April, 1970

ENVIRONMENTAL AROCLOR PROJECT

It appears that disposal of PCB's can be accomplished successfully by thermal oxidation. This conclusion was made after analysis of gas samples from John Zink Company. They were analyzed for PCB, chloride, chlorine, and total organic chlorine. Data were sent to CED and a report is being issued. Additionally, heats of combustion of the PCB's were estimated for CED evaluations.

Water and sediment samples from 6 Pydraul customers were analyzed for PCB's. It is concluded that PCB's are finding their way to sewers, streams and lakes inasmuch as amounts ranging from low ppb to high ppm were determined.

EC/GC determinations for PCB's showed low levels of materials in muriatic acid from Monsanto's central drumming facility. These contaminants appear to be lower chlorinated biphenyls. Confirmation studies are underway.

Paint scrapings from silos and raw milk extracts showed presence of PCB by GC/Mass and EC/GC analysis. The paint scrapings contained 2.87% Aroclor 1254. The raw milk contained an estimated 0.6-0.7 ppm PCB, probably originally Aroclor 1254 but with an altered isomer distribution.

Analytical methods and data collected to date were discussed with Westinghouse in an informational meeting on the PCB problem.

A preliminary report on biodegradation of Aroclor was written. Tentatively, Aroclor 1221 degrades in river die-away experiments. Higher Aroclor products may not. More conclusive experiments are underway. A current study involves removal of certain fractions from Aroclor products which might be intractable to biodegradation. Electron-capture chromatograms suggest that most refractory materials can be separated by distillation.

We are continuing to assist the WGK Plant in monitoring bromine content of chlorine they use to make Aroclor products. Seven samples, collected in the past month, were shown by XRF to contain 250-400 ppm bromine.

We are preparing to determine the presence of dioxins in the presence of other chlorinated materials. GC studies show that octachlorodibenzo-p-dioxin can be determined but that the tetrachloro isomer will have interference from the components in Aroclor 1260.

A better method to determine percentages of various components in undistilled Aroclor is necessary. A suitable GC method appears to have been developed as a consequence of studies made on 60 distillation cuts from 4 Aroclor samples.

SKYDROL 500

Flight tests are currently being performed on Skydrol 500C by various airlines. Only tests involving at least 80% Skydrol 500C will be

Jan 1971

SPECIALTY PRODUCTS

Aroclor Environmental Program

Ppb levels (24-30) of PCB's most similar to our production grade Aroclor 1254 and 1260 were found in cows milk from GE at Pittsfield, Massachusetts. These results will be confirmed with analyses of additional milk.

Current JFQ Bisphenol A (Lot QM-852) contains 26 ppb PCB calculated as Aroclor 1242. The chromatograms are not identical with either Aroclor 1232 or 1242.

The analysis of all fish collected in the last survey of the streams associated with our Anniston Plant for chlorinated hydrocarbons (Aroclor 1242, Aroclor 1254, and DDE) are greater than 50% completed (55 samples). The levels found in the completed samples have been forwarded to the plant personnel. They in turn have identified 6 "target" species on which we will focus future analytical efforts in order to decrease the number of man hours required to complete the analysis of this set of samples.

A research representative of Agway Company was instructed on the techniques Monsanto uses to analyze biological samples for PCB's. During his visit, commercial chicken chow, chicken muscle and eggs were analyzed for PCB's. EC gas chromatography indicated that PCB's were present in all samples analyzed including an egg composite extracted in Agway's Research laboratories. The presence of tri, tetra, and pentachloro biphenyls in the Agway extract was confirmed by GC/Mass as well as EC/GC. The estimated levels of PCB's (as Aroclor 1248) found in the feed ranged from 0.10 to 0.17 ppm and in the muscle from 0.29 to 1.55 ppm.

Aroclor 1242 Competitive Products

Thirteen samples of PCB products representing the world-wide competition to Aroclor 1242 in dielectric applications were analyzed quantitatively by GC for isomer distribution. All products including Prodelec 1499 and 1500, which were claimed to contain very little of the higher chlorinated biphenyls, were found to contain similar levels of penta (3.9 to 8.7%) and hexachlorinated (0.3 to 2.2%) biphenyls to WGK plant and MCL Aroclor 1242 products. However, MCS 1016 (Aroclor 1242 distilled to remove high boilers) was found to contain only 0.8% penta and <0.05% hexachlorinated biphenyls. This means that MCS 1016 should contribute less (by an order of magnitude) of the higher chlorinated biphenyls found in environmental samples.

