

Use of Isotopic Abundance Ratios in Identification of Polychlorinated Biphenyls by Mass Spectrometry

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Polychlorinated biphenyls are industrial compounds which are being detected throughout the global ecosystem. Because of the presence of other residues, combined gas chromatography-mass spectrometry must be used for their confirmation. Using isotopic abundance ratios, the theoretical probability of the occurrence of ions of different masses in the molecular cluster has been calculated for the polychlorinated biphenyls, polychlorinated terphenyls, and chlorinated naphthalenes. Mass spectra of mono- through decachlorobiphenyl have been taken with a unit resolution, computer-controlled GC-MS system and parent ion clusters were matched with the theoretical isotopic patterns. This method provides the unambiguous identification of polychlorinated biphenyls in the presence of other substances.

Chlorinated hydrocarbons other than insecticide residues were detected in environmental samples in the mid-1960's (1, 2). Since polychlorinated biphenyl (PCB) compounds and common chlorinated insecticides have similar retention times (3), unknown peaks could not be identified by gas-liquid chromatography (GLC) alone. Jensen (4) and Widmark (5), using combined gas chromatography-mass spectrometry (GC-MS), provided the first analytical evidence for the presence of PCB's in environmental samples. Since then, several laboratories have used GC-MS to confirm the presence of PCB's in a range of organisms (6-9).

Several workers have recently used GC-MS to characterize the PCB components in technical mixtures (10-15), and a method for measuring PCB isomers has been based on MS identification of the components in commercial Aroclors (16). Safe and Hutzinger have used MS to confirm photolysis of a hexachlorobiphenyl isomer (17) and to show chlorine randomization in PCB's prior to fragmentation (18). The same workers have obtained the most informative data on the mass spectra of PCB's to date (19).

Mass spectra are available for most of the com-

mon organochlorine pesticides and fungicides which might be present in samples containing PCB's (20-24). Polychlorodibenzo-p-dioxins and polychlorodibenzofurans, toxic minor components in some commercial PCB's and chlorophenol fungicides, can be distinguished by MS if they are present in samples (25). Mass spectra are also available for chlorinated naphthalenes (CN) (26), polychlorinated terphenyls (PCT) (11), and phthalate esters (27), all of which interfere in PCB analysis (28-30).

We describe a method which provides unambiguous identification of PCB's in the presence of the above interfering substances. The mass spectra of PCB's, PCT's, and CN's are characterized by a strong parent ion cluster with an isotopic abundance ratio determined by the number of chlorine atoms substituted on the rings. Although most workers using MS are probably aware of these isotopic clusters, no one to date has compared theoretical ion intensities to observed spectra. Uk et al. (21) calculated the intensities of chlorine isotope peaks for up to 12 chlorine atoms, but offered no spectra for comparison.

From the natural abundance ratios of chlorine and carbon isotopes (31), we have calculated the theoretical probability of the occurrence of ions of different masses in the molecular cluster for all degrees of chlorination in CN's, PCB's, and PCT's. Using a computer-controlled GC-MS system, we have taken the mass spectra of mono- through decachlorobiphenyl and have compared the observed isotopic abundance ratios to the theoretical values. The matching of PCT and CN ratios is the subject of another paper in preparation.

METHOD

Apparatus

The gas chromatograph-mass spectrometer employed was a Finnigan Model 1015 C quadrupole with a glass column and glass jet separator. The GC-MS was controlled by a Maxam Industries 160 computer. The details of this entire unit have been described (32).

Specific in this study are approximately multiplier 2; temperature; The values were used: n OV-1 on 60-8 temperature 245-260 approximately; grammae as d

Materials

Technical A (anto) were d 0.5 µg/al for Individual ion 2,2'-di- (H. W 2,4,5,2',4',5'-h phenyl. The prepared in a chlorination e chloride (33).

