COPY

# EXHIBITS

TRANSWESTERN V. MONSANTO
JUNE 11-12, 1992
VOLUMES I & II

Robert Ellis Keller

**FIRST CLASS MAIL**

STLCOPCB4027091

SEP 5 - RECD

MONSANTO CHEMICAL COMPANY

FROM (LOCATION): General Offices

DATE: September 1, 1961

SUBJECT: Aroclor 1242

REFERENCE:

cc: R. S. Gordon
L. J. Machlin
W. J. McCarville
df: University of Missouri

TO: Elmer P. Wheeler

You have asked for a status report on Aroclor 1242 with regard to chick edema. O'Dell is not planning to do any more work beyond that that he has already told us about. However, the word has gotten around in toxicology circles and more studies are under way.

Samples of Aroclor 1242 have been submitted to Proctor and Gamble, to MIT, and to the National Dairy Products Corporation, for evaluation in chick edema studies. The Proctor and Gamble people spent a day with us about six weeks ago and gave us a detailed report on their work with chick edema factor. It appears that Aroclor 1242 does indeed produce the same symptoms in chickens as does the chick edema factor that has occurred in commercial fats. However, the Proctor and Gamble people are convinced that the degree of toxicity is so much less for Aroclor 1242 than for the naturally occurring material, that the two are not identical. Furthermore, their physical-chemical data available on the chick edema factor indicates that it is structurally different from Aroclor 1242. I believe that we are pretty much out of the woods on this problem because of this marked difference in degree of toxicity.

Most researchers in this field realize that many compounds can produce the chick edema factor and I believe that the problem will be solved within the next six months, and that Aroclor 1242 will not be involved.

Kenneth H. Maddy

KHM:cs

9/19/61

Talked with Maddy and he is sure it won't bother us.

R.E.K.

DEPOSITION EXHIBIT
MFS 278
6-11-92

TRAN 016778

STLCOPCB4027092

MONSANTO INDUSTRIAL CHEMICALS COMPANY
Technology Planning & Evaluation
June 1, 1972

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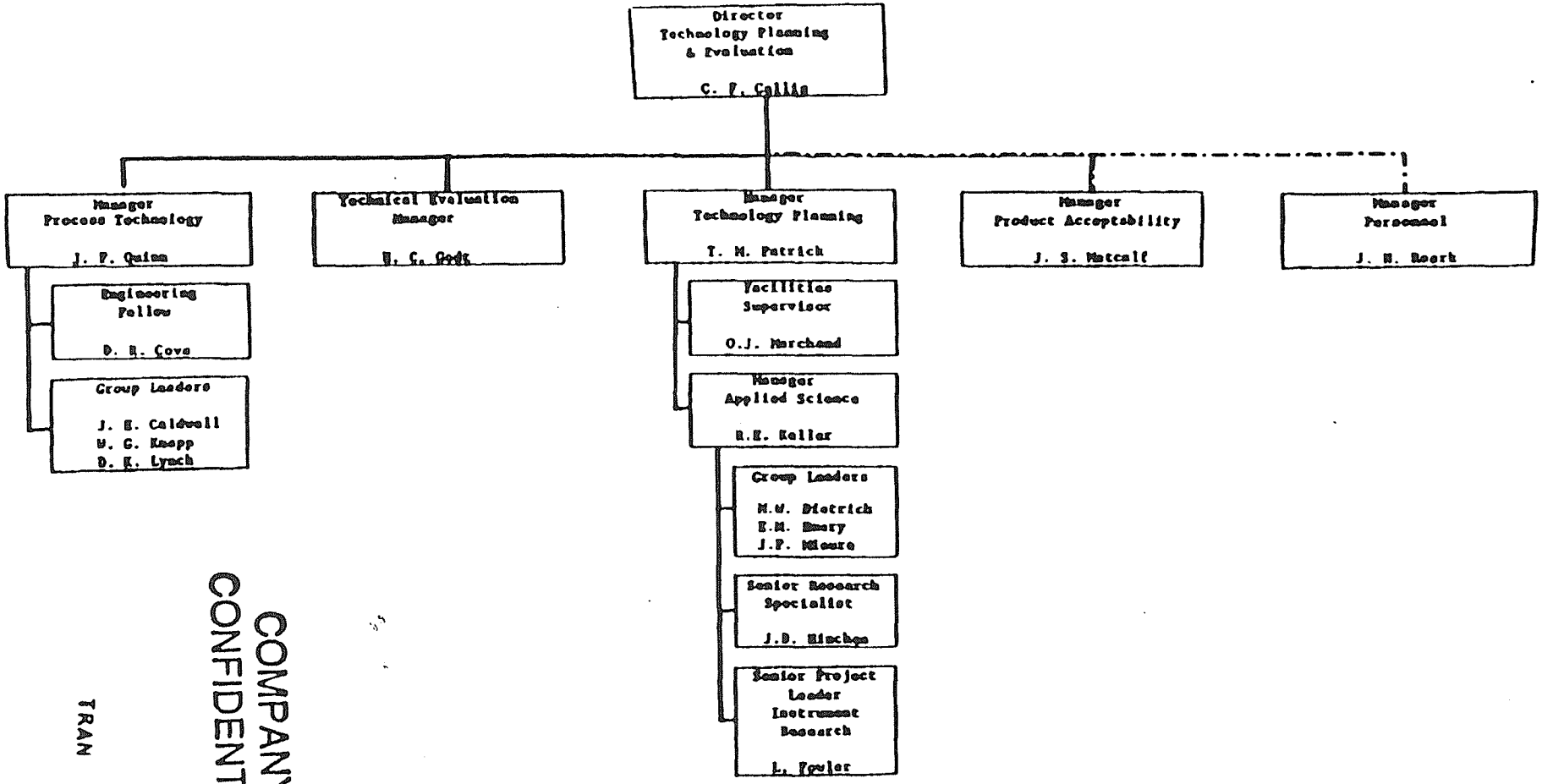
COMPANY
CONFIDENTIAL

