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To

OECD Study of Pesticide
Residue 1967-68

Results obtained by analysis of the OECD exchange samples 1, 3 and 4.

Sample No 1.
(Standard prepared in Holland)

The sample was diluted 200 times with petroleum ether (b.p. 60-70°C), and 5 to 10 µl aliquots were injected into the gas chromatograph (Varian Aerograph 204). The stationary phase was a mixture (modified as below) of SF-96 and QF-1 (1:3); 5 % total coating on silanized Gas Chrom P (100-120 mesh). The column (glass; length 150 cm; i.d. 1,8 mm) was prepared by blending in small portions of the two coatings.

After conditioning the column for 24 h or more the flow of carrier gas was adjusted (usually 25-28 ml/min) to give air a retention time of 13 sec. The temperature of the column was selected (usually 180-190°C) to give ppDDT a retention time of 20.0 minutes.

The preliminary analyses of sample No 1 showed the presence of the following pesticides, eluted in the order given and separated almost totally on the column used:

Heptachlor epoxide, ppDDE, Dieldrin, Endrin, ppDDD and ppDDT.

The two pesticides, Heptachlor epoxide and Endrin, are not in common use in Sweden, and thus a separate analytical investigation was performed at this institute. Endrin was found to decompose on the stationary phase described, and Heptachlor epoxide gave irreproducible results. Additional impregnation of the stationary phases (with 0.17 % Epikote 1001) was found to result in a sufficient deactivation demonstrated by studies of recovery of Endrin. A simple change of an old reference standard of Heptachlor epoxide was sufficient to give acceptable analytical results for this compound.

Seven analyses of the sample No 1 gave the following result, reported as ng/ml of the undiluted solution delivered.

Sample No 1

	ng/ml mean value	mean deviation
Heptachlor epoxide	3800	± 300
pp DDE	5100	± 200
Dieldrin	5500	± 300
Endrin	6600	
pp DDD	10400	± 300
pp DDT	10200	± 500

Further qualitative analyses were made by separate injections of the sample on SF-96 or QF-1 columns, and after pre-treatment of the sample with fuming sulphuric acid and potassium hydroxide respectively. No indications were found for the presence of compounds other than those reported.

Sample No 3
(cod liver oil from Scotland)

Since this sample contains PCB compounds a direct method has been chosen for this study. However, this method only allows the detection of very stable compounds and all oxygen containing pesticides are decomposed at the cleaning up stage of the method.

Procedure: 200 mg of the oil were dissolved in 5 ml of hexane and shaken for 1-2 minutes with a 1 ml of mixture (1:1) of conc sulphuric acid and fuming sulphuric acid (fuming sulphuric acid; 10 % SO_3). The organic phase, after being washed and then dried, was injected into the column described.

The above mentioned columns gave reasonably good resolution of the following peaks of PCB: 2,3,5,8,10,11,12,13 and 14. None of these coincided with any pesticide peak found in the sample. Among the pesticides we were able to detect, ppDDE and ppDDT do not interfere with any of the PCB peaks, but ppDDD overlaps PCB 9, a minor component. The peaks of opDDT and PCB 7 also overlap, as well as those of opDDD and PCB 6. However, the last two generally appear as very small peaks which can be neglected (but not in this sample!). The usually-obtained pattern of PCB residue analysis, especially the relation of the height of peaks 7, 8 and 10, leads us to assume that in this sample half of the peak in the position of PCB 7 is given by opDDT.

A quantitative estimation of the amount of Dieldrin present in sample No 3 was separately made by measurement of the difference in peak heights between chromatograms of an extract cleaned-up by acetonitrile - petroleum ether partition and chromatograms of the same extract after treatment with the acid mixture discussed above.

Sample No 3

Results given in ng/g cod liver oil as delivered

Stable pesticides

pp DDE	400	
op DDD	240	(PCB interference)
pp DDD	490	
op DDT	85	(the rest of the peak reported as PCB 7)
pp DDT	550	
Dieldrin	90	
PCB		
2	80	
2'	90	
3	260	
5	70	
7	85	
8	140	
9	-	obscured by ppDDD
10	140	
11	90	
13	80	
14	50	
	(1085)	1100 ng/g

Sample No 4

(Sample of egg prepared in Holland)

A portion (0.5 g) of egg powder in the ampoule was shaken (15 minutes) in a stoppered test tube together with 10 ml of a hexane-benzene mixture (1:1). After standing overnight the tube was shaken again and then centrifuged. The extract was injected without further treatment into the column previously described.

Sample No 4	ng/g (ppb) mean value	mean deviation
DDE	34.1	± 1.4
Dieldrin	44.1	± 2.3
Endrin	39.1	± 1.3
pr DDD	4.0	± 1.0
pp DDT	103.0	± 4.6

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