Gas Chroma

Our efforts a spectrum for PCB's. Where few grains of) by solid probe spectrometer. by MS scanion of Aroclor

Depending a temperatures : lows: Aroclor 1 240°C; Aroclor 1200°C; for each of isomers was : tion of Aroclor

In all cases, a computer to a chlorinated spigital output : most intense le:

Calculation of Abundances

The theoretic molecular clus: natural abundan: The probability for ¹³C and ¹⁸O abundances, the pe: ¹³C, one carbon atom being ¹³C ated naphthalen

Specific mass spectrometric conditions used for this study are as follows: ion source temperature approximately 125°C; electron energy 70 eV; electron multiplier 2.8 kV; transfer line 240°C; separator temperature 250°C.

The following gas chromatographic conditions were used: column 5' X 2 mm id packed with 3% OV-1 on 60/80 mesh Supelcoport; injector temperature 245–260°C; carrier gas helium, flow rate approximately 25 ml/min; column temperature programmed as described below.

Materials

Technical Aroclor® 1232, 1254, and 1268 (Monsanto) were dissolved in hexane to concentrations of 0.5 µg/µl for GC separation prior to MS analysis. Individual isomers analyzed directly by MS include: 2,2'-di- (R. Webb), 2,5,2',5'-tetra- (O. Hutzinger), 2,4,5,2',4',5'-hexa- (C. Engel), and dechlorobiphenyl. The completely substituted biphenyl was prepared in one of our laboratories by exhaustive chlorination of Aroclor 1268 with antimony pentachloride (33).

Gas Chromatography-Mass Spectrometry

Our efforts were directed at obtaining a good mass spectrum for each degree of chlorination in the PCB's. Where individual isomers were available, a few grains of pure crystal were introduced directly by solid probe into the ionizing chamber of the mass spectrometer. The remaining spectra were obtained by MS scanning of selected peaks after GC separation of Aroclor mixtures.

Depending on the Aroclor being separated, column temperatures were programmed at 4°/min as follows: Aroclor 1232, 150–220°C; Aroclor 1254, 170–240°C; Aroclor 1268, 190–250°C. Total amount injected for each Aroclor was 0.5–1.0 µg. Identification of isomers was assisted by previous MS characterization of Aroclor mixtures (10–15).

In all cases, a graphical output was plotted by the computer to show the entire spectrum of each chlorinated species. The computer also provided a digital output of each molecular cluster, with the most intense ion normalized to 100%.

Calculation of Theoretical Isotopic Abundances

The theoretical relative abundances of ions in the molecular cluster were determined by using the natural occurrence of carbon and chlorine isotopes. The probabilities of carbon isotopes used are 0.989 for ^{12}C and 0.011 for ^{13}C (34). From these abundances, the probability of all carbon atoms being ^{12}C , one carbon atom being ^{13}C , and two carbon atoms being ^{13}C can be determined for the chlorinated naphthalenes (CN), polychlorinated biphenyls

(PCB), and polychlorinated terphenyls (PCT). The CN molecules have 10 carbon atoms and therefore the probability of all being ^{12}C = 0.989, one ^{13}C = 0.101, and two ^{13}C = 0.005. PCB molecules have 12 carbon atoms, giving a probability of all being ^{12}C = 0.874, one ^{13}C = 0.118, and two ^{13}C = 0.007. PCT molecules have 18 carbon atoms, giving a probability of all being ^{12}C = 0.817, one ^{13}C = 0.166, and two ^{13}C = 0.016.

The probabilities of chlorine isotopes used are 0.754 for ^{35}Cl and 0.246 for ^{37}Cl (31). The occurrence of ^2H was not considered in the calculations because of its small natural abundance.

The theoretical values were computed on a WANG Model 360 electronic calculator for CN's having from 1 to 8 chlorine atoms, for PCB's having from 1 to 10 chlorine atoms, and for PCT's having from 1 to 14 chlorine atoms per molecule. A binomial expansion was used to determine coefficients, and the various combinations of chlorine and carbon isotopes were worked into the probability formula used by Beynon (31) and Uk *et al.* (21). The theoretical abundances are computed for M^+ , $M^+ + 1$, $M^+ + 2$, etc., and are expressed as the corresponding *m/e* for each chemical species. The values are expressed relative to the most intense ion in the molecular cluster, with the parent ion always being the lowest *m/e* in the group. Table 1 gives the parent ion (M^+) and most intense ion for all chlorinated species of CN's, PCB's, and PCT's.