DEPOSITION
EXHIBIT
PIFs 279
6-11-92 [illegible]

TRAN 085902



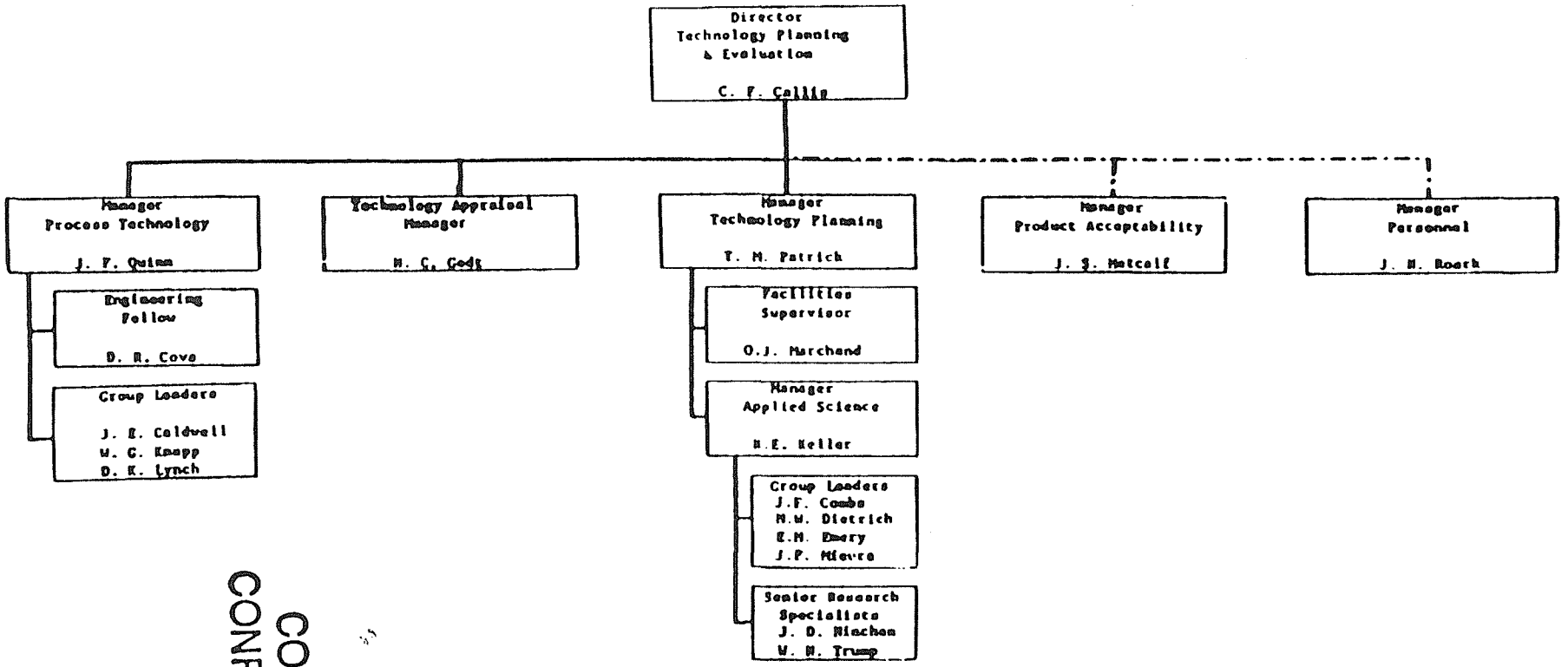

COMPANY CONFIDENTIAL

TRAN 085919

MONSANTO INDUSTRIAL CHEMICALS COMPANY
