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POLYCHLORINATED BIPHENYLS

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Polychlorinated Biphenyls

Introduction

Within the last few years, a sudden interest developed in a hitherto relatively unknown class of industrial organochlorine compounds: the polychlorinated biphenyls (PCB).

Traces of these compounds were reported to be present in environmental samples by Jensen in 1966 (1) and in the following years PCB contamination was found to be almost universal (2-7) including human milk (8), human adipose tissue (8,9) and brain and liver of small children (10). Little is known about the toxic effects of PCB in humans, but an endemic poisoning ("Yusho") by rice oil contaminated with PCB was reported (11).

Information on PCB can be found in the reviews (2-7) mentioned above, in two technical reports (12,13), in a trade publication (14), an encyclopedia article (15) and in collected papers from a recent symposium (16). A comprehensive review of the chemistry of chlorobiphenyl, is in preparation (17).

Commercially, PCB are prepared by chlorination of biphenyl (15) and this procedure results in complex mixtures containing isomers of chlorobiphenyls with different chlorine content. The trade names of some of the products (country in which sold) are: Aroclor (U.S., U.K., Canada); Kanechlor (Japan); Phenoclor (France); and Clophen (Germany). PCB with different chlorine content are available; information for one line of products (Aroclor, ref. 14) is given in Table 1.

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Some of the major uses of PCB are shown in Table 2. Annual production of PCB in the U.S.A. increased from 19,000 metric tons in 1960 to 43,000 metric tons in 1970 (18). The amount of PCB produced in Japan in 1970 was about 12,000 metric tons (19). Data on the production of PCB in other countries are not available. An estimated annual consumption of PCB in Finland is about 250 metric tons (20).

The characteristic properties of PCB which make them desirable for industrial use are the high dielectric constant and their thermal and chemical stabilities. The stability of these compounds, on the other hand, combined with the fact that PCB are readily taken up by living organisms and accumulated at higher levels of the food web, causes problems in the environment.

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TABLE I

Information on Aroclor preparations

Aroclor	%Cl	Average number of Cl per molecule	[illegible]
Aroclor 1221	21	1.15	[illegible]
Aroclor 1232	[illegible]	[illegible]	[illegible]
Aroclor 1242	[illegible]	[illegible]	[illegible]
Aroclor 1248	[illegible]	[illegible]	[illegible]
Aroclor 1254	[illegible]	[illegible]	[illegible]
Aroclor 1260	[illegible]	[illegible]	[illegible]
Aroclor 1262	[illegible]	[illegible]	[illegible]
Aroclor 1268	[illegible]	[illegible]	[illegible]

Since PCB are complex mixtures of chlorinated compounds they give a large number of peaks when analyzed by gas chromatography (Fig. 1 and 2) using the electron capture detector. From this it is evident that another problem is caused by PCB in environmental samples: interference in analyses for pesticide residues (21).

TABLE II

Major uses of PCB

Dielectric fluids	capacitors, transformers
Industrial fluids	hydraulic systems, gas turbines, vacuum [illegible]
Plasticizer	adhesives, textiles, surface [illegible]
Heat transfer fluid	[illegible]
Fire retardant	[illegible]

Individual PCB Isomers

It was soon recognized that the complexity of industrial PCB preparations placed a severe limitation on the development of analytical methodology and on the study of metabolism and biological properties. Quantitative analysis, for example, cannot be based on absolute standards, different toxicities of different isomers cannot be evaluated and metabolites cannot be recognized and identified easily.

Several research groups have attempted to characterize individual compounds in commercial PCB mixtures and by now the structures of most components of the Aroclor series, as well as of some major constituents of Kanechlor-400, and Phenoclor DP-6 are known. Separation by capillary GLC, retention indices, comparison with authentic standards, mass spectrometry and 220 MHz NMR have been used in the structure elucidation of Aroclor components (22; see Fig. 2 and 4). GLC, synthesis and infrared spectroscopy was used for the identification of Aroclor components by another group (23; Fig. 4) and components of Aroclor 1221 have been identified by compansion with synthetic samples (24). 5 Major components of Kanechlor-400 were identified by comparison with synthetic standards after information on the products was obtained by UV and GLC (25; Fig. 4). Seven major constituents of Phenochlor DP-6 were identified by comparison with synthesized material (26,27; Fig. 4).

Evidence was presented recently that some of the toxic effects of PCB preparations may be due to chlorodibenzofuran (Fig. 5) impurities (28,29).

Metabolism and Photochemical Breakdown

"Disappearance" of certain peaks in PCB extract from rats (30) and birds (31) and the absence of certain peaks at higher trophic levels (Fig. 6) would indicate metabolic breakdown. The only published study on the identification of chlorobiphenyl metabolites reported, however, is the formation of 4-chloro-4'-hydroxybiphenyl and some of its conjugation products from 4-chlorobiphenyl in rabbits (32). Very recently, however, it has been shown that hydroxylation of the lower chlorine homologues seems to be a general metabolic pathway in the rat and pigeon (e.g. Fig. 7) Hydroxylated products, however, could not be isolated from fish treated with chlorobiphenyls (33).

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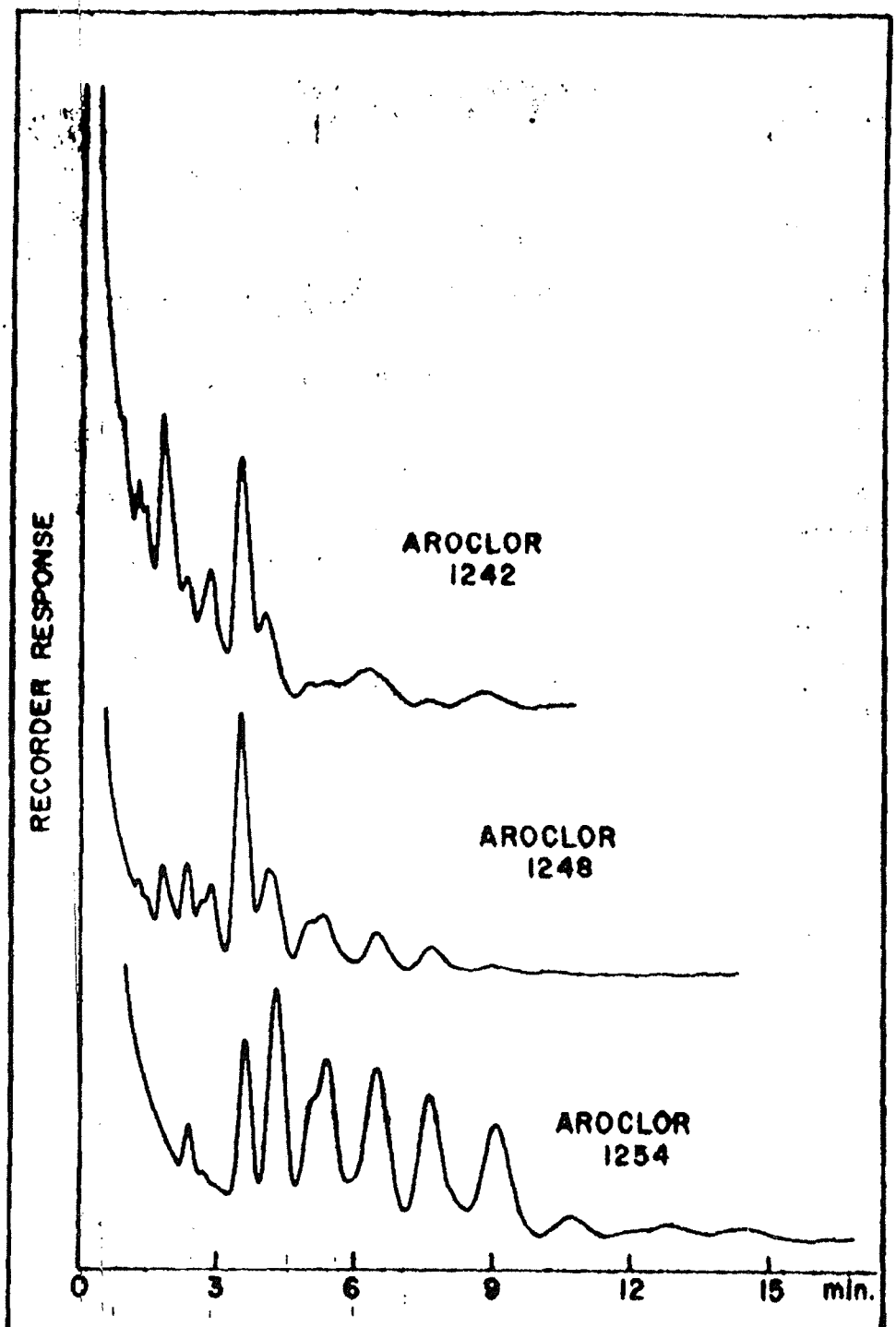