Results and Discussion

Table 2 presents the theoretical probability of the occurrence of ions in the molecular cluster for CN's, PCB's, and PCT's. As the probability of the occurrence of ^{37}Cl increases with increasing degree of chlorination, the differences between intensities in the clusters become less pronounced. This is accompanied by a shift of the most intense ion in the cluster from the parent ion (M^+) to $M^+ + 2$, then to $M^+ + 4$, and finally, in the higher chlorinated terphenyls, to $M^+ + 6$. These patterns of intensity are unique for each chemical species, thus providing a ready means of identification.

This becomes evident when the observed *m/e* intensities of mono- through dechlorobiphenyl (Table 3) are compared to the theoretical PCB values. The observed ratios in the molecular cluster are nearly a perfect match with that expected for each PCB. The only places where there are noticeable deviations from the expected values are seen at *m/e* 253 in tri-; *m/e* 466 in nona-; and *m/e* 494, 498, and 500 in deca-. An explanation for this may be that these spectra

Table 1. Parent ion and most intense ion for CN's, PCB's, and PCT's

Chlorinated naphthalenes (CN) C ₁₀ H ₆ Cl ₂ -Cl ₈			Polychlorinated biphenyls (PCB) C ₁₂ H ₆ Cl ₂ -Cl ₁₀			Polychlorinated terphenyls (PCT) C ₁₈ H ₆ Cl ₂ -Cl ₁₆		
No. of Cl	M ⁺	Most Intense ^a	No. of Cl	M ⁺	Most Intense	No. of Cl	M ⁺	Most Intense
0	128		0	154		0	230	
1	162		1	188		1	264	
2	196		2	222		2	298	
3	230		3	256		3	332	
4	264	(266)	4	290	(292)	4	366	(368)
5	298	(300)	5	324	(326)	5	400	(402)
6	332	(334)	6	358	(360)	6	434	(436)
7	366	(368)	7	392	(394)	7	468	(470)
8	400	(404)	8	426	(430)	8	502	(506)
			9	460	(464)	9	536	(540)
			10	494	(498)	10	570	(574)
						11	604	(608)
						12	638	(644)
						13	672	(678)
						14	706	(712)

^a Parent ion is most intense unless otherwise indicated in parentheses.

were taken while the concentrations of the compounds were changing in the ion source, thus affecting ion intensities. Even with these deviations, a chi-square test shows the matching of observed to expected intensities to be significant at the 99% level for all PCB's.

Table 2. Theoretical probability of the occurrence of ions in the molecular cluster in CN's, PCB's, and PCT's^a

m/e	CN	PCB	PCT
162	mono		
163	100.00		
164	11.28		
165	3.68		
188		mono	
189		100.00	
190		13.53	
191		33.44	
191		4.41	
196	di		
197	100.00		
198	11.28		
199	65.77		
199	7.25		
200	11.00		
201	1.20		
222		di	
223		100.00	
224		13.53	
224		66.04	
225		8.82	
226		11.17	
227		1.44	
230	tri		
231	100.00		
232	11.27		
232	98.37		
233	11.03		
234	32.44		

(Continued)

Table 2. (Continued)

m/e	CN	PCB	PCT
235	3.59		
236	3.65		
237	0.39	tri	
256		100.00	
257		13.53	
258		98.64	
259		13.23	
260		32.70	
261		4.31	
262		3.73	
263		0.47	
264	tetra		mono
265	76.35		100.00
265	8.61		20.30
266	100.00		34.55
267	11.23		6.62
268	49.25		0.63
269	5.49		
270	10.86		
271	1.19		
272	0.92		
290		tetra	
291		76.20	
291		10.31	
292		100.00	
293		13.44	
294		49.42	
295		6.57	
296		10.97	
297	pent	1.43	di
298	61.14	0.95	100.00
299	6.83		20.30
300	100.00		67.15
301	11.24		13.73
302	65.54		11.90
303	7.33		2.16
304	21.55		
305	2.39		
306	3.57		
307	0.39		
308	0.24		