Technology Planning and Evaluation
October 1, 1975



STLCOPCB4027094




COMPANY
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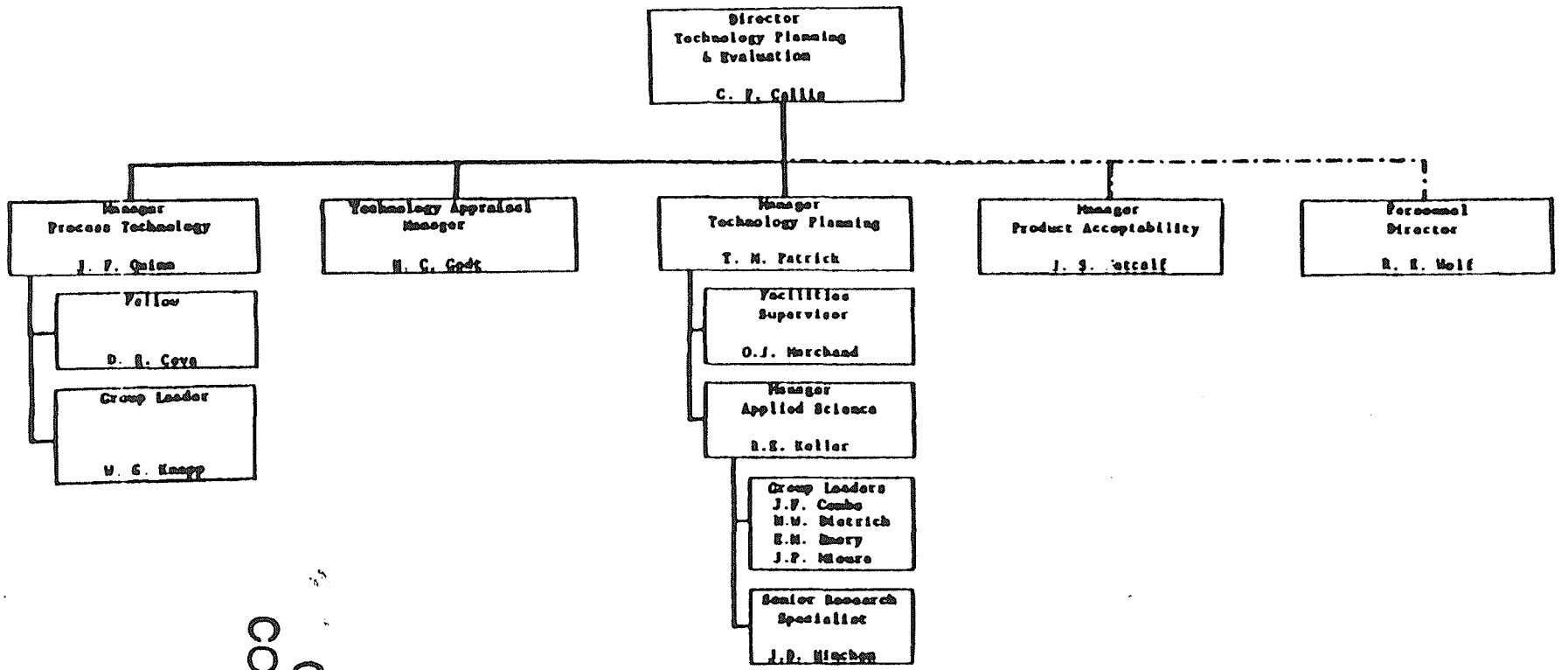
TRAN 085926

