# PROGRESS REPORT

ORGANIC CHEMICALS DIVISION

TECHNOLOGY DEPARTMENT

| REPORTED BY | FOR | DETAILS | REPORT NO | DATE |
|---|---|---|---|---|
| W. M. Mees<br>W. J. Litschgi<br>E. S. Tucker | October 1971 | YES | 3 | 10-29-71 |

| LOCATION | JOB NOS |
|---|---|
| St. Louis - South 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 or legal and regulatory purposes.

## SUMMARY

PCB's typical of our Aroclor products have been found at levels below and above recommended guidelines in:

- Coated paper and paper coatings
- Machine oils and plant effluents from one customer manufacturing site
- Fourteen Monsanto Industrial Chemical Company products from four manufacturing sites
- Chicken Chow used as feed by Industrial Bio-Test Laboratories
- Sewer effluents from one Monsanto Plant site
- Tissue turkey extracts from the U.S.D.A.
- Two competitive products.

PLAINTIFF'S EXHIBIT
P-1142

PCT's typical of Aroclor 5432 were found in:

- Plant effluent samples from one customer manufacturing site.

A table showing the sample sources, types, and the level and type of Aroclor found as well as the support objective is presented in the details section.

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

- An on site methodology review for F. B. Chamberlin (Director of Research and Development) and R. M. Wilkinson (Senior Project Manager) representatives of the Alton Packaging Division, Alton Box Board Co.
- PCB Seminar presentation on analytical methodology for the Research Council of Poultry and Egg Institute of America in Chicago, Ill.
- PCB problem and progress review for T. Katayama (Marketing Manager) and S. Uneno (Product Director) from Mitsubishi Monsanto Chemical Co.
- Preliminary write up of methods for the determination of PCB's in Santoquin and CaMHA.
- Evaluation of PCB perchlorination for quantitation of altered PCB residues.
- Analysis of Monsanto and Competitor Aroclor products for polychlorinated dibenzofurans.
- General internal and external analytical consultation.
- Distribution of Aroclor methodology and reference standards.

FUTURE PLANS

PCB Analysis

1. Three to four J. F. Queeny monthly composite sewer effluent samples.
2. Forty-six machine oil and plant effluent samples from Saginaw Grey Iron Casting Plant, Chevrolet Motor Division.
3. Two oil samples skimmed off trade waste settling tanks from Buick Motor Div., General Motors Corp., Flint, Mich. (2 samples).
4. Four coated paper samples from Champion Papers, Hamilton Mill, Hamilton, Ohio (4 samples).
5. Four fluid samples from an imported VW automobile.
6. Five ACL-59 samples from the W.G. Krummrich Plant.
7. Three raw material samples from P.P.G. Industries.
8. Twelve film and paper extracts from St. Regis Paper Co.
9. One fish from a stream associated with a General Electric Plant.
10. Five intra-lab method check samples from a General Electric Plant Laboratory.

PCT Analysis

1. Ten plant effluent samples from Monsanto's Anniston plant.
2. Saginaw Foundries, Chev. Motor Div. plant effluent samples.

W. J. Litschgi

E. S. Tucker

TABLE I

CUSTOMER SERVICE

| Sample Source | Sample Matrix | No. of Samples | Level Found | Associated Aroclor | Support Objective |
|---|---|---|---|---|---|
| General Motors Corp., Buick Div. | Machine oils | 11 | Per Cent | A-1242 | Pollution Control |
| | Drainage streams around plant | 2 | PPB | A-1242 | Pollution Control |
| Champion Paper Company | Paper (writing) | 3 | PPM<br>Per Cent | A-1242<br>A-5432 | Pollution Control |
| | Paper Coatings (Micron II) | 3 | PPM<br>Per Cent | A-1242<br>A-5432 | |

MANUFACTURING SUPPORT

| | | | | | |
|---|---|---|---|---|---|
| J. F. Queeny | Sewer effluent composites | 5 | PPB | A-1242 | Pollution Control |
| | HCl | 1 | PPB | A-1242 | |
| J. F. Queeny, W. G. Krummrich, Del. River, and Everett | Finished Products | 28 | N.D.* to PPB | A-1242 | |

COMPETITIVE PRODUCTS

| | | | | | |
|---|---|---|---|---|---|
| Ag. Division, Nitro | Santoquin | 6 | PPB | A-1242 | Determination of level and sources of contamination of competitive animal feed additives |
| | CaMHA | 3 | PPB | A-1242 | |

*None Detected

(Cont'd on next page)

