.98 (Aroclor 1248) and 8.11 ppm (Aroclor 1254) for IX; and 290 (Aroclor 1248) and 540 ppm (Aroclor 1254) for X in raw samples. The routinely used EPA procedure could not deconvolute Aroclor formulations and reported the presence of Aroclor 1254 in the amounts of 29 and 621 ppm for samples IX and X, respectively.

It should be noted that the concentrations of PCBs in the standards and controls used for GC/MS analysis are about 1000 times higher than those encountered in real environmental water samples. Samples III and IV are extracts obtained from 1 L of water and concentrated to 1 mL; thus the PCB concentration levels resemble actual samples. The PCB concentrations in the two field sediments are comparable with those commonly encountered. Furthermore, since the sample size and the final volume of the extract can be adjusted before submitting to GC/MS analysis, the quantitative aspect of this approach should be valid when applied to environmental samples.

CONCLUSIONS

Multple regression analysis using single ion GC/MS data of reference Aroclors and test samples provides a convenient and reliable method of PCB analysis. The regression process resolves the need for the determination of PCB weight percentages in Aroclors and their response factors. The validity of this approach is proven by the following: (1) the high values of coefficient of determination and small constant terms indicate that the right model is being used for the data reduction; (2) the successful use of statistical inference procedures (F ratio) as an indicator for rejecting unjustified Aroclors from entering the model demonstrates that this approach can be used for qualitative Aroclor analysis; (3) except for sample III, where loss of Aroclor in the extraction process is apparent,

almost all expected concentration values fall within two standard errors of the regression coefficients, indicating valid quantitative Aroclor analysis. The ability to analyze specific Aroclor(s) provides valuable information that is needed to identify possible sources of contamination for samples that have not significantly degraded. However, in order to make conclusive judgements on badly degraded samples, additional studies to determine how the coefficient of determination is affected by differential degradation and/or metabolism of PCBs are needed.

ACKNOWLEDGMENT

The authors thank Edwin G. Piotrowski, John G. Phillips, Thomas K. Folgia, Chacko T. Joseph, Thomas H. Kim, and Gilbert Frye for helpful discussion and E.G.P., J.G.P., and T.A.F. for their critical reading of the manuscript.

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RECEIVED for review February 17, 1984. Accepted May 1, 1984. Small portions of this work were done while at the Eastern Regional Research Center, U.S. Department of Agriculture, and at the Central Regional Laboratory, EPA. Preliminary multiple regression analysis was done using the computer facilities at the University of Illinois at Chicago.