MONSANTO INDUSTRIAL CHEMICALS COMPANY
Technology Planning and Evaluation
June 1, 1976



STLCOPCB4027095




COMPANY CONFIDENTIAL

TRAN 085930

MONSANTO INDUSTRIAL CHEMICALS COMPANY
Technology Planning and Evaluation
April 1, 1977



STLCOPCB4027096

INSTITUTE OF ANALYTICAL CHEMISTRY
UNIVERSITY OF STOCKHOLM
Kemigrvägen 90, Stockholm 50, Sweden

Stockholm, December 29, 1966

Mr. Ford
Monsanto Chemical Co
800 Lindbergh Road
St Louis, Missouri 63177
USA

Dear Mr. Ford.

Last year I had the great pleasure of visiting you at the Monsanto Co in St.Louis to discuss analytical problems of the phenolic composition of TCP. We were later kindly supported with samples by Mr. J.S. Thomas and Mr. A.P. Uelner. Our analytical investigations are almost finished and we will send you a reprint of the report when they will be available.

This letter is to inform you that we recently have identified most of the unknown GC-peaks at residue analysis of pesticides in biological samples. By the aid of combined GC and mass spectrometry Mr. Jensen of this institute has found the unknowns to be polychlorinated biphenyls which among other companies are manufactured by Monsanto, trade name Aroclor.

Added to this letter you will find a preprint of the synopsis of our publication which will appear in Acta Chem. Scand. (Summer 1967). Two gas chromatograms are also added to show that the chlorinated biphenyls are enriched in ecological series. - In the sample of the sea eagle were found: Polychlorinated biphenyls 80 ppm; ordinary chlorinated pesticides 80 ppm and mercury (I) 40 ppm.

At present we have no knowledge of the biological significance of the presence of these compounds in living organisms. We have started a collaboration with the Dept. of Toxicology at the Carolin Institute but we should very much appreciate to work with your toxicologists over this problem.

We will be glad to give you any further informations of the results obtained in this laboratory.

Sincerely Yours

Gunnar Widmark

Gunnar Widmark

TRAN 055797

DEPOSITION EXHIBIT
PIES 280
6-11-82

STLCOPCB4027097

Toke

# CHLORINATED BIPHENYLS

## CHRONOLOGICAL EVENTS

11-22-66 - JENSEN LECTURE, WENNER-GREN CENTER MEETING, STOCK-

12-15-66 Report of New Chemical Hazard - New Scientist Dec. '66
(Jensens Work)
First published report about PCB

12-29-66 Widmark letter to Monsanto
Head of Institute of Analytical Chemistry
University of Stockholm, Sweden
Found unknowns to be polychlorinated biphenyls
made by Monsanto, et al - used GC/Mass.
Sea eagle - 80 ppm polychlorinated biphenyls
- 80 ppm pesticides
- 40 ppm mercury
No knowledge of biological significance in living organisms.
Collaboration started with Dept. of Toxicology/Carolin Institute.
Would like to work with Monsanto Toxicologists on problem

1-10-67 Press Release, LKB Instrm. Co.
(Jensens Work)

1-26-67 D. Wood letter to Buchanan
Visited Sweden
Confirmed chlorinated biphenyl report of Jensen

1-30-67 Editorial in Chemical Engineering
Covers Jensens work

2-13-67 Wilde letter to Kelly

2-21-67 Hardy letter to Kelly
of - Jensen may have jumped gun in blaming chlorinated biphenyls

