PROGRESS REPORT
ORGANIC CHEMICALS DIVISION
TECHNOLOGY DEPARTMENT

REPORTED BY	FOR	DETAILS	REPORT NO.	DATE
W. J. Litschgi W. M. Mees J. P. Mieure E. S. Tucker	Sept. 1971	YES	2	9-28-71

LOCATION	JOB NOS
St. Louis - S. Second Street	2-21-760.01-13480, 85010, 16300

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PROJECT TITLE

ENVIRONMENTAL ANALYTICAL STUDIES - RESEARCH AND DEVELOPMENT, MANUFACTURING AND OUTSIDE SUPPORT STUDIES

PROJECT OBJECTIVE

Identify, locate and assess the magnitude of Aroclor pollution problems, help determine what needs to be done to control pollution sources, verify the effectiveness of proposed pollution control procedures, guide development of recycle and/or disposal methods, and provide collaborative data for legal and regulatory purposes.

SUMMARY

During the month of September ~60 environmental and research samples have been analyzed for polychlorinated biphenyls (PCB's) by electron capture gas chromatography. Analysis of these samples represents a minimum man power expenditure of 210 hours.

PCB's, typical of our Aroclors, have been found at levels below and above action levels in:

- Fish meal, soybean meal and meat meal (animal and poultry fed components)
- Paperboard and polyethylene film (food packaging materials)
- Environmental samples from three customer sites (oil, sediment, and water)
- Two Monsanto products
- Fish and sewer effluents from two Monsanto manufacturing sites

An over view of the analytical results is shown in Table I in the detail section.

Additional external and internal support has been provided in the form of:

- On site PCB methodology training for laboratory personnel from Ralston-Purina, St. Louis; St. Regis Paper Co., Dallas, Texas; and Monsanto, Ag Div., Nitro, West Virginia
- Attendance at pertinent meetings on PCB problems (Working Group on Pesticides, Washington, D.C.; National Broiler Council, Scientific Advisory Committee and Lake State's Governors Interdisciplinary Pesticide Conference)
- General internal and external analytical consultation
- Distribution of Aroclor methodology and reference standards
- Preparation of, and participation in, inter- and intra-laboratory checks on PCB methods of analysis

FUTURE PLANS

1. J. F. Queeny monthly sewer effluent composites.
2. St. Regis Paper Company - Polyethylene film and paperboard samples.
3. Distribution of PCB's in whole milk (fat vs skim).
4. Complete PCT extraction and separation work.
5. Request pertinent plant effluent samples from Anniston for PCT analysis.
6. Complete recovery check and write up of methods for determination of PCB's in Santoquin and CaMHA.
7. Evaluate Dr. O.W. Berg's PCB perchlorination technique for quantitation of altered PCB residues.
8. Analysis of MCS 1016 and Kanechlor for polychlorinated dibenzofurans.
9. Complete inter-laboratory PCB methodology check.
10. Participate in panel discussion of PCB methodology.
11. Analysis of USDA turkey tissue extracts to help establish contamination source(s).

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CUSTOMER SERVICE

Sample Source	Sample Matrix	No. of Samples	Level Found	Associated Aroclor	Support Objectives
Ralston-Purina St. Louis, Mo.	Fish, meat & soybean meal	10	PPM	A-1242	Determination of level and source of PCB contamination in animal feed components
Westinghouse S. Boston, Va.	Oil, water & sediment samples	7	PPB-PPM	A-1242	Pollution Control
National Cash Register Dayton, Ohio	a) NCR 24-hour sewer sediment	1	PPB	A-1242	Pollution Control
	b) Wash solution from 14.3.1, wash job	1	PPM	A-1242	
Frigidaire Div., GM Corp. Dayton, Ohio	Monthly effluent composites from plants 2 & 3	4	PPB	A-1242	Pollution Control
St. Regis Paper Co. Dallas, Texas	Polyethylene film & paperboard	2	ND*to PPB	A-1242	Pollution Control

MANUFACTURING SUPPORT

Ag Division Nitro Plant	Santoquin	7	PPB	A-1242	Determination of level and source of contamination of animal feed additives
	MHA	4	PPB	A-1242	
J.F. Queeny	Sewer effluent composites	8	PPB	A-1242	Pollution Control
Pensacola, Fla.	Fish	2	PPB	A-1254	Determination of environmental contamination

*ND = None Detected

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RESEARCH AND DEVELOPMENT

Sample Source	Sample Matrix	No. of Samples	Level Found	Associated Aroclor	Support Objectives
Inter-plant Round Robin (WGK, Anniston, & Applied Sciences)	Water spiked with Aroclor	8	-	A-1242 A-1254 A-1260	Insure the maintenance of precision and accuracy in Monsanto labs analyzing for PCB's
National Broiler Council Round Robin	Chicken fat spiked with Aroclor	4	-	A-1242	Cooperative government-industry PCB methodology check

DETAILS (Cont'd)

OUTSIDE SUPPORT STUDIES

Ralston-Purina Company

The results of the analysis of 6 meal samples submitted by Ralston-Purina were as follows;

Sample No.	Sample Description	ppm PCB's(Aroclor 1242) Ralston	Monsanto
661	Fish Meal (69196)	12	29
662	Meat Meal (69197)	11	25
663	Soybean Meal (69200)	4	9
664	Meat Meal (69201)	13	32
665	Soybean Meal (69202)	6	11
666	Soybean Meal (69203)	<0.5	0.6

As the table indicates, with the exception of the lowest containing PCB sample, our results are not in agreement. The fairly consistent factor, 2-2.5X between the numbers leads me to believe that they are not extracting the PCB quantitatively from the meal.