Twenty-six drums of fractionated Aroclor 1142 from MRC - Dayton were analyzed by GC for isomer distribution to guide blending operations in the manufacture of MCS 1016.

Biodegradation Testing

MCNS 059807

SCAS Statistical Study

The statistical study of the semi-continuous activated sludge (SCAS) variables has now been in progress for three weeks of the

SPECIALTY PRODUCTS

AROCLOR ENVIRONMENT PROGRAM

EC/GC analyses of the January composite sample of the JFQ sewer effluent for PCB's verified that the amount being discharged is remaining low. The amount found, 13.0 ppb, is in close agreement with last month's 12.1 ppb. The internal specification for plant discharges is ~10 ppb total PCB's.

Analysis of a second composite egg sample from Agway Company again verified the presence of PCB's. The level found was 190 ppb calculated as Aroclor 1248 on a wet weight basis. "Bakery meal", a component of their chicken feed appears to be the most logical source of the PCB's found in the feed and in chickens that consume it. EC/GC analysis of a bakery meal from Agway confirmed this suspicion. 480 ppb PCB was found and the EC chromatogram of the meal extract was almost identical to earlier chromatograms of the feed extracts. Since "bakery meal" is prepared by pulverizing stale bakery goods, containers and all, it is possible that recycled coated paper may be the ultimate PCB source. Alternately, the PCB's could be introduced into the meal during processing.

Effluent samples from Mead Company were found to contain low levels of Aroclor 1242; from <1.5 ppb to 2.2 ppb except for a 24 hour composite secondary effluent which contained 24 ppb.

Comparative EC/GC analysis of duplicate Anniston plant effluent samples for PCB's were carried out at Anniston and St. Louis. The levels of PCB's found, calculated as Aroclor 1242, were 2 to 4 times greater than those reported by Anniston. The reason for the disagreement lies in the methods used to estimate the PCB level. Anniston uses the peak height technique, while we employ the total area of the envelope. The two techniques produce equivalent results when the isomer distribution of the Aroclor in question is unaltered. The search for a more absolute quantification will be continued. Additional exchange samples will be analyzed using the total area calibration procedure.

Analysis of tissues from the Bio-Test toxicity studies has not been initiated, although rat tissues from the first 12 months of the two year chronic oral study of Aroclor 1242, Aroclor 1254 and Aroclor 1260 in albino rats are in hand.

The status of the fish samples from the last survey of the water shed associated with the Anniston plant is unchanged, about 50% of the samples have been analyzed to date.

BIODEGRADATION TESTING

The eight week statistical study of the effect that seven different variables have on the SCAS disappearance rates of three model compounds is completed with the exception of a few sample extracts that remain to be analyzed by EC/GC. Variables being evaluated are:

TRIBUTYL PHOSPHATE

Conductivity data indicate that a magnetic flowmeter can be used to monitor process stream flow at 25°C for the tributyl phosphate step II reaction product. A conductance of about 200 micromhos/cm was obtained.

THERMINOL 77

Product made at Findett Corporation, were found by gas chromatography to contain from 5.5% to 7.0% 4,4'-bisphenoxybiphenyl which is well above the specification of 1.5% for premium grade. This will result in a penalty to Findett on these lots.

p-CUMYL PHENOL

Several batches of p-cumyl phenol produced in the pilot plant were found by gas chromatography to assay at 98.2 to 98.3%.

MONOCHLOROBIPHENYLS

Monochlorobiphenyl produced at MRC - Dayton was found to contain less than 1% dichlorobiphenyls by gas chromatography.

MCS 1016, 1016A and 1043

Isomer analyses were provided by capillary column GC for identification and comparison purposes on these products.

AROCLOR ENVIRONMENTAL PROGRAM

An analytical program covering the analysis of tissues and materials from toxicity work at Industrial Bio-Test the past year was started on March 15. A two man continuous effort is being directed toward establishing polychlorinated biphenyl residues for correlation with toxicity data from animal studies.

Analyses confirmed that a bakery by-product meal from Agway contains about 750 ppm polychlorinated biphenyl typical of Aroclor 1248. The source of the PCB is unknown.