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RESEARCH AND DEVELOPMENT

| Sample Source | Sample Matrix | No. of Samples | Level Found | Associated Aroclor | Support Objective |
|---|---|---|---|---|---|
| Inter-Laboratory Round Robin (WGK, JFQ and Anniston) | Distilled water spiked with known quantitites of Aroclor | 7 | PPB to PPM | A-1242<br>A-1254<br>A-1260 | Insure the maintenance of precision and accuracy in Monsanto labs analyzing for PCB's |
| Bio-Test Laboratories, Chicago, Ill. | Ralston-Purina Chicken Chow | 2 | PPB | A-1242 | Determination of PCB background in feed used in the toxicity studies being performed at Bio-Test |
| USDA | Turkey Extract | 1 | - | A-1248 | Determination of type of PCB and how the PCB contamination occurred |

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DETAILS (Cont'd)

Customer Service

General Motors Corporation - Buick Division

Due to increased pressure from the Michigan State Regulatory Agency concerning PCB releases into the environment, Monsanto was requested to analyze samples of Buick's plant effluent and nearby drainage stream. At the same time, samples of oil used in their presses and machines were submitted to determine residual PCB levels remaining after Buick switched from Pydraul 312 to Pydraul 312A.

| Sample Description | Level Found | Aroclor |
|---|---|---|
| Water from trade waste prior to Hickory stream | 8 PPB | A-1242 |
| | 15 PPB | A-5432 |
| Water from Hickory rain stream prior to river | 13 PPB | A-1242 |
| | <5 PPB | A-5432 |
| Machine #1 | 14.8% | A-1242 |
| Machine #2 | 2.5% | A-1242 |
| Machine #3 | 13.8% | A-1242 |
| Machine #4 | 8.9% | A-1242 |
| Machine #5 | 3.9% | A-1242 |
| Machine #8 | 4.4% | A-1242 |
| Machine #9 | 24.3% | A-1242 |
| Trim Press #1 | 11.0% | A-1242 |
| Trim Press #2 | 0.9% | A-1242 |
| Trim Press #8 | 0.8% | A-1242 |
| Trim Press #9 | 16.8% | A-1242 |

Champion Paper Company

Coated paper and the used coating (Micron II) samples submitted by Champion Paper Company were analyzed to determine the level of residual PCB's in the PCT's used in the paper coating fluid.

| Sample Description | Level Found | Aroclor |
|---|---|---|
| Paper - 3866F | 203 PPM | A-1242 |
| | 0.29% | A-5432 |
| Paper - 3878F | 140 PPM | A-1242 |
| | 0.25% | A-5432 |
| Paper - 3758M | 217 PPM | A-1242 |
| | 0.28% | A-5432 |
| Micron II - Sample #1 | 242 PPM | A-1242 |
| | 0.48% | A-5432 |
| Micron II - Sample #2 | 331 PPM | A-1242 |
| | 0.75% | A-5432 |
| Micron II - Sample #3 | 211 PPM | A-1242 |
| | 0.56% | A-5432 |

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DETAILS (Cont'd)

Manufacturing Support

J. F. Queeny Plant

| Sample Description | Level Found | Aroclor |
|---|---|---|
| Clean Acid Sewer (August) | 19 PPB | A-1242 |
| DA Sewer (August) | 29 PPB | A-1242 |
| MSD Gatewell (August) | 77 PPB | A-1242 |
| Clean Acid Sewer (September) | 3 PPB | A-1242 |
| MSD Gatewell (September) | 31 PPB | A-1242 |

The DA sewer (August) was supposed to be the Clean Acid Sewer (August) spiked with ~20 PPB A-1242. As can be seen, our results reflect only a 50% recovery. Discussion with the J. F. Queeny pollution group is planned in an effort to resolve this problem.

Monsanto Industrial Chemicals Company

The following Monsanto Industrial Chemicals Company products have been analyzed for PCB's to determine if any of them were significantly contaminated with PCB's.