Subsequent analyses of four of these samples, spiked at a level equivalent to that found, indicates that our procedure recoveries were in the range of 100-106%. The actual recoveries are shown in the following table.

Sample No.	Sample Description	Level of PCB's Added	Total Found	% Recovery
661	Fish Meal (69196)	21 PPM	53 PPM	106
662	Meat Meal (69197)	11 PPM	36 PPM	100
664	Meat Meal (69201)	12 PPM	44 PPM	100
665	Soybean Meal (69202)	11 PPM	23 PPM	105

Approximately 1-1/2 days were spent in familiarizing D. Viviano, Supervisor, Fat Soluble Laboratory - Ralston-Purina, with Monsanto PCB methodology.

Frigidaire Division, GM Corporation

Sample No.	Sample Description	PCB Level Found	Aroclor
600	Plant 2 (June)	<0.3 PPB	1242
601	Plant 3 (June)	0.8 PPB	1242
651	Plant 2 (July)	<0.1 PPB	1242
652	Plant 3 (July)	1.2 PPB	1242

The results shown have also been corrected for the 0.3 ppb background value.

NCR - Dayton, Ohio

Sample No.	Sample Description	PCB Level Found	Aroclor
667	Wash solution from 14.3.1, wash job, installation after one week use, Finishes Control Labs, NCR Co. 7-26-71	1.58 PPM	1242
653	NCR Sewer Composite (7-15-71)	<0.4 PPB	1242

Our calculated detection limit based upon observed background and sample size was 0.3 PPB. The results shown have been corrected for the 0.3 PPB blank. After orally reporting these results to Paul Henry, he indicated that they would not need much more support analysis. Possibly one more set to confirm the low PCB level observed in their 7-15-71 sewer composite sample.

Westinghouse, S. Boston, Virginia

Sample No.	Sample Description	PCB Level Found
	Oil skimmed from oil separator	8.06%
645	Final effluent from oil separator	95 :
646	Effluent from neutralizer plant	144 PPB
647	Lawson's Creek	0.5 PPB
648	Dan River, above confluence with Lawson's Creek	1.5 PPB
649	Dan River, below confluence with Lawson's Creek	1.1 PPB

St. Regis Paper Company, Dallas, Texas

Paperboard and polyethylene extracts, used to package food products, were analyzed by EC/GC for PCB's. The polyethylene film extract contained no apparent PCB's while the paperboard contained ~0.5 PPM. J. Box and E. Albert visited our laboratory and were familiarized with Monsanto PCB methodology.

MANUFACTURING SUPPORT WORK

Ag Division

Sample Description	PCB Level Found	Aroclor
Santoquin Composite		
8-30-71	146 PPB	1242
9-2-71	108 PPB	1242
Santoquin Distillation Leg		
9-3-71	91 PPB	1242
Santoquin Composite		
9-19-71	111-405 PPB	1242
Detection Limit	89 PPB	1242

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An attempt was made to decrease the PCB detection limit by increasing the sample size by a factor of 10X. Matrix interferences were encountered which required that a maximum and minimum be reported (Santoquin composite 9-19-71).

GC/Mass analysis of the residues extracted from Santoquin verified that if PCB's are present they would have to be at a level less than 0.5 PPM.

Spiking of three samples of Santoquin at the 1 PPM level with A-1242 yielded recoveries of 73%, 92% and 96%. A tentative method will now be written but further research is planned in order to remove EC/GC interferences.

Sample Description	PCB Level Found	Aroclor
CaMHA:Na 09-46	18.5 PPB	1242
CaMHA:Na 09-46	237 PPB	1242
Detection Limit	15 PPB	1242

Spiking of A-1242 at a 2 PPM level to CaMHA yielded a recovery of 96%.

PLANT SUPPORT

J. F. Queeny Plant

Sample No.	Sample Description	PCB Level Found	Aroclor
589	MSD Gatewell (June)	12 PPB	1242
599	Clean Acid Sewer (June)	14 PPB	1242
591	DC Sewer (June)	13 PPB	1242
654	Clean Acid Sewer (July)	22 PPB	1242
655	MSD Sewer (July)	99 PPB	1242

The sample "DC Sewer (June)" was supposed to be the "Clean Acid Sewer (June)" spiked with ~10 PPB Aroclor 1242. As you can see, our analytical results do not reflect the added PCB's. I have talked to Al Peterson about this apparent discrepancy and it was decided that spiked samples would be included with their August set to see if this problem can be worked out.

Pensacola, Florida

Sample Description	PCB Level Found	Aroclor
Large Crappie (Fish)	494 PPB	1254
Small Shell Cracker (Fish)	130 PPB	1254

Both fish were skinned and fileted and only the edible portion of each fish was used for analysis as requested.

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RESEARCH AND DEVELOPMENT

Inter-Plant PCB Methodology Check

Duplicate sets consisting of eight water and six fat samples spiked with production Aroclors at various levels have been prepared and distributed to the plant laboratories. The results of each laboratory analysis of these samples are to be compared to insure consistent internal reporting.

National Broiler Council Intra-Laboratory PCB Methodology Check

Ten sets of soybean oil and chicken fat samples were prepared by Monsanto for distribution by Ralston-Purina to government and industrial laboratories analyzing samples for PCB's. The objective of this intra-laboratory check is to establish the validity of the different PCB methods currently being employed.