U.K. Agency 103

MONSANTO CHEMICALS LIMITED
RESEARCH AND DEVELOPMENT DEPARTMENT
RUABON

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CONFIDENTIAL

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H.A. Vodden, Ruabon
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W.R. Richard, St. Louis
M.W. Farrar "
E.P. Wheeler "
R.E. Keller "
E.S. Tucker "
R.A. Baxter, Ruabon
N.E. Williams, Newport
D.V.N. Hardy, London
Ruabon T.I.C. (Restricted Access)

INDUSTRIAL CONTACT REPORT

AT: The Laboratory of the Government Chemist,
Cornwall House,
Waterloo Bridge Road,
London.

BY: R.A. Lidgett.

DATE: Friday, 31st November 1969 (p.m.)

PRESENT: Mr. G.B. Collins
Dr. D.C. Abbot - Part time.

This visit was made with the understanding that the discussion would be concerned with methods and technique only, the results obtained for Monkswood on the sea birds obtained recently from the Irish Sea could not be discussed at the present time.

Most of the time was spent with Mr. Collins, although Dr. Abbot did join the discussion for a short period.

Sources of P.C.B.'s

This was raised by both Collins and Abbot and questions were also asked about the method of manufacture (reproducibility etc.), the latter being concerned with the appearance of unexplained peaks in chromatograms of extracts containing P.C.B. (see later).

Apart from the extracts from sea birds and their eggs, "P.C.B." chromatograms have been obtained from mutton fat (possibly only a single sample) and foods (butter?), but it was not suggested that this area of P.C.B. contamination is being actively pursued at present. In addition, "P.C.B.'s" have been found in London rain water (level not given); it was emphasised that at present "P.C.B.'s" had not been detected in either river or sea water - levels are too low for the present method.

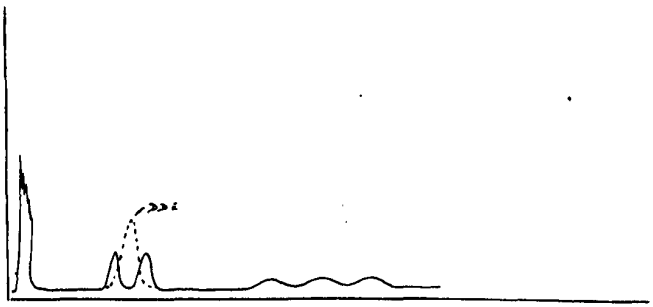
Identification and Measurement

Qualitative identification of extracts suspected of containing P.C.B.'s is carried out by comparison of their T.L.C. and gas chromatographic behaviour with that of a commercial P.C.B. (generally Aroclor 1260 or 1262).

The laboratory carries out routine monitoring of eggs for P.C.B.'s and pesticides and it is evident that there is not sufficient time available to subject every extract to a rigorous qualitative examination. Thus, nominally straightforward samples are considered to contain P.C.B.'s if the components have similar T.L.C. behaviour and if the retention times on one gas chromatographic column compare well with that of the commercial material. If samples require a more detailed analysis, the comparison of retention times is extended by the use of a second and if necessary a third g.c. column (the three columns used have packings with the following stationery phases: Apiezon L, silicone oil and cyano silicone oil). No mention of g.c./mass spectrometry for absolute confirmation of identity was made.

While at the laboratory, a number of chromatograms were examined from previous work on a variety of extracts and from the egg monitoring project. These were less convincing than the chromatograms of extracts containing P.C.B's published in the literature for a number of reasons :-

(a) The chromatograms of the egg extracts were comparatively simple (see sketch) and did not have the complex fingerprint expected from a commercial P.C.B.



(b) Some of the chromatograms were not suggestive of "clean" samples. Later peaks merged to give envelopes with high points and it would be difficult in these cases to rely on retention time as a reliable means of identification.

(c) As far as could be ascertained, the P.C.B. standards were either 1260 or 1262 - no mention was made of the use of other P.C.B.'s.

(d) It has been found that in tabulating the retention times of peaks in complex extracts that peaks are observed which do not relate easily to the commercial P.C.B. It was suggested that this might be due to differences in the manufacturing methods of various companies, but two other explanations are also possible :-

(i) Composite peaks may alter in composition causing change in RT, e.g.



(ii) Other components than P.C.B.'s may be present. This was considered possible, but unlikely.

The equipment used in this laboratory is made up of components from various chromatographs; the detectors are built on site and incorporate a tritium source. While the equipment appeared to be functioning well on a routine basis, no comment was made about contamination of the detector from high boilers. From the size of the major peaks in the egg extracts ($\sim$ 10% E.S.D. for low retention time components and 2 - 5% F.S.D. for high retention time components, see previous sketch), it may be that certain of the higher boiling P.C.B's (if present) are being missed. Because of the emphasis on the determination of pesticides (and now P.C.B.) analyses are carried out almost completely with electron capture detection. The flame ionisation detector is not used routinely because of the interference of non pesticide components in the extracts.

The above observations have been made from a limited amount of information. Although the techniques used were discussed freely, extension to results and interpretation of all data was definitely discouraged. This situation may change if the Monkswood figures are published.

At the present time it is unlikely that frequent contact with the Laboratory of the Government Chemist will be advantageous to Monsanto.

R. A. Lidgett

R.A. LIDGETT

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