Fig. 1 – GLC tracings of three Aroclor preparations.
Conditions: glass column (6' x 4 mm), filled with 4% SE-30 on acid washed Chromosorb W and operated at 200°. Carrier gas nitrogen at 60 ml/min. Electron capture detector.

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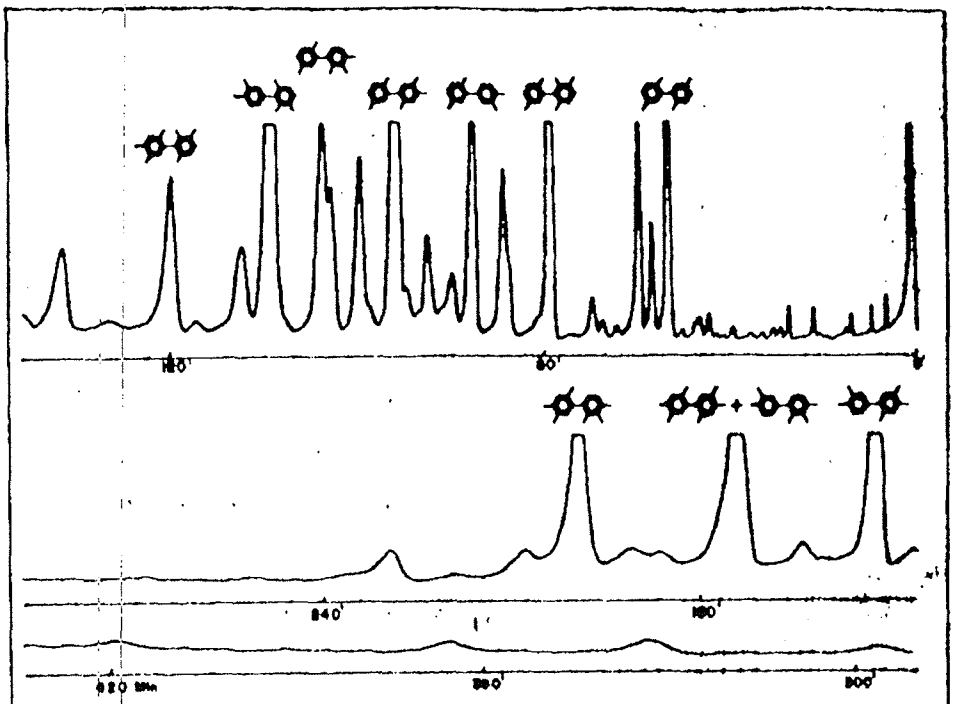
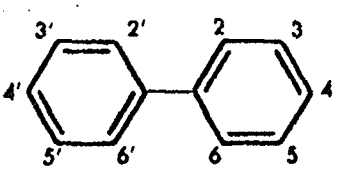


Fig. 2 — Separation of Aroclor 1254 by capillary column-gas chromatography. Conditions: 50 ft. x 0.02 in. SCOT Apiezon-L column; 205°; He flow 2.5 ml/min; flame-ionization detector. Structures of isomers responsible for major peaks are indicated.



Numbering system used for naming PCB's.

Under laboratory conditions, at an irradiation wavelength of 310 nm chlorobiphenyls decompose relatively readily to give products which are formed by reductive dechlorination, isomerization, and polymerization (34, 35). In the presence of air and water, polar products were observed. 2,2'5,5'-Tetrachlorobiphenyl gave products, on exposure to sunlight, which were chromatographically similar to those obtained under laboratory conditions.

Chromatography and Analysis
Gas Chromatography

The electron-capture detector response to chlorobiphenyls generally increases strongly from the mono- to trichlorobiphenyls whereas only a small increase is observed between tetra- and decachlorobiphenyl. It has also been reported (36) that in some cases the detector response differences between some isomers can be larger than the differences between chlorobiphenyls containing differing numbers of chlorine atoms. On a weight basis the detector response to decachlorobiphenyl is 500 times stronger than its response to 4-chlorobiphenyl but only 2.1 times stronger than the

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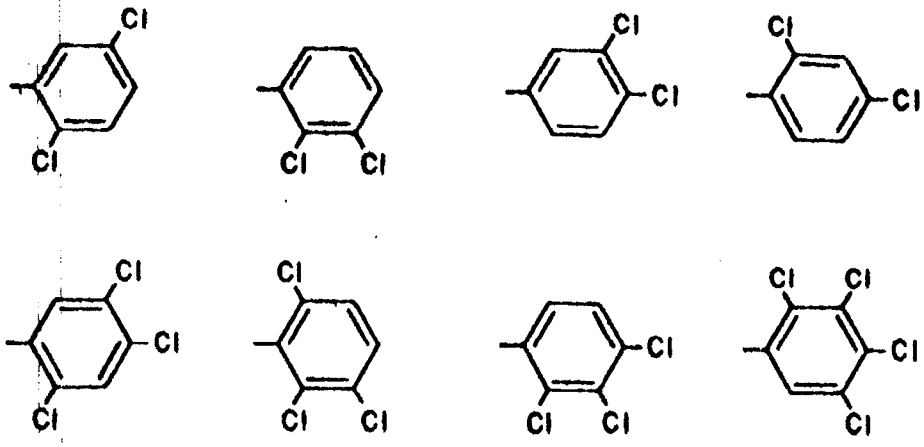

Fig. 4 – Prevalent substitution pattern of compounds found in commercial PCB preparation. Only one phenyl-ring is shown. The most abundant tetrachlorobiphenyls, for example, are those from the dichlorophenyl-moieties shown. One di- and one trichlorophenyl- would give the most abundant penta-chlorobiphenyls etc.

