...te of Analytic Chemistry
...ity of Stockholm
... STOCKHOLM 50

THE OECD STUDY OF ANALYSIS OF PCB

December 1968

Notes concerning samples sent to OECD members September 1968: ampoule 1 - synthetic mixture of PCB fractions from preparative GC; ampoule 2 - industrially-used PCB mixture.

PCB (polychlorinated biphenyls) is the abbreviation used by this Institute for the 'residues' of the mixture of compounds found in nature in residue analysis for chlorinated pesticides. According to the technical literature, PCB - under several trade names - has been in considerable use for a variety of technical purposes since 1929, but never as a pesticide. It is assumed in this laboratory that all technical PCB is manufactured by the chlorination of biphenyl.

We have proved by mass spectrometric analysis that the PCB mixture most often used industrially consists of compounds containing between four and eight chlorines per molecule. If it is assumed that the chlorines are distributed between the rings almost equally, 102 different PCB compounds are possible, as the enclosed report discusses. We have shown that at least 24 of these are present; since some may have similar retention volume, this number may be larger.

Owing to the large number of PCB compounds possibly present, quantitative gas-chromatographic analysis is difficult. Using an electron capture detector the response of closely related isomers may vary considerably, and thus a correct scheme for calibration of individual ECD-responses has to be found. Since different methods of technical manufacture are very likely to give somewhat different isomeric composition, further calibrations may have to be done during the quantitative analysis of an unknown technical sample. In residue analysis a further complication may arise from the partial metabolism of PCB, which may increase the content of the more stable PCB isomers.

If these limitations are born in mind, it is clear that quantitative residue analysis of PCB cannot yet be very accurate. However, the wide-spread presence in nature of PCB demands prompt analytical activity and the two samples (PCB 1 and 2) now circulated among the OECD laboratories together with the results given in this note may be found useful in standardising the analysis of PCB within the above limitations.

We have attempted, in the following way, roughly quantitative analyses for the compounds which produce the 14 peaks marked on the enclosed chromatograms. While resolution is incomplete, the numbers of peaks and their relative areas were the same for both columns. It was therefore possible to determine by GC/MS the number of chlorine atoms per molecule in the major components of

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each peak. The total chlorine present in the original PCB mixture, and its distribution between the different peaks, was determined by GC-microcoulometry using the same column. These values were used to determine the amounts of individual PCB components present in each peak, and hence EC-detector responses. The table lists data obtained for the original PCB mixture (ampoule 2).

Analysis[x] of industrially-used PCB mixture (OECD ampoule 2)

Peak No	number of chlorine per molecule	%
1	4	7.4_9
2 + 2'	5 + 4 (ca 35% + 65%)	11.0_4
3	5	16.8_8
4	5	5.8_4
5	5	11.4_9
6 + 7	6 + 5 (ca 35% + 65%)	15.3_1
8	6 + min of 5	12.6_5
9 + 10	6 + 6	11.5_9
11	6	2.1_9
12	8 ev min amount of 7	2.4_7
13	8 " " "	2.0_3
14	8 " " "	1.0_3
		100.

x) Data obtained on SF-96 column with EC-detector

In our routine analyses we have up to now reported the total sum of PCB components. We realize that this procedure is not strictly correct, and it may be better in future to sum over each of the five individual molecular weights.

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S.Jensen
B.Nucci
G.Widmark

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