2-21-67 Kelly letter to Wilde - No need to contact Swedish people

2-27-67 Widmark letter to Wilde
Reviews lecture at Baeyer in Germany

2-27-67 Kelly letter to

3-16-67 Wilde letter to Kelly - Widmark Letter

5-67 S.A.O.A.C. - Pub. - Widmark 50 1069 (1967)

10-21-67 Holmes, Simmons, Tatton - Nature 216 227 (1967)
Confirms work of Jensen

PRR 021889
SCM 037311

11-1-67 Hardy letter to at
Status of work at ?

11-2-67 Richardson letter to Hardy
Confirmed by GC/Mass. Aroclor 1254 in eggs from 100 ppm feeding studies - confirmed Govt. Labs.

DEPOSITION EXHIBIT
PHS 281
6-11-92 989

E419

STLCOPCB4027098

12-30-67 Holden, Marsden - Nature 216 1274 (1967)
PCB in seals
PCB appears in birds with aquatic environment - suggests river contamination.
Human in UK contain little PCB <0.1 ppm in fat.
Suggests pollution is of aquatic origin - if air more would be found in human fat.
Conen found & $LD_{50}$ - unlikely that residues of PCB constitute direct toxic hazard.

1-8-68 Heininger letter to Robson/Anagnostopoulos.
Birdsall, Wisconsin Alumni Res. Foundation (WARF)
Studies to be on shore birds
Funded by U.S. Dept. of Interior, Fish & Wildlife Service
Suggested that Monsanto-WARF work openly

1-16-68 Wilson letter to Gentlemen/Monsanto
Res. Chemist, U.S. Dept. of Interior
Fish & Wildlife Service
Investigation of chlorinated polyphenyls in avian and animal tissues

1-26-68 Lincer letter to Patterson, MCL
Res. Assistant, Syracuse Univ. Res. Corp., New York
Wanted polychlorinated biphenyl references & materials

2-8-68 Spencer letter to Richard
Consultant, Nat. Ag. Chemicals Assoc-
Interferences with chlorinated hydrocarbons

2-13-68 Baxter letter to Richard - Ref. to Syracuse

2-19-68 Richard letter to Baxter - Aroclor sa. status

2-23-68 Hardy letter to Richard

4-26-68 Paton letter to Kelly

5-3-68 Newell letter to Hunt
Chairman Dept. of Ag. & Environmental Toxicology, Stanford Res. Institute
Toxicological results/birds & fish/enzyme induction stimulated
Recommended tests in cooperation with Monsanto rather than seek govt. support
Med. Dept. recommended no action.

5-6-68 Kelly memo to Robson/Anagnostopoulos - Reference to 5-3-68

5-20-68 Richard to Davis and back

5-23-68 Richard to Davis and back

PRR 021890

SCM 037312

7-15-68 Glasgov, Chief Pesticides - requested info on Aroclors
Ref. Stds. Section
Dept. Health, Education & Welfare
Blickenstaff - USDA - quality control for formulations
Dykstra - USDI - Pautuxent, Maryland Labs - Aroclor sent

7-31-68 Paton Memo

8-23-68 Wheeler to Early, trip plan

9-5-68 Wheeler plan memo for visits

9-11-68 Wheeler memo to Richard - Biotest Protocols

10-9-68 Risebrough - Confidential pre-publication
PCB in fish & birds
Comments from Monsanto solicited

10-21-68 Wheeler to Richard - Risebrough paper

11-1-68 Risebrough to Conkin - Publication Review

11-6-68 Wheeler to Richard

11-13-68 Conkin to Risebrough - Comments on review of manuscript

11-14-68 Wheeler memo to Richard

11-27-68 Paton to Richard
Include Aroclor 5460

11-68 Gunther - Prof. of Entomology & Chemistry, Univ. of Calif.
School of Ag. Sciences
Dept. of Entomology
Citrus Res. Ctr & Ag. Expt. Sta. - Riverside

State USDA Research Area
Committee including Hawaii
DDT & chlorinated hydrocarbons

12-3-68 Gunther to Paton - Request for Aroclor Sas.