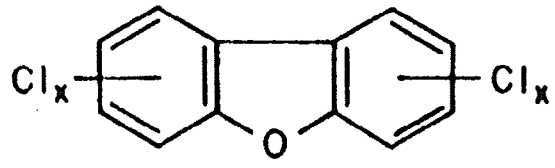

Fig. 5 – Formula for polychlorodibenzofuran.

response to 3,4,3',4'-tetrachlorobiphenyl (36). This is important for the quantitation of PCB in environmental samples. Most of these samples contain tetra-, penta-, hexa-, and heptachlorobiphenyls. The detector response to these compounds is not likely to vary more than by a factor of two, which is a reasonably good basis for quantification of such a complex mixture of compounds. Since appropriate standard chlorobiphenyls were not available at that time the quantification had to be based on commercial PCB preparations. Some authors use total peak area (37) or area of one or more peaks (38-41), others use peak heights (30,42,43). A method based on the average electron-capture detector response to chlorobiphenyls, containing from one to seven chlorines, has been described (44). Aroclor 1254 was quantified by perchlorination with antimony pentachloride and the resulting decachlorobiphenyl was determined by gas chromatography (45).

Without standardized quantification techniques, an international exchange-sample program indicates that the results agree within ± 20% (46). This is a reasonable precision in terms of residue analysis. It has to be emphasized, however, that the results are not accurate, since the analyses are not based on standards of individual chlorobiphenyls.

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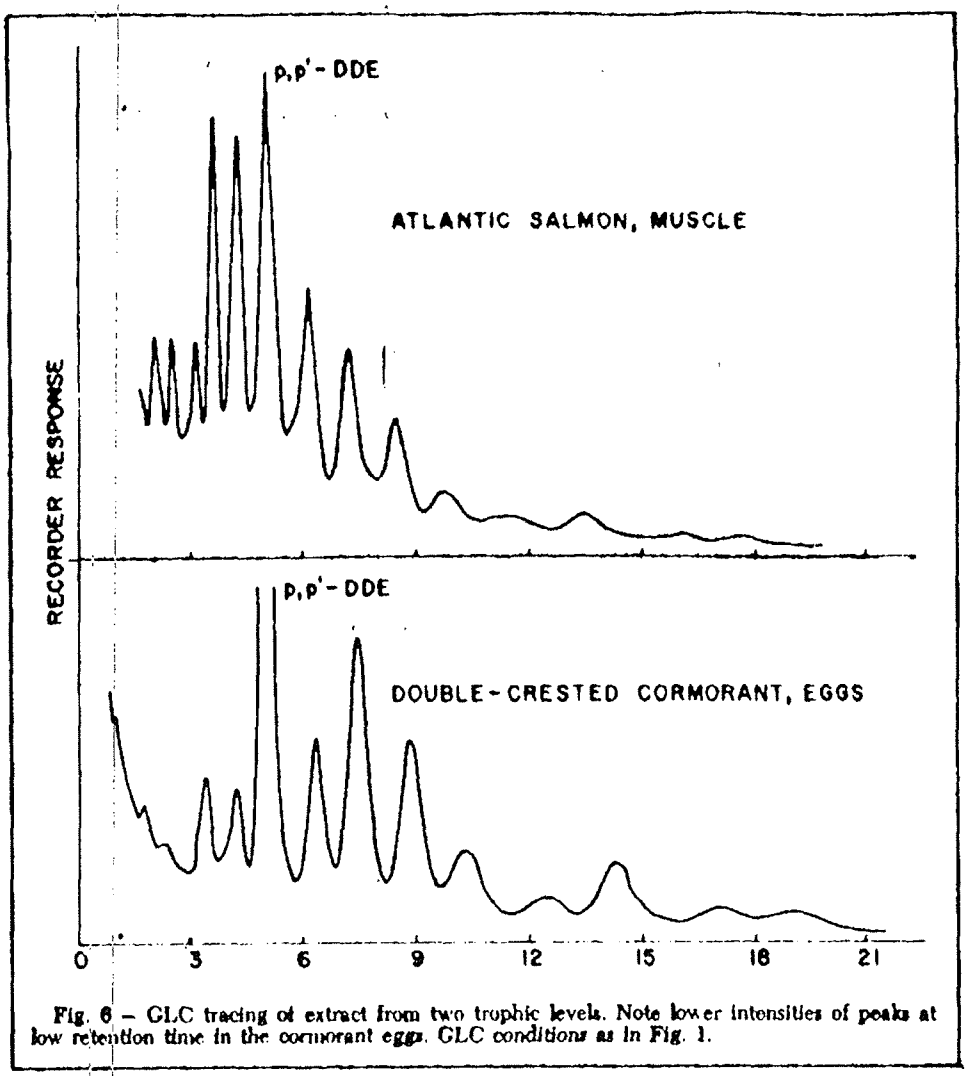

Fig. 6 – GLC tracing of extract from two trophic levels. Note lower intensities of peaks at low retention time in the cormorant eggs. GLC conditions as in Fig. 1.

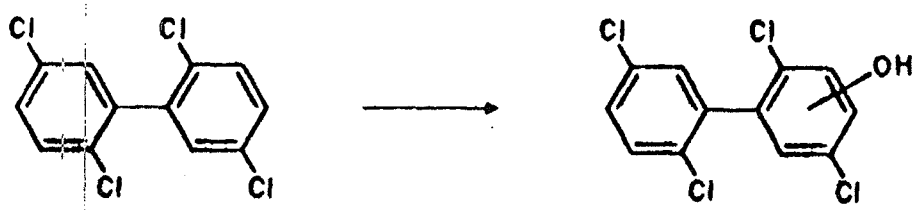

Fig. 7 – Metabolic conversion of 2,2',5,5' -tetrachlorobiphenyl by rat and pigeon.

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Column Chromatography, Thin Layer Chromatography, Clean-up

In the analysis of environmental samples, PCB must be separated from chlorinated hydrocarbon pesticides since peaks of both groups of compounds have similar retention times on gas chromatography. PCB can be separated quite easily from most common chlorinated hydrocarbon pesticides, such as p,p'-DDT, p,p'-DDD, dieldrin, and endrin by column chromatography. Thus PCB are eluted from Florisil columns with hexane, DDT and other pesticides with a mixture of ether and hexane (47). Elution patterns on silicic acid are similar (48,49). PCB were also separated from chlorinated hydrocarbon pesticides by column chromatography on charcoal. The latter are eluted with 25% acetone in ethyl ether and PCB are subsequently recovered by elution with benzene (45). Column chromatography on alumina is used to separate PCB from chlorinated dibenzodioxins (50). The separation of PCB from p,p'-DDE is, however, difficult, it may be carried out on silicic acid under very carefully controlled conditions (36), or on silica gel activated at 200° for 8 hours (51). The purity of nonpolar solvents, used in these separations is very important. Small amounts of benzene, present in some pesticide-grade hexanes significantly change the elution patterns (52). Some contamination of the pesticide fraction with PCB in the procedure of Armour and Burke (37) was detected by a combined GLC-MS procedure (53). GLC-MS techniques do not require as thorough a cleanup as conventional GLC methods (54) and the separation of PCB from pesticides is not always necessary with this approach. Less than 10 ng of p,p'-DDE can be identified and measured in the presence of PCB (Aroclor 1254) by mass spectrometric monitoring of the m/e = 246 peak in the GLC effluent (55).