(Continued)

Table 2. (Continued)

m/e	CH	PCB	PCT
<i>penta</i>			
374		61.04	
375		8.76	
376		100.00	
377		13.46	
378		65.70	
379		0.78	
380		21.69	
381		2.66	
382	<i>hexa</i>		<i>tri</i>
383		50.98	100.00
384		5.75	20.29
385		100.00	99.74
386		11.24	19.85
387		81.83	32.79
388		9.16	6.47
389		35.79	4.09
390		3.98	0.70
391		8.84	
392		0.97	
393		1.17	
<i>hexa</i>			
394		50.91	
395		6.89	
396		100.00	
397		13.47	
398		81.98	
399		10.98	
400		35.95	
401	<i>hepta</i>	4.77	<i>tetra</i>
402		43.71	75.56
403		4.53	15.34
404		100.00	100.00
405		11.25	19.99
406		98.12	50.10
407		11.00	9.78
408		53.56	11.41
409		9.98	2.13
410		17.58	1.06
411		1.95	0.17
412		3.48	
413		0.38	
414		0.39	
<i>hepta</i>			
415		43.66	
416		5.91	
417		100.00	
418		13.49	
419		98.28	
420		13.18	
421		53.76	
422	<i>octa</i>	7.16	<i>penta</i>
423		33.44	60.63
424		3.77	12.30
425		87.40	100.00
426		8.83	20.06
427		100.00	66.35
428		11.72	13.08
429		65.44	22.26
430		7.31	4.26
431		26.81	1.83
432		2.98	0.69
433		7.05	0.29
434		0.78	
435		1.16	
436		0.13	

hepta (PCT)
pentaMost
Intense(368)
(402)
(436)
(470)
(506)
(540)
(574)
(608)
(644)
(678)
(712)

PCT

mono
100.00
70.30
34.55
6.67
0.63di
100.00
70.30
57.19
13.23
11.90
2.16

(Continued)

Table 2. (Continued)

m/e	PCB	PCT
426	33.36	
427	4.51	
428	87.78	
429	11.77	
430	100.00	
431	13.43	
432	65.56	
433	9.76	<i>hexa</i>
434	26.92	50.62
435	3.57	10.28
436	7.10	100.00
437	0.93	70.10
438	1.18	82.62
439	0.15	16.38
440	0.11	36.65
441		7.12
442		9.26
443		1.74
444		1.99
445		0.23
446		0.08
<i>nona</i>		
460	25.87	
461	3.51	
462	76.42	
463	10.31	
464	100.00	
465	13.44	
466	76.41	
467	10.23	<i>hepta</i>
468	37.59	43.45
469	5.00	8.82
470	12.36	100.00
471	1.63	20.12
472	2.72	98.90
473	0.35	19.68
474	0.39	54.57
475		10.69
476		18.20
477		3.49
478		3.69
479		0.68
480		0.43
<i>deca</i>		
494	20.79	
495	2.81	
496	67.96	
497	9.17	
498	100.00	
499	18.45	
500	87.27	
501	11.70	<i>octa</i>
502	50.04	33.04
503	6.67	6.71
504	19.71	86.82
505	2.61	17.49
506	9.48	100.00
507	0.71	19.96
508	1.02	66.02
509	0.13	13.01
510		27.37
511		5.30
512		7.32
513		1.38
514		1.24
515		0.22
516		0.12

(Continued)

(Continued)

Table 1. (Continued)

m/e	PCT
536	none
537	25.75
538	5.23
539	76.06
540	15.34
541	100.00
542	20.00
543	76.87
544	15.21
545	38.11
546	7.44
547	12.66
548	2.42
549	2.83
549	0.53
550	0.41
570	deca
571	20.54
572	4.19
573	67.68
574	13.55
575	100.00
576	20.03
577	87.72
578	17.41
579	50.52
580	9.93
581	20.10
582	3.89
583	5.58
584	1.06
585	1.07
586	0.70
587	0.14
588	undeca
589	16.91
590	3.43
591	60.96
592	12.30
593	100.00
594	20.06
595	98.57
596	19.61
597	64.89
598	12.79
599	29.99
600	5.84
601	9.93
602	1.90
603	2.36
604	0.44
605	0.40
606	dodeca
607	12.09
608	2.61
609	50.67
610	10.23