12-9-68 Wheeler to Richard - Calendra Minutes

12-9-68 Richard to Wheeler - Calendra Minutes

12-14-68 Risebrough - Nature 220 1098 (1968)

12-19-68 Richard to Tucker - DDT, et al

12-20-68 Wheeler to Calendra - Letter of authorization

12-23-68 Bryant to Harris - Rome Pollution Laboratory

12-30-68 Richard to Kuhn - Legal problems

12-30-68 Tucker to Richard - Chlorinated Naphthalenes

SCM 037313

PRR 021891

| | |
|---|---|
| 12-31-68 | Tucker ~~to Tucker~~ more - Aroclor at Anniston |
| 1-7-69 | Wheeler to Richard - Aroclor Wildlife |
| 1-9-69 | Wheeler to Richard - Meeting |
| 1-10-69 | Wheeler to Calendra - Feeding of Halowaxes |
| 1-16-69 | Wheeler to Tucker - Widmark Paper |
| 1-21-69 | Wheeler to Fancher - Proceed with chickens |
| 1-21-69 | Wheeler to Richard - Pesticide Structures |
| 1-23-69 | Hodges to Buckley - Aroclors in Plant Effluents |
| 1-24-69 | Paton to Keller, et al - Montar |
| 1-30-69 | Tucker to Wood - Update |
| 2-4-69 | Wood to Tucker - Reply |
| 2-7-69 | Tucker to Wheeler - Pesticide Sas. |
| 2-10-69 | Wheeler to Fancher - DDT, et al sas. |
| 2-24-69 | Perlman - San Francisco Chronicle |
| 2-25-69 | Tucker to Keplinger |
| 2-25-69 | Tucker to Wheeler - Widmark's Paper |
| 2-25-69 | Tucker to Wheeler/Richard - Anniston River Results |
| 2-26-69 | Notes on meeting - Burgen, et al |
| 2-26-69 | Tucker to Stark - Solubility |
| 2-28-69 | Rough Draft - Keller - Position Statement |
| 3-4-69 | Tucker to Richard - Combustion |
| 3-4-69 | Wheeler to Wood - Dept. of Fish & Game |
| 3-5-69 | Paton to Richard - DDT on Trial |

PRR 021892

SCM 037314

R. E. Keller
3/10/69

STLCOPCB4027101

St. Louis

February 21, 1967

G. R. Buchanan - C3UCH

R. E. Keller - QUEENY

Mr. Gene Wilde
GUILD

I talked to Dave Wood in Brussels this morning and I list below the salient features of our conversation:

1. He has 90% of the information asked for in my letter of February 10 and will have the rest obtained in the next day or so and will forward the bundle to me. It should arrive within a week.

2. His customers in Europe are seemingly less concerned than they were, especially since there has been no particular government activity and no increase in the newspaper articles. The customers would like some reassurance on the toxicity of Aroclor (I explained to Dave that there just was no information available on the action of nanograms of Aroclor in the human body over a lifetime). There is no toxicological work going on at present in Sweden and it appears there is some likelihood that it will not be able to obtain financing and might not be done. Everybody over there is 100% convinced that what Jensen and Widmark found was Aroclor.

3. There is no necessity at present for anyone from St. Louis to contact the Swedish people. In fact, it would be unwise at present.

I am to get in touch with Mr. Wood after I receive the bundle of information he is sending me. Unless something unusual happens in the United States, I would be of the opinion that we should do no further action until we have time to evaluate this information.

R. Emmet Kelly, M. D.

REK/ln

PLAINTIFF'S EXHIBIT
#121 CH 8-6-8

DEPOSITION EXHIBIT
Plf's 282
6-11-92

BIR 007699

STLCOPCB4027102

Monsanto

FROM (NAME & LOCATION): St. Louis - General Offices

DATE: February 15, 1968

SUBJECT: THERMINOL/SANTOTHERM Contamination of Cooking Oil - Smith Potato Crisps, Holland

REFERENCE:

cc D. S. Cameron - BRUSSELS
D. Wood - BRUSSELS
M. Schuitema - THE HAGUE
L. R. Stark - LSTAR

TO : R. E. Keller - QUEENY
Research Lab 211

Bob, I am sending to your attention under separate cover a sample of peanut oil used for cooking potato crisps at subject company in Holland. This particular sample is from a quantity of fluid that had been contaminated with SANTOTHERM FR-1 (Aroclor 1242). It is reported from our Brussels office that the contamination is approximately 100 parts per million.