Thin-layer chromatography on alumina with n-heptane (56) and on silver nitrate-coated alumina with 5% benzene in hexane (57) was used to separate PCB from chlorinated hydrocarbon pesticides. A two-dimensional technique was also described (58). Reversed phase TLC is very effective for the separation of PCB components (59,60).

A method based on reversed liquid-liquid partition between water and Chromosorb W, coated with n-undecane and Carbowax 4000 was used to recover PCB in the part per trillion range from water (61). Porous polyurethane foam adsorbs PCB from water (62). PCB can be then recovered by extraction with acetone and hexane.

During the analysis for PCB, great care must be taken to avoid contamination of samples because of the presence of PCB in a variety of materials such as paper, plastic, paint, etc. Dust in a laboratory contained PCB (Aroclor 1254) at a level of 45 μ g/g. The source of this contamination was the floor wax (63).

Recently, a clean-up procedure based on gel permeation chromatography was described (64).

Entry, Accumulation and Levels in the Environment

As the above mentioned sources of PCB indicate, PCB enter the environment via waste-disposal systems. In addition, industry may contribute by spills or leakages of PCB from heat-exchange and hydraulic systems. This type of PCB discharge occurred in Escambia Bay, Florida (65). The concentration of PCB in raw sewage from the Glasgow, Scotland area ranged from 0.1 to 14 μ g/g as Aroclor 1254 on wet weight basis with an estimated annual PCB discharge of 1 ton (66). In California, sewage outfalls discharge per year from 0.06 to 7 tons of PCB in water and from 0.6 to 36 tons in sludge (67). Aroclor 1260 and Aroclor 1242 in concentrations of 0.02-0.26 μ g/l and 0.08-2.07 μ g/l, respectively, were found in the Milwaukee River in August, 1969. Their origin was traced to both industrial and municipal wastes (68). Water of Lake Biwa, Japan, contains 0.1-1.0 μ g/l of PCB (19).

Once in the environment, PCB accumulate in the biomass. The distribution of PCB in fish from the U.S.A., Sweden, and Canada is presented in Table 3.

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TABLE III

Distribution of PCB in fish

PCB range µg/g Net weight	Number of fish U. S. A. (60) Fresh water	Sweden (73) Marine	Sweden (73) Fresh water	Canada (13) Marine
0 - 0.1		223	115	11
0.2 - 0.5	2	538	21	51
0.6 - 1.0		80	5	6
1.1 - 2.0		13	4	1
2.1 - 3.0	11	8		
3.1 - 5.0				
5.1 - 10.0	11			
10.1 - 25.0	5			
25.1 - 100	5			
101 - 250	6			

The levels of PCB in aquatic animals in Japan are comparable to those found in the U.S.A. Fish from Tokyo Bay contain 0.1-3.4 mg of PCB in meat. Gulls from Osaka Bay (*Larus ridibundus* and *L. crassirostris*) contain respectively 5-36, and 13-27 mg/g wet weight of PCB in meat (19). PCB levels in livers of birds from Denmark ranged from 1.3 ppm in buzzard (*Buteo buteo*) to 272 in fawning owl (*Strix aluco*) (69). A comprehensive study of PCB levels in birds in Great Britain has also been published (41). The following is an example of a somewhat different route of environmental contamination with PCB. PCB (Aroclor 1254) used in silo sealants at a level of 2,000-10,000 ppm) contaminated the silage and resulted in significant PCB levels in milk (70). Corn silage extract had a remarkable ability to solubilize PCB. Contamination of two steers and a milking cow with Aroclor 1254 was reported in Canada. The steers contained 1.37 and 0.132 ppm PCB in the muscle, 4.96 and 1.38 ppm in perimuscular fat. The source of PCB was probably water from a contaminated stream (71). Milk of two Jersey cows, given orally 10 and 100 mg of Aroclor 1254 per kg of body weight, respectively, pooled for 4 days, contained 3.9 and 36 mg/kg of PCB, respectively. This corresponds to an excretion of 6.2 and 3.8% of the administered dose, respectively (72).

Last but not least, the contamination of food products such as cereals, biscuits, and rice by PCB, migrating from the paper-board packages, should be mentioned (63,73,74).

Mass Spectrometry

Mass spectrometric analysis was used in the early work to confirm the presence of PCB suggested by GLC data (75,76) and GLC-mass spectrometry has since been used to identify PCB in samples such as human adipose tissue (9) and bald eagles (77). GLC-mass spectrometry was also used to characterize the molecular composition (number of chlorine atoms) of components of PCB preparations (9,78,79).

A method for the identification of organochlorine compounds, including PCB, in mixtures and crude samples, by high resolution mass spectrometry has recently been described (80,81). Some other applications of mass spectrometry to analysis are discussed in the section on gas chromatography.

Although commercial PCB preparations (e.g. Aroclor 1254) are complex mixtures the mass spectra are relatively simple (e.g. Fig. 8) since all isomers of a particular molecular composition give identical molecular ions. The mass spectrum also reveals the typical isotope distribution pattern which is highly characteristic of the number of chlorine atoms present in the PCB isomers. The presence of these patterns readily facilitates identification of small quantities of PCB which may be present in a complex mixture.

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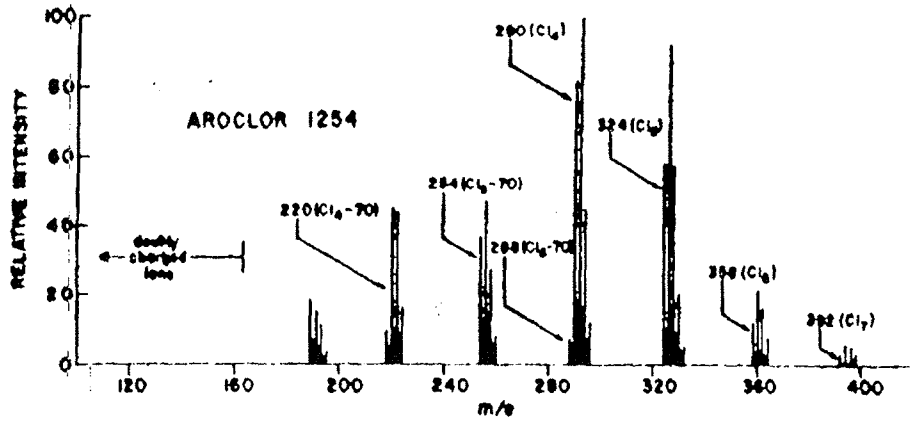
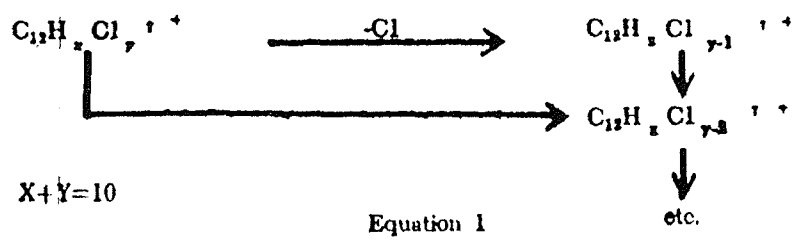