| Sample Description | Level Found | Aroclor |
|---|---|---|
| Aspirin QA-1187 9-13-71 | N.D. | |
| Aspirin QA-1213 10-4-71 | N.D. | |
| Maleic Anhydride QA-1295 9-16-71 | N.D. | |
| Maleic Anhydride QA-1333 10-6-71 | N.D. | |
| Sodium Saccharin QA-153 8-31-71 | N.D. | |
| Sodium Saccharin QA-168 9-11-71 | N.D. | |
| Saccharin QA-209 5-5-71 | 0.03 PPM | A-1242 |
| Saccharin QA-1012 6-23-71 | 0.02 PPM | A-1242 |
| Vanillin USP Ba#1025 | 0.20 PPM | A-1242 |
| Vanillin USP Ba#1027 | 0.18 PPM | A-1242 |
| Tall Oil Rosin Rec'd 1-25-71 | N.D. | |
| Tall Oil Rosin Rec'd 5-25-71 | N.D. | |
| CDP-OR175000 #4 | 0.17 PPM | A-1242 |
| CDP-04175000 #3 KA-79 | 0.12 PPM | A-1242 |
| S-334F EUT #A-134 10-4-71 | 0.26 PPM | A-1242 |
| S-334F EUT #A-135 10-4-71 | 0.21 PPM | A-1242 |
| S-711 "A" Line 10-6-71 | 0.27 PPM | A-1242 |
| S-711 "B" Line 10-6-71 | 0.36 PPM | A-1242 |
| S-160 DA-614 | 0.36 PPM | A-1242 |
| S-160 DA-803 D.R. | 0.35 PPM | A-1242 |
| HB-40 #1 Stg Tk 10-7-71 | N.D. | |
| HB-40 #3 Stg Tk 10-6-71 AA-275C | N.D. | |
| Biphenyl AA-194 9-27-71 | 0.67 PPM | A-1242 |
| Biphenyl #2 Stg Tk 10-6-71 | 0.15 PPM | A-1242 |
| Santowax R 10-6-71 | N.D. | |
| Santowax R Rec'd 9186 10-7-71 | N.D. | |

DETAILS (Cont'd)

Competitive Products

| Sample Origin | Sample Description | Level Found | Aroclor |
|---|---|---|---|
| Thompson-Hayward Chemical Co. | Ethoxyquin (66.7%) | 749 PPB | A-1242 |
| HIC 7-21-71 | Neoquin | 184 PPB | A-1242 |
| Traveler's Rest | Santoquin Emulsion | 151 PPB | A-1242 |
| Traveler's Rest | Santoquin Mix 6 | 570 PPB | A-1242 |
| Rexolin Chemical | Ethoxyquin (100%) | 155 PPB | A-1242 |
| SNS 8-30-71 | Abiquin | 133 PPB | A-1242 |

Our detection limit based upon observed background and sample was ∿50 PPB.

| | | | |
|---|---|---|---|
| Roche Ag Lot 2-4-71 Mfgd by Degussa | DL-Methionine | 2.5 PPB | A-1242 |
| Upland, Calif. A3NG, SNS | DL-Methionine | 21.4 PPB | A-1242 |
| Thompson-Hayward Chemical Co. | DL-Methionine | 45 PPB | A-1242 |

These samples were analyzed to determine if products in competition with Monsanto's Santoquin and CaMHA were also contaminated with PCB's. In all cases, they were found to contain an equal or greater level than the corresponding Monsanto products.

Methods for the determination of polychlorinated biphenyls in Santoquin (Organic Research Method 71-26) and CaMHA (Organic Research Method 71-27) have been written and will be sent to the Nitro plant where they will be used to monitor their Santoquin and CaMHA production.

Research and Development

Analysis of Chicken Feed from Bio-Test

The chicken feed used in the Bio-Test toxicity studies was purchased from the Ralston-Purina Company. In the past months, attention has focused upon the fact that Ralston's feed has, in some instances, become contaminated with PCB's. For this reason, two samples of the feed used in the chicken feeding studies were analyzed for PCB's.

| Sample Origin | Sample Description | Level Found | Aroclor |
|---|---|---|---|
| WCRF-1 | Chicken Feed | 240 PPB | A-1242 |
| WCRF-II | Chicken Feed | 263 PPB | A-1242 |

USDA Turkey Extracts

A USDA extract of a PCB contaminated (Swift) turkey tissue sample was submitted to our laboratory to see if we could establish what original Aroclor product caused the contamination and possibly the type of exposure. Using EC/GC and GC/Mass, we were able to determine that (1) PCB's were really present, (2) Aroclor 1248 was most probably the product involved, and (3) the birds either received a large single dose early in their lives or more probably a lower dose over an extended period of time.

PCT Methodology Status

A method has been developed and presently is being written (Analytical Method #71-37) for analysis of water and SCAS sludge samples for PCT's. Recovery data obtained from samples spiked with A-5432 and 5460 were essentially quantitative at a concentration level of >2 PPM.

This method is currently being used to analyze samples from the biodegradation studies and is at the same time being adapted for use on customer service and tissue samples.