(Continued)

Table 2. (Continued)

m/e	PCT
642	91.39
643	38.35
644	100.00
645	19.94
646	73.97
647	14.62
648	38.99
649	7.63
650	15.02
651	2.90
652	4.27
653	0.81
654	0.89
655	0.15
656	0.14
672	trideca
673	9.93
674	2.01
675	42.78
676	8.54
677	83.14
678	16.71
679	100.00
680	19.97
681	82.11
682	16.78
683	40.52
684	9.55
685	21.37
686	4.15
687	7.06
688	1.35
689	1.76
690	0.33
691	0.33
706	tetradeca
707	7.81
708	1.58
709	35.81
710	7.24
711	76.25
712	15.34
713	100.00
714	20.00
715	90.25
716	17.93
717	59.31
718	11.69
719	29.78
720	5.72
721	11.04
722	7.13
723	3.20
724	0.61
725	0.71
726	0.13

* Probabilities used: $^{12}\text{C} = 0.989$, $^{13}\text{C} = 0.011$; $^{35}\text{Cl} = 0.754$, $^{37}\text{Cl} = 0.246$.

The isotopic patterns are better seen in the plotted spectra (Figs. 1-10). To save space, only the area of the molecular cluster and the major fragmentation ions are shown. The spectra show the molecular cluster always to be the most in-

tense with the loss of even numbers of chlorine atoms favored upon fragmentation.

MS identification of PCB's will not be affected by organochlorine isocoumarins as they have none parent ions, relative to the PCB's, and can be

Table 1. Observed *m/e* intensities in the molecular cluster of PCB's*

<i>m/e</i>	Intensity	<i>m/e</i>	Intensity
mono			
188	100.00	395	13.02
189	13.53	396	96.09
190	33.36	397	13.08
191	4.34	398	53.07
di			
222	100.00	399	6.51
223	78	400	16.51
224	44.84	401	7.12
225	8.59	402	3.14
226	10.54	403	6.45
227	1.95	404	0.30
tri			
256	100.00	426	34.36
257	13.45	427	4.56
258	92.61	428	88.10
259	12.13	429	11.53
260	29.81	430	100.00
261	3.43	431	13.43
262	3.16	432	65.33
263	0.26	433	8.73
tetra			
290	77.14	434	26.31
291	10.44	435	3.39
292	100.00	436	6.99
293	13.67	437	0.87
294	48.84	438	1.16
295	6.82	439	0.11
296	10.84	440	0.11
297	1.50	penta	
298	0.96	460	25.89
penta			
324	61.84	461	3.48
325	7.85	462	75.55
326	100.00	463	10.01
327	12.96	464	100.00
328	66.33	465	13.27
329	8.72	466	72.07
330	21.94	467	9.69
331	2.99	468	35.93
332	3.86	469	4.95
333	0.37	470	11.59
334	0.24	471	1.42
hexa			
358	51.12	472	2.43
359	6.83	473	0.75
360	100.00	474	0.33
361	13.32	hepta	
362	96.95	494	25.59
363	10.35	495	3.34
364	35.61	496	78.55
365	4.65	497	9.84
366	6.71	498	100.00
367	1.21	499	14.28
368	1.17	500	92.42
369	0.15	501	11.64
hepta			
392	43.78	502	51.58
393	5.90	503	6.86
394	100.00	504	20.28
		505	2.71
		506	5.42
		507	0.75
		508	1.04
		509	0.14

* 2-mono; 2,2'-di; 2,4,4'-tri; 2,5,2',5'-tetra; 2,4,5,2',5'-penta; 2,4,5,2',4',5'-hexa; 2,4,4,6,2',5'-hepta; 2,3,4,6,2',5'-octa; 2,1,4,5,6,2',5',4',5'-nona; decachlorobiphenyl.

detected by their characteristic base peaks (20-23). Chlorobiphenyls and chlorodiphenyls can easily be distinguished by their mass spectra, both usually having a C₁₂-C₁₂ fragment upon ionization. O-Phthalate esters have characteristic spectra with a base peak usually at *m/e* 149 and have no isotopic cluster (27).