The synthesis of individual PCB isomers also permitted a detailed investigation of their primary ion mass spectra and fragmentation patterns (82,83). The mass spectra of each set of isomeric compounds are similar and the fragmentation pattern of all the compounds from the dichlorobiphenyl isomers to decachlorobiphenyl feature successive expulsion of chlorine atoms and molecules (Equation 1 and Fig. 9), with elimination of HCl from the less chlorinated homologues also evident. The explusion of both chlorine atoms and molecules are in many cases accompanied by metastable ions whose relative intensity varies according to the degree of chlorine substitution at the 2 and 6 positions. The [metastable ion] / [daughter ion] ratio for the reaction (a) – (c) for the di- and tetrachlorobiphenyls is much less for those isomers with 2 or more chlorine groups at the 2 and 6 positions.



Equation 1

Other Techniques

Ultraviolet, infrared-, nuclear magnetic resonance spectroscopy has greatly helped in the identification of isomers present in the complex PCB mixtures.

Plasma chromatograms of a series of mono to decachlorbiphenyls have been recorded (84). The method is extremely sensitive and may find useful analytical applications.

Neutron activation has been used to determine chlorobiphenyls in rat adipose tissue (85).

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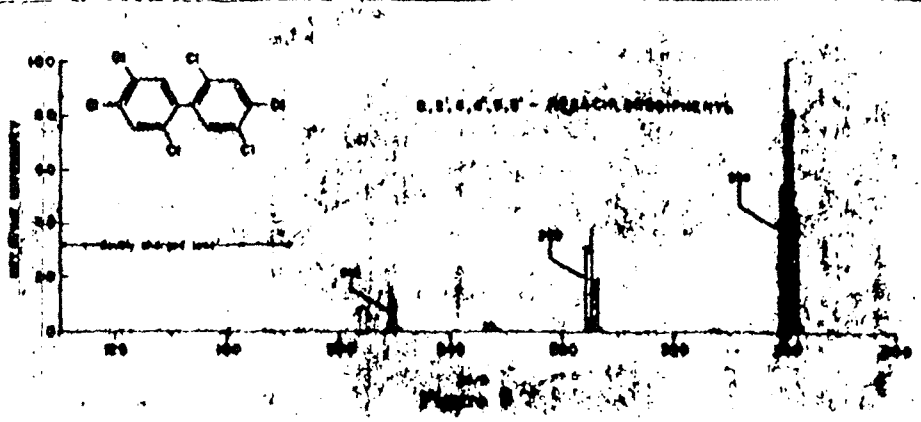

Outlook

Environmental levels of PCB indicate only that the environment is contaminated. Monitoring of PCB over a period of time is required to determine the environmental dynamics. As illustrated in Fig. 6, for example which presents PCB patterns at two trophic levels (fish, cormorant), there is evidence that at least some less chlorinated biphenyls are metabolized. Very little is known about the efficiency of uptake and excretion of PCB and it is possible that a steady-state situation exists in some areas.

More research is required to answer these questions. In the meantime, PCB discharge should be limited as much as possible. A hurried introduction of PCB substitutes with an unknown environmental impact, such as chlorinated paraffins, may however, create additional problems.

References

1. Anonymous, *New Scientist* 32, 612 (1966).
2. R. W. Risebrough, P. Rieche, D. B. Peakall, S. G. Herman and M. N. Kirven, *Nature* 220, 1098 (1968).
3. C. G. Gustafson, *Environ. Sci. Technol.* 4, 814 (1970).
4. D. B. Peakall and J. L. Lincer, *BioScience* 20, 958 (1970)
5. C. D. Veith and G. F. Lee, *Water Research* 4, 265 (1970).
6. A. Richardson, J. Robinson, A. N. Crabtree and M. K. Baldwin, *Pesticide Monitoring Journal* 4, 169 (1971).
7. R. Edwards, *Chem. Ind.* 1340 (1971).
8. L. Acker and E. Schulte, *Naturwissenschaften* 57, 497 (1970).
9. F. J. Biros, A. C. Walker and A. Medbery, *Bull. Environ. Contam. Toxicol.* 5, 317 (1970).
10. J. F. S. Crocker, O. Hutzinger, W. D. Jamieson, S. Safe and V. Zitko, in preparation.
11. M. Kuratsune, T. Yoshimura, J. Matsuzaka and A. Yamaguchi, *H.S.M.H.A. Health Reports* 86, 1083 (1971) and *Environmental Health Perspectives* 1, in press.
12. V. Zitko, Fisheries Research Board of Canada, Manuscript Report Series No. 1083 (1970).
13. 13. V. Zitko and P. M. K. Choi, Fisheries Research Board of Canada, Technical Report No. 272 (1971).
14. Anonymous, Aroclor Plastizisers, Monsanto Technical Bulletin O/PL-306A.
15. H. L. Hubbard, Kirk-Othmer Encyclopedia of Chemical Technology, 2nd Edition, 5, 289 (1964).
16. Environmental Health perspectives, Vol. 1, No. 1 (1972), in press.
17. O. Hutzinger, S. Safe and V. Zitko, submitted to Toxicol. Environ. Chem. Rev.
18. Anonymous, *Chem. Eng. News*, Dec. 6, p. 15 (1971).
19. N. Isono, *Critical News* No. 1 (1971).
20. M. Helminen, PCB Conference, National Swedish Environmental Protection Board, Stockholm, Sept. 29 (1970).
21. L. M. Reynolds, *Residue Revs.* 34, 27 (1971).
22. D. Sissons and D. Welti, *J. Chromatogr.* 60, 15 (1971) and personal communication.
23. R. G. Webb and A. C. McCall, paper presented at the 162nd National Meeting of the American Chemical Society, Washington, September, 1971, and personal communication.
24. D. E. Willis and R. F. Addison J. Fish. Res. Board Canada, in press.
25. S. Salki, A. Tsutsui, K. Oguri, H. Yoshimura and M. Hamana, *Fukuoka Igaken Zasshi* 62, 21 (1971); *Chem. Abstr.* 74, 146294 (1971).
26. A. C. Tas and R. H. deVos, *Environ. Sci. Technol.* 5, 1216 (1971).
27. A. C. Tas and R. J. C. Kleinpool, personal communication.
28. T. G. Vos, T. H. Koeman, H. L. van der Maas, M. C. Ten Noever de Brauw and R. H. De Vos, *Fd. Cosmet. Toxicol.* 8, 625 (1970).
29. T. G. Vos, *Environmental Health Perspectives* 1, in press.