Gravimetric and GC analyses showed that residual Aroclor 1242, 1254 and 1260 can be satisfactorily removed from used drums at Evans Steel Drum Company, Inc. via kerosene washes. The washes would be shipped to WGK for incineration.

Preparative gas chromatography was used to divide Aroclor 1254 into six fractions. This will be used as standards for calibrating the response of the EC/GC at higher chlorine levels.

April 1, 71

SPECIALTY PRODUCTS

AROCLOR ENVIRONMENTAL PROGRAM

Bio-Test Tissue Analysis

An inventory of all Bio-Test tissues held by Monsanto for PCB analysis has been prepared as well as an estimate of those retained by Bio-Test Laboratories. Analysis of all tissues (muscle, fat, liver and kidney) from the first sacrifice (3 month exposure) of the two year chronic rat toxicity study have been completed.

Customer and Plant Support Analysis

The following PCB analyses were carried out duringtthe month for pollution abatement activities.

No. of Samples	Sample Source	Sample Description	Status of Analysis
1	NCR (Dayton)	Plant effluent composite	Completed
3	Ameripol	Plant effluent composite	Completed
2	J.F. Queeny	Plant effluent composite (February & March)	Completed
1	Ragansky Feed Co.	R-83	Completed
1	C.F. Mueller Co.	Vermicelli	Completed
14	R.A. Kimerle/CRD	Coagulated Anniston Plant effluents	Completed
2	Goodyear - Point Pleasant, W.Va.	Plant effluents	Completed
1	Kimberly Clark (Moraine Mill)	Plant effluents	Completed
25	Central Soya	Products associated with commercial chicken raising	8/25 Completed
8	Westinghouse - Aquatechnics	Treated Plant effluents	Scheduled for Completion 4-30-71

Polychlorinated Terphenyls (PCT)

Hexane extraction of effluent water spiked with Aroclor 5460 demonstrated that it is not a suitable solvent for quantitative extraction of PCT's from plant effluent samples. The recovery (50 ±1%) on duplicate samples was reproducible but much too low to be used for isolation of PCT's from water. Quantitative extractions of PCT's were obtained with either benzene or dichloromethane. The extraction recoveries (99.0%) using dichloromethane were calculated from ultraviolet absorption data. For benzene, total organic chlorine was determined using a sodium biphenyl-silver nitrate titration procedure.

Customer and Plant Support Analysis

No. of Samples	Sample Source	Sample Description	Status of Analysis
25	Central Soya	Products associated with commercial chicken production	8/25 Completed Holding
8	Westinghouse Aquatechnics	Treated Plant Effluents	Completed
1	Woodson-Tenent Laboratories	Chicken Feed	Completed
2	~~NCR (Dayton)~~	24 Hour Storm Sewer Composite Reclaimed Oil	Scheduled for Completion 6/4/71
2	J. F. Queeny	Plant effluent Composite (April and May)	Scheduled for Completion 6/4/71
1	Ohio Dept. of Agriculture, Div. of Foods, Dairies & Drugs	No. 42379	In Transit
3-4	Inter-County Farmers Coop. Assn., Inc. Woodridge, N.Y.	Finished Chicken Feed	In Transit
1	Rozansky Feed Co.	Chicken Feed R110	Scheduled for Completion 6/9/71

CAPACITOR DIELECTRICS

"Trichlorodiphenylether" was found to be a mixture of mono, di, tri, and tetrachlorodiphenylethers. Tolyxylylsulfone was found to be a mixture of various ortho, meta, para isomers with 0.3% chlorobenzene. The gas chromatography/mass spectrometry analysis of these compounds was carried out to assist in the proper design of biodegradation experiments.

MONOISOPROPYL BIPHENYL (MIPB)

A series of Japanese fluids (Kureha) competitive to MIPB as dye solvents for carbonless "carbon paper" were found to consist primarily of isopropyl naphthalenes and isopropyl tetrahydronaphthalenes. The identification made by gas chromatography/mass spectrometry and NMR will help us counter the world wide threat posed by these new products.