As more laboratories turn to GC-MS analysis of environmental pollutants, the use of isotopic abundance ratios will become an increasingly more valuable aid to identification.

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REFERENCES

- (1) Bohm, J. (1965) *Analyst* 90, 467-475.
- (2) Robinson, J., Richardson, A., Crabtree, A., Coulson, J., & Potts, G. (1967) *Nature* 214, 1307-1311.
- (3) Reynolds, L. (1969) *Bull. Environ. Contam. Toxicol.* 4, 128-143.
- (4) Jensen, S. (1966) *Acc Sci.* 32, 612.
- (5) Widmark, G. (1967) *JAOAC* 50, 1065.
- (6) Koeman, J., ten Noer de Braw, M., & de Vos, R. (1969) *Nature* 221, 1126-1128.
- (7) Muthers, B., Reichel, W., Loeke, J., Lamont, T., Belisle, A., Cromartie, E., Bagley, G., & Prouty, R. (1970) *Pestic. Monit. J.* 4, 141-144.
- (8) Bagley, G., Reichel, W., & Cromartie, E. (1970) *JAOAC* 53, 251-261.
- (9) Biron, F., Walker, A., & Medbery, A. (1970) *Bull. Environ. Contam. Toxicol.* 5, 317-323.
- (10) Hutzinger, O., Jamieson, W., & Zitko, V. (1970) *Nature* 226, 664.
- (11) Stallings, D., & Huckins, J. (1971) *JAOAC* 54, 801-807.
- (12) Sisson, D., & Welf, D. (1971) *J. Chromatogr.* 60, 15-32.
- (13) Tan, A., & de Vos, R. (1971) *Environ. Sci. Technol.* 5, 1210-1218.
- (14) Webb, R., & McCall, A. (1972) *JAOAC* 55, 746-752.
- (15) Willis, D., & Addison, R. (1972) *J. Fish. Res. Board Can.* 29, 592-595.
- (16) Rote, J., & Murphy, P. (1971) *Bull. Environ. Contam. Toxicol.* 6, 377-384.

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= 0.011; ³⁵Cl =

re of chlorine

not be affected
they have weak
a, and can be

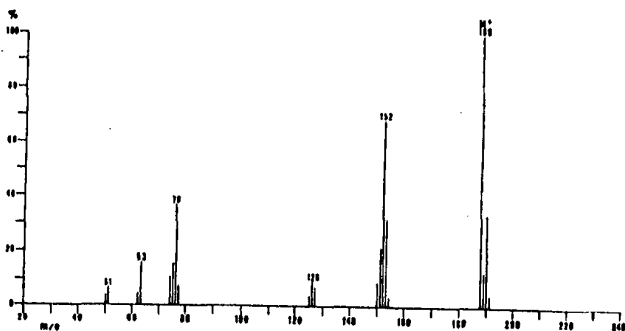


FIG. 1—Mass spectrum of 2-chlorobiphenyl (Aroclor 1232).

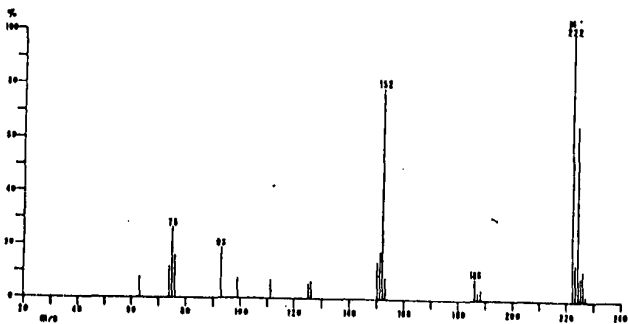


FIG. 2—Mass spectrum of 2,2'-dichlorobiphenyl.