DSW 029377

STLCOPCB4013339

30. D. L. Grant, W. E. J. Phillips and D. C. Villeneuve, *Bull. Environ. Contam. Toxicol.* 6, 102 (1971).
31. S. Bailey and P. T. Bunyan, *Nature* 236, 34 (1972).
32. W. D. Block and B. H. Cornish, *J. Biol. Chem.* 234, 3301 (1959).
33. O. Hutzinger, D. N. Nash, S. Safe, A. S. W. DeFreitus, R. J. Norstrom, D. J. Wildish and V. Zitko, in preparation.
34. S. Safe and O. Hutzinger, *Nature*. 232, 641 (1971).
35. O. Hutzinger, S. Safe and V. Zitko, *Environmental Health Perspectives* 1, in press.
36. V. Zitko, O. Hutzinger and S. Safe, *Bull. Environ. Contam.* Toxicol. 6, 160 (1971).
37. J. A. Armour and J. A. Burke, *J. Assoc. Offic. Anal. Chem.* 53, 761 (1970).
38. J. H. Koeman, M. C. Ten Noever De Brauw and R. H. De Vos, *Nature* 221, 1126 (1969).
39. K. Vermeer and L. M. Reynolds, *Canadian Field-Naturalist* 84, 117 (1970).
40. J. E. Neil, L. E. Priester and S. H. Sandifer, *Bull. Environ. Contam. Toxicol.* 6, 156 (1971).
41. I. Prett, D. J. Jefferies and N. W. Moore, *Environmental Pollution* 1, 3 (1970).
42. D. J. Hansen, P. R. Parrish, J. I. Lowe, A. J. Wilson, Jr., and P. D. Wilson, *Bull. Environ. Contam. Toxicol.* 6, 113 (1971).
43. R. Risebrough, Chlorinated hydrocarbons in material ecosystems, Chemical Fallout 5, (1969), Charles C. Thomas, publisher, Springfield, Ill.
44. J. W. Rote and P. C. Murphy, *Bull. Environ. Contam. Toxicol.* 6, 377 (1971).
45. O. W. Berg, P. L. Diosady and G. A. V. Rees, *Bull. Environ. Contam. Toxicol.* in press.
46. A. V. Holden, personal communication.
47. L. B. Reynolds, *Bull. Environ. Contam. Toxicol.* 4, 128 (1969).
48. A. V. Holden and K. Marsden, *J. Chromatog.* 44, 481 (1969).
49. V. Leoni, *J. Chromatogr.* 62, 63 (1971).
50. M. L. Porter and J. A. Burke, J. Ass. Offic. Anal. Chem. 54, 1426 (1971).
51. D. Snyder and R. Reinert, *Bull. Environ. Contam. Toxicol.* 6, 385 (1971).
52. V. Zitko, *J. Chromatog.* 59, 444 (1971).
53. S. W. Bellman and T. L. Barry, *J. Ass. Offic. Anal. Chem.* 54, 499 (1971).
54. E. J. Bonelli, Computer-controlled GC/MS for the analysis of polychlorinated biphenyls. American Laboratory, February (1971).
55. E. J. Bonelli, *Anal. Chem.* 44, 603 (1972).
56. G. Westoo and K. Noren, *Acta Chem. Scand.* 24, 1639 (1970).
57. B. M. Mulhern, E. Cromartie, W. L. Reichel and A. A. Belisle, *J. Ass. Offic. Anal. Chem.* 54, 548 (1971).
58. N. V. Fehringer and J. E. Westfall, *J. Chromatogr.* 57, 397 (1971).
59. R. H. De Vos and E. W. Peet, *Bull. Environ. Contam. Toxicol.* 6, 184 (1971).
60. D. L. Stalling, Presentation at the Meeting on Analysis of Residues of PCB's (working group on pesticides monitoring panel), Sept. 17, 1971, and personal communication.
61. B. Ahling and S. Jensen, *Anal. Chem.* 42, 1483 (1970).
62. H. D. Gesser, A. Chow, F. C. Davis, J. F. Uthe and J. Reinke, *Anal. Lett.* 4, 883 (1971).
63. V. Zitko, unpublished experiments.
64. D. L. Stalling, R. C. Tindle and J. L. Johnson, *J. Assoc. Offic. Anal. Chem.* 55, 32 (1972).
65. T. W. Duke, J. I. Lowe and A. J. Wilson, *Bull. Environ. Contam. Toxicol.* 5, 171 (1970).
66. A. V. Holden, *Nature* 228, 1220 (1970).
67. T. T. Schmidt, R. W. Risebrough, and F. Gress, *Bull. Environ. Contam. Toxicol.* 6, 235 (1971).
68. G. D. Veith and G. F. Lee, *Water Res.* 5, 1107 (1971).
69. O. Karlag, I. Kraul and Sv. Dalgaard-Mikkelsen, *Acta Vet. Scand.* 12, 310 (1971).
70. R. F. Skrentny, R. W. Hemken, and H. W. Dorough, *Bull. Environ. Contam. Toxicol.* 6, 409 (1971).
71. N. S. Platonow, P. W. Saschenbrecker and H. S. Funnel, *Can. Vet. Journ.* 12, 115 (1971).
72. N. S. Platonow, H. S. Funnell, D. H. Bullock, D. R. Arnott, P. W. Saschenbrecker and D. G. Grieve, *J. Dairy Sci.* 54, 1305 (1971).
73. G. Westoo, K. Noren, and M. Andersson, *Var Foeda* 341, (1971).
74. A. C. Kobbey, Environmental Health Perspectives, (in press).
75. G. Widmark, *J. Assoc. Offic. Anal. Chem.* 50, 1069 (1967).
76. T. H. Koeman, M. C. Ten Noever de Brauw and R. H. De Vos, *Nature* 221, 1126 (1969).
77. C. E. Bagley, W. L. Reichel and E. Cromartie, *J. Assoc. Offic. Anal. Chem.* 53, 251 (1970).
78. D. L. Stalling and T. N. Huckins, *J. Assoc. Offic. Anal. Chem.* 54, 801 (1971).
79. T. Murata, S. Takahashi and R. *Tachikawa, Shimadzu Review* 28, 29 (1971).
80. O. Hutzinger, W. D. Jamieson, and V. Zitko, *Nature* 226, 664 (1970).
81. O. Hutzinger and W. D. Jamieson, Pesticide Chemistry Proc. 2nd International IUPAC Congress, (A. Talim, Ed.) Vol. IV, 7 (1971).
82. S. Safe and O. Hutzinger, *Chem. Comm.* 446 (1971).
83. S. Safe and O. Hutzinger, *J. Chem. Soc. Perkin Trans* 1, 686, (1972).
84. F. W. Karasek, *Anal. Chem.* 43, 1982 (1971).
85. T. Mamri, Y. Shinbori S. Murakawa and Y. Mochizuki, *Radioisotopes* 20, 371 (1971).

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Fig. 2 is redrawn from: D. Sissons, and D. Welti, "Structural identification of polychlorinated biphenyls in commercial mixtures by GLC. NMR and mass spectrometry, with the permission of authors and publisher.