June 1971

SPECIALTY PRODUCTS

AROCLOR ENVIRONMENTAL PROGRAM

Customer and Plant Support Analysis

No. of Samples	Sample Source	Sample Description	Status of Analysis
25	Central Soya	Products associated with commercial chicken production	8/25 Completed (Holding)
4	NCR Dayton	24 Hr. Storm Sewer Composite Samples - 5/19 - 5/20	Completed
		6/2 - 6/3	Completed
		6/16 - 6/17	Scheduled for Completion 7/9/71
		Reclaimed Red Paraffin Oil	Completed
2	J.F. Queeny	Plant Effluent Composites April	Completed
		May	Completed
		June	Scheduled for Completion 7/9/71
1	Ohio Dept. of Agri., Div. of Foods, Dairies, & Drugs	No. 42379	Scheduled for Completion 7/9/71
4	Inter-County Farmer's Coop. Assn, Inc. Woodridge, N.Y.	Chicken Feed	Completed
1	Rozansky Feed Co.	R110	Completed
2	Frigidaire Div. GM Corp., Dayton, Ohio	Plant effluents	Completed
1	GM Central Foundry Bedford, Ind.	Plant effluent	Completed
5	Monsanto Ag. Div.-Nitro Plt	Santoquin	Completed
1	WGK Dept. 246 W. Krule	Sewer Sample from 1105 run	Completed
2	Kimberly-Clark Morane Mill	Plant effluent	Completed
7	WGK Plant	Sewer effluents (exchange samples)	Completed

MONS 059812

SPECIALTY PRODUCTS

AROCLOR ENVIRONMENTAL PROGRAM

Customer and Plant Support Analysis

No. of Samples	Sample Source	Sample Description	Status of Analysis
25	Central Soya	Products associated with commercial chicken production	8/25 Completed (Holding)
3	NCR, Dayton	24 Hour Storm Sewer Composite Samples 6/16 - 6/17 (1) 7/15 - 7/16 (2)	 Completed Scheduled 8/6/71
3	J. F. Queeny	Plant effluent Composites June	Scheduled 8/6/71
1	Ohio Dept. of Agri. Div. of Foods, Dairies & Drugs	No. 42379	Completed
8	Westinghouse Elec. Corp., S. Boston, Va.	Water effluents, oil, & mud associated with plant oil separator	Scheduled 8/10/71
3	Frigidaire Div., GM Corp., Dayton,O.	Plant effluents - June	Scheduled 8/6/71
40	Monsanto - Ag. Div.	- - - - - - -	23/40 Completed Scheduled 8/3/71
8	Monsanto - Org.Res.	Biphenyl scrubbing studies	Completed

BIO-TEST TISSUE ANALYSIS

Analyses of all tissues (160 samples) from the 3, 12, and 24 month sacrifices of the Industrial Bio-Test Laboratories' Two Year Chronic Oral Toxicity Study of Aroclor in Albino Rats #B7298 have been completed. This completes the PCB support analysis for this study as the proposed 30 and 60 day recovery tissues were not retained by Bio-Test. A special study report will be prepared and issued.

All tissues (24 month sacrifice and 30 and 60 day recovery sacrifices) from the Industrial Bio-Test Laboratories Two Year Oral Toxicity Study of Aroclor in Beagle Dogs #C7299 have been received. Approximately 17% of these tissues have been prepared for EC/GC analysis.

Aug 1971

SPECIALTY PRODUCTS

Customer and Plant Support Analyses

No. of Samples	Sample Source	Sample Description	Status of Analysis
25	Central Soya	Products associated with commercial chicken production	8/25 Completed (Holding)
1	NCR	24-Hr. storm sewer composite 7/15 - 7/16	Completed
5	JFQ	Plant effluent composites; June and July	Completed
8	Westinghouse Elec. Corp., S. Boston, Va.	Water effluents, oil and mud associated with plant oil separator	7/8 Completed Scheduled for Completion 9/5/71
5	Frigidaire Div., GM Corp., Dayton, Ohio	Plant effluents, June	3/5 Completed Scheduled 9/5/71
42	Monsanto - Ag Div. Nitro	Plant products	9/42 Completed Scheduled 9/5/71
1	JFQ	PNP	Completed
1	WGK	HCl	Completed
9	Monsanto - Ag Div. Nitro	Competitive Products	Scheduled for Completion by 9/30/71
2	Pensacola Plant	Fish	Scheduled for Completion by 9/30/71

(Tucker, Litschgi, Mees)

α-METHYL STYRENE LINEAR DIMER

This material was analyzed for ppm sulfur via the Granatelli olefin procedure for correlation with data obtained at the Luling plant. Sulfur is a known catalyst poison in the hydrogenation procedure used in this process. Levels found were in the low normal range (2 and 9 ppm). (Hicks)

PYDRAUL 312

An isomer of dichloroterphenyl has been identified as the cause of quality control problems in Pydraul blends containing Aroclor 5432. This material, obtained as acetone insolubles from recent plant batches of Aroclor 5432, was identified by gas chromatography and solids probe mass spectrometry. (Mieure)

Sept 1971

SPECIALTY PRODUCTS

Customer and Plant Support Analysis

The status of analytical work during the month is as shown below. Details are reported in the monthly progress report.

