Analytical Chemistry
Special Study 70-11
Job No. 1630346

DETERMINATION OF PCB RESIDUES IN FISH FROM LAKE MICHIGAN

INTRODUCTION

Upon ammendment of the WARF Institue Lake Michigan pesticide investigation to include analysis of selected samples for apparent polychlorinated biphenyls (PCB's), Monsanto as an industrial sponsor agreed to carry out collaborative analysis of a limited number of selected samples for PCB's. The results of our analysis of sample extracts supplied by WARF are reported.

SUMMARY

Four fish extracts and one human biopsy extract were analyzed by EC/GC and GC/mass techniques. The presence of all chlorinated hydrocarbons as reported by WARF in two of the samples were confirmed with the exception of BHC which was not analyzed for. Additionally a sample (#9070652) contained lower levels of two unidentified chlorinated compounds observed by GC/mass. PCB's typical of Aroclor 1254 were found in all of the above samples.

MONSANTO COMPANY
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4-70 - E. S. Tucker, J. Litschgi, R. E. Keller

WARF Sample No.	Sample Type & Location	Sample Wt (g)	PPM DDE WARF	PPM DDE Mon	PPM DDD WARF	PPM DDD Mon	PPM DDT WARF	PPM DDT Mon	PPM Dieldrin WARF	PPM Dieldrin Mon	PPM EC WARF	PPM EC Mon	PPM FCE Estimated WARF	PPM FCE Estimated Mon	PCB [illegible]
9070876	Lake Herring, Fish Extracts Manistee R., Mich., Sample Point No. 52	12.0	2.90	2.54	1.17	—*	1.31	—*	0.13	0.08	0.034	—**	4.37	2.31	?
9070770	Alewife, Perch Extracts Burns Ditch, L. Mich., Sample Point No. 37	12.0	1.56	1.28	1.64	—*	0.13	—*	0.14	0.16	0.030	—**	2.33	2.73	+
9070652	Chub Extracts Manitowoc R., Wis, Sample Point No. 18	12.0	2.34	3.46	0.52	—*	1.31	—*	0.17	0.34	0.021	—**	0.87	1.56	+
9070639	Chub Extracts Kewaunee R., Sample Point No. 15	12.0	2.08	3.05	0.35	—*	1.07	—*	0.12	0.17	0.021	—**	1.35	0.75	?
9120924	Human biopsy Fat Extracts	0.13	2.93	5.48	<0.01	—*	0.21	—*	0.21	—***	0.31	—**	10.1	7.69	o

* Observed by EC/GC Not Analyzed For
** Not Analyzed For
*** 2nd Elution Not Sent

TLCC TABLE II

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TABLE II Relative Gas Chromatographic Retention Times, R_x (DDE = 1.21)

Aroclor 248	Aroclor 1254	Aroclor 1260	# 9070 876 1st Elution	# 9070 876 2nd Elution	9070 770	# 9070 770 2nd Elution	9070 652 1st Elution	#9070 652 2nd Elution	9070 639 1st Elution	#9070 639 2nd Elution	9120 924
0.21	0.43	0.69	0.27	~~0.43~~ 1.21*	0.27	1.21*	0.27	0.79	0.27	0.78	0.26
0.27	0.52	0.75	0.35		0.35		0.35	1.21*	0.35	0.98	0.34
0.31	0.54	1.00	0.43		0.43		0.43		0.43	1.06	0.42
0.32	0.60	1.12	0.53		0.53		0.53		0.53	1.21*	0.49
0.35	0.66	1.29	0.54		0.55		0.54		0.56		0.52
0.40	0.70	1.51	0.60		0.61		0.62		0.62		0.55
0.41	0.79	1.66	0.68		0.68		0.68		0.69		0.62
0.52	0.85	1.72	0.72		0.71		0.71		0.72		0.67
0.54	0.94	1.93	0.82		0.81		0.81		0.82		0.71
0.60	1.03	2.13	0.88		0.89		0.88		0.88		0.82
0.68	1.19	2.22	1.00		1.00		1.00		1.00		1.00
0.72	1.41	2.45	1.12		1.14		1.12		1.13		1.12
0.82	1.60	2.66	1.22		1.24		1.24		1.24		1.14
0.87	2.03	3.00	1.31		1.48		1.32		1.34		1.23
0.94	2.18	3.54	1.45		1.53		1.46		1.47		1.45
1.04	2.51	4.31	1.50		1.64		1.65		1.66		1.66
1.21	3.34	5.50	1.63		2.24		2.26		2.26		2.26
1.59	—		2.25		2.48		2.46		2.47		2.65
			2.46								

*Dieldrin

FM-402 Biomedical G.C./N_2^{63} ECD
6mm x 6' Glass Column, 4% XE-60 on
80/100 mesh Chromosorb W, HP, AW-DMCS
mp. point – 205°C column – 1[illegible] detector – [illegible]

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II GC/Mass Results

Sample No 9070376 — Sample lost due to punctured septum.

Sample No. 9070770 — tetra- and penta-chlorobiphenyl homologs detected.

Sample No. 9070652 — DDE, tri-, tetra, penta, and hexa-chlorobiphenyl homologs detected.
Two un-identified chlorinated hydrocarbons with molecular weights of 370 and 404 respectively and 3 chlorinated pesticides detected.

Sample No. 9070639 — Ion beam of mass spectrometer during GC/mass scan destroyed due to large amount of extraneous material present in the sample extracts.

Sample No. 9120924 — Amount of chlorinated hydrocarbons below the present detection capabilities of our GC/Mass system.