DSW 029378

STLCOPCB4013340

99% Pure PCB Isomers

For clarity we have kept the position numbering separate for each ring. For Chemical Abstracts nomenclature transpose numbers listing lowest number first.

PC1100	Biphenyl	7.00/500 mg	
PC1102	2-Chlorobiphenyl	7.00/100 mg	
PC1103	3-Chlorobiphenyl	7.00/100 mg	
PC1101	4-Chlorobiphenyl	7.00/100 mg	
PC2113	2, 2'-Dichlorobiphenyl	18.00/100 mg	New
PC2116	3, 3'-Dichlorobiphenyl	18.00/100 mg	
PC2111	4, 4'-Dichlorobiphenyl	18.00/100 mg	
PC2212	2, 4'-Dichlorobiphenyl	32.00/50 mg	New
PC2204	2, 3-Dichlorobiphenyl	14.00/100 mg	
PC2201	2, 4-Dichlorobiphenyl	14.00/100 mg	
PC2205	2, 5-Dichlorobiphenyl	14.00/100 mg	
PC2202	2, 6-Dichlorobiphenyl	14.00/100 mg	
PC2203	3, 4-Dichlorobiphenyl	14.00/100 mg	
PC2206	3, 5-Dichlorobiphenyl	14.00/100 mg	
PC3211A	2, 4, 4'-Trichlorobiphenyl	32.00/20 mg	New
PC3211C	2, 5, 2'-Trichlorobiphenyl	28.00/50 mg	New
PC3211I	2, 5, 3'-Trichlorobiphenyl	28.00/50 mg	New
PC3215	2, 5, 4'-Trichlorobiphenyl	28.00/50 mg	New
PC3219	3, 4, 2'-Trichlorobiphenyl	28.00/100 mg	
PC3303	2, 3, 4-Trichlorobiphenyl	26.00/50 mg	New
PC3304	2, 3, 6-Trichlorobiphenyl	36.00/50 mg	New
PC3302	2, 4, 5-Trichlorobiphenyl	22.00/100 mg	
PC3301	2, 4, 6-Trichlorobiphenyl	26.00/100 mg	
PC4221H	2, 3, 2', 3'-Tetrachlorobiphenyl	28.00/100 mg	
PC4221A	2, 4, 2', 4'-Tetrachlorobiphenyl	28.00/100 mg	
PC4221K	2, 5, 2', 5'-Tetrachlorobiphenyl	28.00/100 mg	
PC4227	2, 6, 2', 6'-Tetrachlorobiphenyl	28.00/100 mg	New
PC4221D	3, 4, 3', 4'-Tetrachlorobiphenyl	28.00/100 mg	
PC4221I	2, 3, 2', 5'-Tetrachlorobiphenyl	35.00/50 mg	
PC4225	2, 4, 2', 5'-Tetrachlorobiphenyl	22.00/20 mg	New
PC4221F	2, 5, 3', 4'-Tetrachlorobiphenyl	35.00/50 mg	
PC4402	2, 3, 4, 5,-Tetrachlorobiphenyl	20.00/50 mg	New
PC4403	2, 3, 5, 6-Tetrachlorobiphenyl	20.00/50 mg	
PC5321R	2, 3, 4, 2', 5'-Pentachlorobiphenyl	25.00/20 mg	
PC5321T	2, 3, 6, 2', 5'-Pentachlorobiphenyl	22.00/10 mg	New
PC5321L	2, 4, 5, 2', 3'-Pentachlorobiphenyl	22.00/10 mg	New
PC5322	2, 4, 5, 2', 4'-Pentachlorobiphenyl	22.00/10 mg	New
PC5326R	2, 4, 5, 2', 5'-Pentachlorobiphenyl	35.00/50 mg	New
PC5501	2, 3, 4, 5, 6-Pentachlorobiphenyl	28.00/100 mg	
PC6331D	2, 3, 4, 2', 3', 4'-Hexachlorobiphenyl	32.00/20 mg	New
PC6338	2, 3, 4, 2', 4', 5'-Hexachlorobiphenyl	28.00/10 mg	New
PC6331H	2, 3, 6, 2', 3', 6'-Hexachlorobiphenyl	36.00/20 mg	New
PC6337	2, 4, 5, 2', 4', 5'-Hexachlorobiphenyl	36.00/100 mg	
PC6331A	2, 4, 6, 2', 4', 6'-Hexachlorobiphenyl	32.00/50 mg	
PC6421F	2, 3, 4, 5, 2', 5'-Hexachlorobiphenyl	28.00/10 mg	New
PC6421G	2, 3, 5, 6, 2', 5'-Hexachlorobiphenyl	28.00/10 mg	New
PC7525	2, 3, 4, 5, 6, 2', 5'-Heptachlorobiphenyl	36.00/10 mg	New
PC8444	2, 3, 4, 5, 2', 3', 4', 5'-Octachlorobiphenyl	26.00/20 mg	New
PC8446	2, 3, 5, 6, 2', 3', 5', 6'-Octachlorobiphenyl	26.00/20 mg	
PC9542	2, 3, 4, 5, 6, 2', 3', 4', 5'-Nonachlorobiphenyl	36.00/10 mg	New
PC0551	2, 3, 4, 5, 6, 2', 3', 4', 5', 6'-Decachlorobiphenyl	7.00/100 mg	

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Bibliography on PCB literature available on request.

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REFERENCE STANDARDS FOR POLLUTANTS

Analabs is your one-stop supply source for reference standards and reagent chemicals for monitoring environmental contaminants.

A threat to our environment exists from contamination by persistent organochlorine compounds. Pesticide, and more recently polychlorinated biphenyl (PCB) residues from animal and plant life have received the attention of the environmental scientists. There is also a need to look into other persistent and potentially harmful organochlorine compounds, as well as different classes of environmental contaminants.

Pesticide Standards
(Analabs, Inc. Cat. No. 16, pp 118-9)

99% PCB Isomers
(Analabs Research Notes, 12, (1), pp 8-9, Feb. 1972 and this issue)

Polychlorinated Terphenyls-Used as plasticizers in chlorinated rubbers, paint and marine finish formulation. (See list of compounds below).

Polychlorinated Naphthalenes – Used in lubricants, engine capacitors (See list of compounds below).

Polychlorinated Dibenzo-p-dioxins – Show up as impurities in processes involving chlorophenols and organochlorine pesticides (See list of compounds below).

Polychlorinated Dibenzofurans – Suspect in commercial PCB formulation (See list of compounds below).

Other Pollutant Standards – such as hexachlorophene – used in soap and toiletry – formulation and perchlorinated p,p' – DDE (See list of compounds below).

99% POLYCHLORINATED TERPHENYLS

PCT's in the environment may originate from commercial mixtures such as Arocks 5442 and 5460. For instance, the presence of Aroclor 5460 was detected in herring gulls and confirmed by comparing the perchlorinated field sample to authentic tetradecachlorterphenyl.

Reference: Polychlorinated terphenyls in the environment. V. Zitko, O. Hutzinger, W. D. Jamieson and P. M. K. Choi, Bull. Environ. Contam. & Toxicol., 7, 200-1 (1972).