Can you determine:

1) whether or not this contamination can be detected?

and . . .

2) whether this sample can be utilized in your present program for determining a feasible test to detect THERMINOL/SANTOTHERM contamination in cooking oils?

We are particularly interested in reporting to our Brussels office whether or not we are able to detect the SANTOTHERM contaminant in this cooking oil sample; therefore, we await an early reply.

Donald E. Roush

DER:lh

DEPOSITION EXHIBIT
PH's 283
6-11-92

COMPANY CONFIDENTIAL

TRAN 008410

St. Louis - General Offices

March 25, 1968

FF

Therminol/Santotherm Contamination of Cooking Oil - Smith Potato Crisps, Holland

D. S. Cameron - BRUSSELS
M. Schuitema - THE HAGUE
NTJ/RD/SS
T. Denton-Roberts - LONDON

D. Wood - BRUSSELS FROM:

Dave, I have affixed copies of two reports from Dr. Keller's group on subject cooking oil contamination. The latest memo dated March 7, 1968, indicates that there is less than 20 parts per million Therminol/Santotherm FR-1 in the used peanut oil. Also indicated is the need for more sensitive measurements, which will be made when the analytical equipment has been repaired.

Dr. Keller's group is cognizant of the problem we have with chlorinated biphenyl contamination in the food processing industry and have been doing a fine job with this very difficult analytical problem. I shall notify you of further developments in this area when the information is available.

Best regards.

Don Roush

DR:lh
Attach.

COMPANY
CONFIDENTIAL

TRAN 057807

DEPOSITION EXHIBIT
Plf's 284
6-11-92 989

STLCOPCB4027104

# Monsanto

Swedish

Monsanto Company
800 N. Lindbergh Boulevard
St. Louis, Missouri 63166
(314) Oxford 4-1000

April 7, 1969

AIRMAIL

Professor Gunnar Widmark
Institute of Analytical Chemistry
University of Stockholm
Roslagsvägen 90, Stockholm 50, Sweden

Dear Professor Widmark:

Dr. Keller is going to be in Great Britain later this month for other business and we would like to plan to jointly meet with you and your associates in Stockholm on May 5. We are making reservations to arrive in Stockholm on Sunday, May 4 and plan to be there until Thursday, May 8.

We would appreciate an opportunity during the period of Monday through Thursday of reviewing the data concerning polychlorinated biphenyls in tissues with you and others in the Institute of Analytical Chemistry as well as any scientists doing research at the Karolinska Institutet.

I hope that this short notice will not make it impossible for us to see you.

Sincerely yours,

Elmer P. Wheeler
Manager, Environmental Health
Medical Department

EPW:cjs

TRAN 059255

DEPOSITION EXHIBIT
PHS 285
6-11-92 89

STLCOPCB4027105

Monsanto

FROM (NAME & LOCATION) E. P. Wheeler - Medical Dept.

DATE April 16, 1969

SUBJECT POLYCHLORINATED BIPHENYLS IN THE ENVIRONMENT

REFERENCE

TO : R. E. Soden - Brussels

cc
J. W. Barrett - London
D. N. V. Hardy - London
R. A. Baxter - Ruabon
H. R. Newman - Newport
D. Wood - Brussels
J. Haggert - Brussels
W. R. Richard
R. E. Keller
R. E. Kelly

Perhaps you have heard that Bob Keller from Organic Research and I plan to visit a number of people in Great Britain, Sweden, the Netherlands and Germany concerning the recent identification of polychlorinated biphenyls in wildlife tissues in various parts of Europe and the United States. Your people in Europe were the original contacts in late 1966 and early 1967 with scientists in university and government laboratories who identified PCBs as interfering substances when determining chlorinated pesticide residues in fish, sea eagles, pine needles, etc.