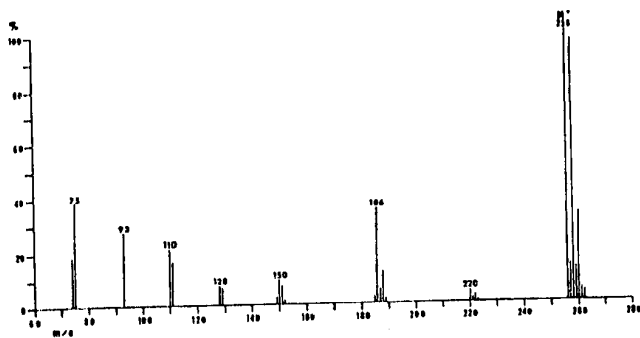
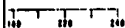


FIG. 3—Mass spectrum of 2,4,6-trichlorobiphenyl (Aroclor 1232).

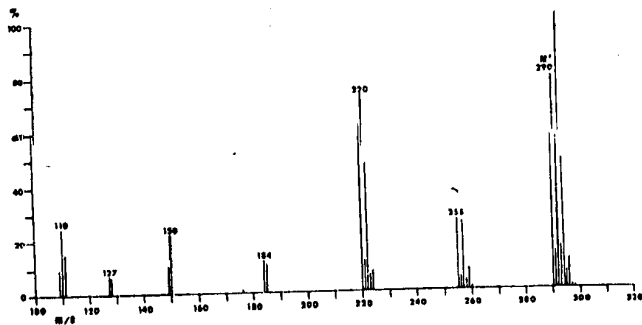
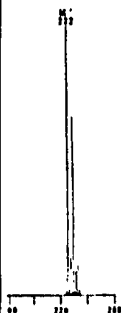


FIG. 4—Mass spectrum of 2,5,2',5'-tetrachlorobiphenyl.

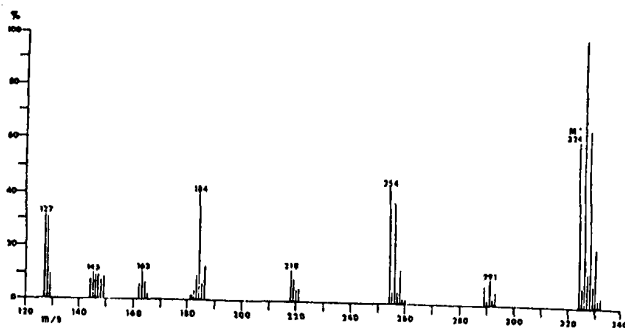


FIG. 5—Mass spectrum of 2,4,5,2',5'-pentachlorobiphenyl (Aroclor 1254).

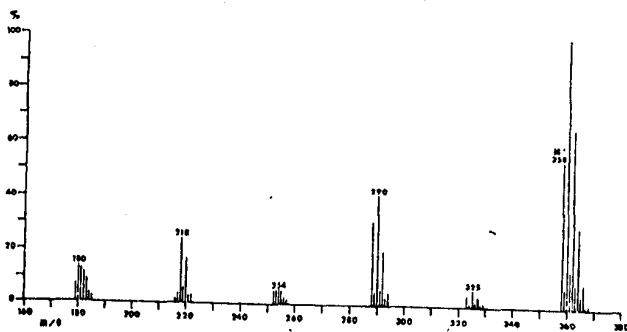


FIG. 6—Mass spectrum of 2,4,5,2',4',5'-hexachlorobiphenyl.

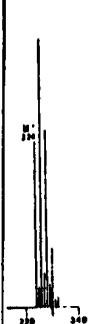


FIG. 7—Mass spectrum of 2,3,4,5,6,2',3',5'-heptachlorobiphenyl (Aroclor 1268).

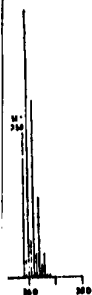


FIG. 8—Mass spectrum of 2,3,4,5,6,2',3',5'-octachlorobiphenyl (Aroclor 1260).