No. of Samples	Sample Source	Sample Description	Status of Analysis
25	Central Soya	Products associated with commercial chicken production	9/25 Completed (Holding)
3	JFQ	Plant effluent Composites; August	Completed
2	Frigidaire Div., GM Corp., Dayton, Ohio	Plant effluents, July	Completed
31	Monsanto - Ag Div. Nitro	Plant Products	6/31 Completed (Holding)
2	Pensacola Plant	Fish	Completed
10	Ralston-Purina St. Louis, Mo.	Meat, Soybean, & Fish Meal	Completed
10	St. Regis Paper Co., Dallas, Texas	Polyethylene & Paperboard	Holding
1	Broiler Council Round Robin	Chicken Fat	Completed
8	Inter-Plant Round Robin (WGK, Anniston & Applied Sciences)	Water Samples	Completed

(Tucker, Mees, Litschgi)

BIO-TEST TISSUE ANALYSIS

Analysis of 180 tissue samples from the "Two Year Chronic Oral Toxicity Study of Aroclor 1242, Aroclor 1254 and Aroclor 1260 in Beagle Dogs" has been initiated. The complete chemical/alumina column clean-up procedure will be necessary with these samples since several interferences to the EC/GC quantitation are present. The target date for completion of the analysis is 10-29-71. (Mees, Parker, Cowell and Litschgi)

Jul 1971

SPECIALTY PRODUCTS

BIODEGRADATION TESTING

Aroclor 5432 and Aroclor 5460

Development of analytical methodology for the determination of polychlorinated terphenyls (PCT's) in laboratory and environmental materials is nearing completion. The method involves isolation of the PCT's by extraction with chloroform followed by liquid/solid chromatographic clean up on alumina. The amount and type of PCT's present are determined by high temperature electron capture gas chromatography. A 2m X 4mm all glass column with 2.5% SE-52 on Gas Chrom Q (80/100 mesh) is employed at column temperatures in the range of 220°C - 260°C. Overall recoveries from water range from 80-100% with absolute detection limits of 5-10 nanograms. (Daust, Saeger)

Trichlorodiphenyl Ether and Tolyl Xylyl Sulfone

TCAS disappearance rate data at a feed rate of 5 mg per 48-hour cycle has been obtained for trichlorodiphenyl ether and tolyl xylyl sulfone. The mean disappearance rates (95% confidence limits) obtained were [illegible] for trichlorodiphenyl ether and 11.8 ±7.9% for tolyl xylyl sulfone. On the basis of these data, both materials would be classified as refractory. Limited testing will be carried out to determine if any significant change occurs with time. (Saeger, Yepez)

BIO-TEST TISSUE ANALYSIS

Analysis of tissues from the two-year study with beagle dogs is nearly complete. Analysis of tissues from the toxicity and reproduction study with chickens has begun. An inventory of the St. Louis freezers indicates that we have not received all of the tissues. An early November meeting with Bio-Test to resolve our sample inventory problems has been set up. (Mees, Litschgi, Cowell, Parker)

A sequential testing plan aimed at minimizing the number of analyses required on the chicken tissue and beagle dogs was proposed. Reductions of up to 75% of the required testing are possible. (Hinchen)

Customer and Support PCB Analysis

The status of analytical work during the month is as shown below. Details are reported in the monthly progress report.

No. Samples	Sample Source	Sample Description	Status of Analysis
25	Central Soya	Products associated with commercial chicken production	Dropped
5	JFQ	Plant effluent composites (August-September)	Completed
9	Monsanto - Ag Div. - Nitro	Plants Products	Completed
12	St. Regis Paper Company	Film and Board Extracts	11-19-71
2	Bio-Test Laboratories	Chicken Chow	Completed

(Cont'd on next page)