Inter-Plant Round Robin

In order to establish the accuracy and precision of the PCB analysis being carried out in Monsanto laboratories, duplicate sets of spiked water samples have been analyzed by Anniston, WGK and Applied Sciences. The results were as follows:

| | PPM PCB (Spiked) | | | Applied Sciences | | | WGK | | |
|---|---|---|---|---|---|---|---|---|---|
| | A-1242 | A-1254 | A-1260 | A-1242 | A-1254 | A-1260 | A-1242 | A-1254 | A-1260 |
| 1W | - | - | - | - | - | - | - | - | - |
| 2W | 0.57 | 1.04 | - | 0.74 | 0.92 | - | 1.04 | 1.05 | - |
| 3W | 0.57 | 0.10 | - | 0.86 | 0.07 | - | 0.57 | 0.15 | - |
| 4W | 5.68 | 1.04 | 1.27 | 1.07 | | | 6.70 | - | 2.84 |
| 5W | 5.68 | 0.52 | - | 5.25 | 0.52 | - | 6.55 | 1.20 | - |
| 6W | - | 0.10 | - | - | 0.06 | - | - | 0.07 | - |
| 7W* | | | | 0.92 | 0.06 | - | - | - | - |
| 8W* | | | | | | | 8.80 | - | 4.16 |

| | Anniston | | |
|---|---|---|---|
| | A-1242 | A-1254 | A-1260 |
| 1W | - | - | - |
| 2W | 0.78 | 0.75 | - |
| 3W | 0.45 | - | - |
| 4W | 5.4 | 2.95 | 2.18 |
| 5W | 9.9 | - | - |
| 6W | - | 0.03 | - |
| 7W* | 0.4 | 0.2 | - |
| 8W* | 8.4 | - | - |

*Applied Sciences
7W = 3W

WGK
7W = 1W
8W = 4W

Anniston
7W = 2W
8W = 5W

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The data have been submitted for statistical analyses.

DETAILS (Cont'd)

National Broiler Council Intra-Laboratory PCB Methodology Check

Ten sets of soybean oil and chicken fat were prepared by Monsanto and distributed 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.

Listed below are the labs responding, however the individual laboratories were not identified.

| | Results PPM | | | | | |
|---|---|---|---|---|---|---|
| | 1S | 2S | 3S | 4C | 5C | 6C |
| Spike Level | 0 | 5.0 | 2.5 | 5.0 | 5.0 | 2.0 |
| Lab A | 0.25 | 5.31 | 2.56 | 4.00 | 5.06 | 3.86 |
| Lab B | 0.61 | 3.7 | 3.0 | 1.8 | 4.2 | 2.1 |
| Lab C (Applied Sciences) | 0.78 | 5.78 | 3.64 | 4.13 | 4.08 | 3.26 |
| Lab D #1 | 0.10 | 4.17 | 1.81 | 2.05 | 2.05 | 1.53 |
| Lab D #2 | 0.10 | 3.58 | 1.83 | 2.18 | 2.09 | 1.55 |
| Lab E | 0.73 | 4.40 | 2.24 | 2.42 | 2.68 | 2.14 |
| Lab F | 0.32 | 5.10 | 2.91 | 2.62 | 2.66 | 2.22 |
| Lab G | <0.10 | 2.9 | 2.0 | 3.0 | 3.6 | 2.9 |
| Lab H | 1.10 | 1.5 | <0.6 | <0.6 | 5.5 | 5.0 |
| Lab I | | | No Report | Received | | |
| Lab J | | | No Report | Received | | |
| WGK | | | No Report | Received | | |
| Anniston | 0.38 | 1.54 | 0.61 | 0.57 | 0.14 | - |

The results of this inter and intra sample exchange will be submitted for statistical analysis as soon as all laboratories have reported.

Chlorodibenzofurans in PCB's and PCT's

Chlorodibenzofurans Found

| Sample Description | <4Cl/Mol. | 4Cl/Mol. | 5Cl/Mol. |
|---|---|---|---|
| Prodelec Phenoclor DP6 | ND(<2 PPM) | Detected | Detected(15-30PPM |
| Prodelec Phenoclor DP6 | ND(<2 PPM) | Detected | Detected(15-30PPM |
| Bayer Clophen A60,Lot 931134 | ND(<2 PPM) | Detected | Detected |
| Kaneclor KC-300 Shizuki, July 1970 (Composition similar to A-1242) | ND(<2 PPM) | ND(<2 PPM) | ND(<2 PPM) |
| Monsanto A-1242,Lot AK-255 | ND(<2 PPM) | ND(<2 PPM) | ND(<2 PPM) |
| Monsanto A-1254,Lot AK-38 | ND(<2 PPM) | ND(<2 PPM) | ND(<2 PPM) |
| Monsanto A-1260,Lot AK-3 | ND(<2 PPM) | ND(<2 PPM) | ND(<2 PPM) |
| Monsanto MCS-1016,Drum B | ND(<2 PPM) | ND(<2 PPM) | ND(<2 PPM) |
| Monsanto MCS-1016,KA-708 | ND(<2 PPM) | ND(<2 PPM) | ND(<2 PPM) |
| Monsanto A-5442,Lot AI-9 | ND(<5 PPM) | ND(<5 PPM) | ND(<5 PPM) |