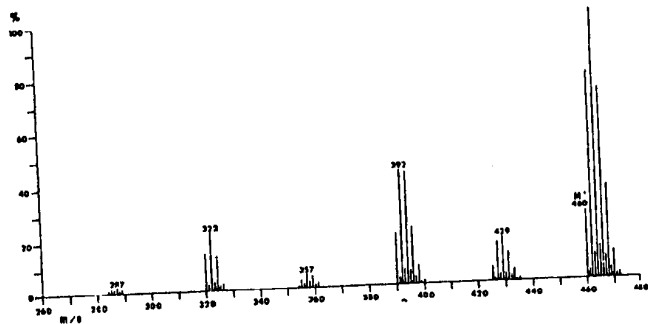


FIG. 9—Mass spectrum of 2,3,4,5,6,2',3',4',5'-nonachlorobiphenyl (Aroclor 1268).

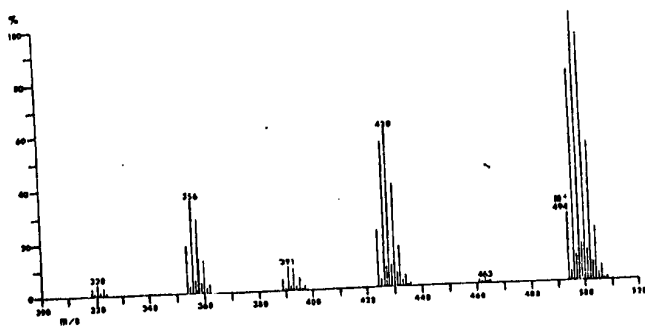


FIG. 10—Mass spectrum of decachlorobiphenyl.

NOTE A

- (17) NaCl
- (18) NaCl
- (19) NaCl
- (20) NaCl
- (21) NaCl
- (22) NaCl
- (23) NaCl
- (24) NaCl
- (25) NaCl

- (17) Safe, S., & Hutzinger, O. (1971) *Nature* 232, 641-642
- (18) Safe, S., & Hutzinger, O. (1971) *Chem. Commun.* 446-448
- (19) Safe, S., & Hutzinger, O. (1972) *J. Chem. Soc. Perkin Trans. I* 5, 986-991
- (20) Hutzinger, O., & Jamieson, W. (1971) *Bull. Environ. Contam. Toxicol.* 5, 587-594
- (21) UK, S., Himel, C., & Dirks, T. (1972) *Bull. Environ. Contam. Toxicol.* 7, 207-215
- (22) Sphon, J., & Damico, J. (1970) *Org. Mass Spectrom.* 3, 51-62
- (23) Damico, J., Barron, R., & Sphon, J. (1968) *J. Mass Spectrom. Ion Phys.* 2, 101-182
- (24) Hutzinger, O., Jamieson, W., & Safe, S. (1971) *JAOAC* 54, 178-180
- (25) Firsirotu, D., Ross, J., Brown, N., Barron, R., & Damico, J. (1972) *JAOAC* 55, 85-92
- (26) Goelitz, D., & Law, L. (1972) *Bull. Environ. Contam. Toxicol.* 7, 243-251
- (27) Tan, J. (1970) *Anal. Chem.* 42, 1381-1386
- (28) Aronow, J., & Burke, J. (1971) *JAOAC* 54, 175-177
- (29) Zitko, V., Hutzinger, O., Jamieson, W., & Choi, P. (1972) *Bull. Environ. Contam. Toxicol.* 7, 200-201
- (30) Stalling, D., Tindle, R., & Johanson, J. (1972) *JAOAC* 55, 32-38
- (31) Beynon, J. (1960) *Mass Spectrometry and Its Application to Organic Chemistry*, Elsevier, Amsterdam, p. 208
- (32) Bonelli, E. (Feb. 1971) *Amer. Lab.* 27-37
- (33) Hutzinger, O., Safe, S., & Zitko, V. (1971) *Bull. Environ. Contam. Toxicol.* 6, 209-219
- (34) McLafferty, F. (1967) *Interpretation of Mass Spectra*, Benjamin, New York, p. 210



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