99% PURE PCT's

PCT00001A	o-Terphenyl	$ 7.00/100 mg
PCT00001B	m-Terphenyl	7.00/100 mg
PCT00001C	p-Terphenyl	7.00/100 mg
PCT42021A	2,5,2",5"-o-Terphenyl	45.00/10 mg
PCT42021B	2,5,2",5"-m-Terphenyl	45.00/10 mg
PCT42021C	2,5,2",5"-p-Terphenyl	45.00/10 mg
PCT99999A	Tetradecachloro-o-terphenyl	30.00/50 mg
PCT99999B	Tetradecachloro-m-terphenyl	30.00/50 mg
PCT99999C	Tetradecachloro-p-terphenyl	30.00/50 mg
PCT10001	PCT-Kit (1 gm each of Aroclor 5432 (32% Cl), Aroclor 5442 (42% Cl) and Aroclor 5460 (60% Cl)	7.00/kit

DSW 029380

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POLYCHLORINATED BIPHENYL MIXTURES

PC0002	PCB-Kit (1 gm each of Aroclor 1232, 1242, 1248, 1254, 1260 and 1268)	9.00/kit

99% POLYCHLORINATED NAPHTHALENES

N0005	Naphthalene	$ 7.00/500 mg
C0660	1-Chloronaphthalene	7.00/100 mg
C0661	2-Chloronaphthalene	7.00/100 mg
C0664	1,2,3,4,5,6,7,8-Octachloronaphthalene	15.00/50 mg
PCN001	Halowax kit (1 gm each of 1031,1000, 1001, 1099, 1013, 1014, 1051)	10.00/kit

Halowax Number	1031	1000	1001	1099	1013	1014	1051
Approx. % Chlorine	22	26	50	52	56	62	70

Number of Chlorines	1	2	3	4	5	6	7	8
% of Chlorine	21.85	36.40	46.00	53.38	59.07	63.6	67.2	70.3

Reference: Behavior of chlorinated naphthalenes in analytical methods for organochlorinated pesticides and polychlorinated biphenyls. J. A. Armour and J. A. Burke, JAOAC, 54, 175-7 (1971).

99% POLYCHLORINATED DIBENZO-P-DIOXINS

PCDD0001	Dibenzo-p-dioxin	$15.00/50 mg
PCDD2111	2,7-Dichlorodibenzo-p-dioxin	25.00/50 mg
PCDD8441	1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin	20.00/50 mg

References:

1. Thin-layer chromatographic separation of two chlorodibenzo-p-dioxins from some polychlorinated biphenyls and organochlorine pesticides. D. T. Williams and B. J. Blanchfield, JAOAC, 54, 1429-31 (1971).
2. Determination of polychlorodibenzo-p-dioxins in chlorophenols and lipids. D. Firestone, Paper presented at the 162nd National ACS Meeting, Pesticide Div., Sept. 12-17, 1971.

99% POLYCHLORINATED DIBENZOFURANS

PCDF1000	Dibenzofuran	$ 7.00/500 mg
PCDF8440	1,2,3,4,5,6,7,8-Octachlorodibenzofuran	15.00/50 mg

Reference: Identification and toxicological evaluation of chlorinated dibenzofuran and chlorinated naphthalene in two commercial polychlorinated biphenyls. J. G. Vos, J. H. Koeman, H. L. van der Maas, M. C. Ten Noever de Brauw and R. H. de Vos, Food Cosmet. Toxicol., 8, 625-633 (1970).

OTHER POLLUTION STANDARDS

B0841	Hexachlorophene [2,2'-Methylenebis-(3,4,6-trichlorophenol)]	$ 5.00/1 mg
PC-SP0080	Perchlorinated p,p'-DDE	20.00/50 mg

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POLYURETHANE-FOAM PLUGS

For complete extraction of pesticides and PCB's from environmental water samples (for instance – river water).
References:

1. The extraction and recovery of polychlorinated biphenyls (PCB) using porous polyurethane foam. H. D. Gesser, A. Chow, F. C. Davis, J. F. Uthe, and J. Reinke. Anal. Lett., 4, 883-6 (1971).
2. Extraction of organochlorine pesticides from water by porous polyurethane. J. F. Uthe, J. Reinke and H. Gesser, Environmental Letters, 3, 117-135 (1972).

Written procedure for extraction of pesticides and PCB's from water samples will be supplied with each order or on request.

Polyurethane-foam plugs (22mm x 38mm)

PFP-U	Untreated	$ 9.00/50
PFP-W	Washed	12.00/25
PFP-DC200	Treated with 0.5% DC-200	18.00/25

Chromatographic Tubes (20mm ID, 400mm long)

CT-1	Tube without fritted disc	$6.00/each
CT-2	Tube with fritted disc	9.00/each

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ANALYSIS OF POLYCHLORINATED AROMATIC CONTAMINANTS BY PERCHLORINATION TECHNIQUES Ref. 1,2,3

Semiquantitative results can be obtained easily by subjecting the complex mixtures isolated from field samples to perchlorination and analyzing for the completely chlorinated compounds by gas chromatography. With an electron-capture (EC) detector, the response will be much enhanced (resulting in increased sensitivity). Complexity of peaks (from isomers) as well as interfering peaks (from compounds with similar retention time) will be reduced in that each class of compounds is converted to a single derivative.

We have prepared a kit of three different reagent mixtures (A_1 + A_2, B and C in sulfuryl chloride solutions) that have been proven to handle all of the following chloroaromatics: Ref. 1

Chlorobiphenyls (PCB's)

Chloroterphenyls (PCT's)

Chloronaphthalenes (PCN's)

Chlorodibenzo-p-dioxins (PCDD's)

Chlorodibenzofurans (PCF's)

p,p'-DDE

For PCB's, PCDD's, PCF's, and p,p'-DDE use perchlorinating mixture A_1 and A_2 (in equal volumes).

For PCB, PCT and p,p'-DDE use perchlorinating mixture B.

For PCN's use perchlorinating mixture C.

References:

1. Analysis of chlorinated aromatic hydrocarbons by exhaustive chlorination: Qualitative and structural aspects of the perchloro derivatives of biphenyl, naphthalene, terphenyl, dibenzofuran, dibenzodioxin and DDE. O. Hutzinger, S. Safe, and V. Zitko. (in press).

2. Column chromatographic separation of polychlorinated biphenyls from chlorinated hydrocarbon pesticides, and subsequent gas chromatographic quantitation in terms of derivatives. O. W. Berg, P. L. Diosady and G. A. V. Rees (in press).

3. Screening method for the detection of chlorodibenzo-p-dioxins in the presence of chlorobiphenyls, chloronaphthalenes, and chlorodibenzofurans. D. T. Williams and B. J. Blanchfield, JAOAC. 55, 93-5 (1972).

PCR-A	Perchlorination reagents A_1 and A_2	$10.00
PCR-B	Perchlorination reagent B	6.00
PCR-C	Perchlorination reagent C	6.00
PCR-K	Perchlorination kit (A_1, A_2, B, C)	16.00

Detailed perchlorination procedures will be supplied on request and with each order.

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