During 1967 and 1968 there were brief comments in the U.S. technical and trade journals concerning the PCBs but not until February of this year did the story break in the public press (see enclosures).

Beginning in mid-summer of last year a considerable research effort has been underway in the Organic Division and in consulting laboratories to develop data to protect the sales and uses of our PCBs. It has been concluded that before committing even greater expenditures, we should determine what efforts are underway by those people who have been in contact with Monsanto requesting samples of our Aroclors, reporting findings and mentioning specific continuing analytical and toxicological research.

I have enclosed a list of the individuals Bob Keller and I hope to call on and an itinerary which is farily definite at this point. Dr. Barrett has been most helpful in arranging for our visits in Great Britain. We in St. Louis have made direct contacts with the scientists in Sweden and in Amsterdam and I hope Dave Wood or others in your group will advise us as to whom we should see in Beyer at Leverkusen. There is general agreement here that it would be desirable to determine whether or not Beyer is as concerned about the potential problems for the marketing of polychlorinated biphenyls and whether or not they have equivalent research efforts underway.

I believe Dr. Kelly went into this subject in some detail with Dave Wood but he is not sure that he discussed it at length with you last month.

Elmer P. Wheeler

TRAN 058769

EPW:cjs
Enclosures

DEPOSITION EXHIBIT
PlF - 286
6-11-92

STLCOPCB4027106

4/15/69

AROCLORS IN ENVIRONMENT

Scheduled Visits by R. E. Keller,
E. P. Wheeler (and W. R. Richard-Stockholm)

Monday, April 28 - Mr. Alan V. Holden
Dept. of Agriculture & Fisheries
Pitlochry, Scotland
Mr. Holden is coordinator of OECD countries programs covering influence of biocides on natural environments.

Tuesday, April 29 - Dr. J. Robinson and Mr. A. Richardson
Shell Research Limited
Sittingbourne Kent
These gentlemen have done considerable research on the presence of chlorinated pesticides in the environment and have told us earlier that they have confirmed the presence of polychlorinated biphenyls in wildlife since April 30.

Wednesday, April 30 Walkers Century Company
Stoke - with Howard Bergen and Bill Richard for discussions of Santosafe toxicity.

Wednesday afternoon or Thursday morning, May 1 - Discussions in Monsanto House with
D. C. Holmes and O. G. Tatton
Labortory of the Government Chemist
These gentlemen have also published papers concerning the environmental contamination by pesticides and one paper concerning the presence of PCB's in residues.

Wednesday afternoon or Thursday, May 1 - Sir Frederick Warner
Chief Partner of Cremer & Warner
Consulting Ecologists
Dr. Barrett feels that this meeting would be helpful.

TRAN 058746

152

Friday, May 2 - Dave Wood & John Haggart - Monsanto Brussels
Review of the Aroclor problem and Monsanto's research efforts to protect product sales and uses.

1)

Monday, May 5 through Thursday, May 8 - Professors Gunnar Widmark and Soren Jensen
Institute of Analytical Chemistry
University of Stockholm
These two gentlemen first identified the interfering substances in chlorinated pesticide residue analysis as polychlorinated biphenyls. Although their work has not been published they have informed us that their research efforts have continued and they are still concerned.

2) Prof. Bo Holmstedt
Division of Toxicology
Kgl. Karolinska Institutet
Avd. Toxikologi
Solnaragen 1 - Stockholm
Either this gentlemen or others at the institute have been doing toxicology work on the polychlorinated biphenyls according to Professor Widmark.

Friday, May 9 - Professor H. Van Genderen
Veterinary Pharmacology and Toxicology
University of Utrecht
Utrecht, The Netherlands
Professor Van Genderen recently wrote us asking for samples of our Aroclors and indicated that he had identified PCB's in wildlife along the coast of the Netherlands. He indicated further that some toxicology work was underway.

Monday, May 12 - Leverkusen (Possibly)
Keller and Wheeler to visit Beyer depending on decisions reached in London and Brussels as to whether we should contact our competitors to determine whether or not they are concerned and whether or not they have undertaken research programs which we may have planned but which might be duplication of their efforts.

15'

TRAN 058747

4/15/69

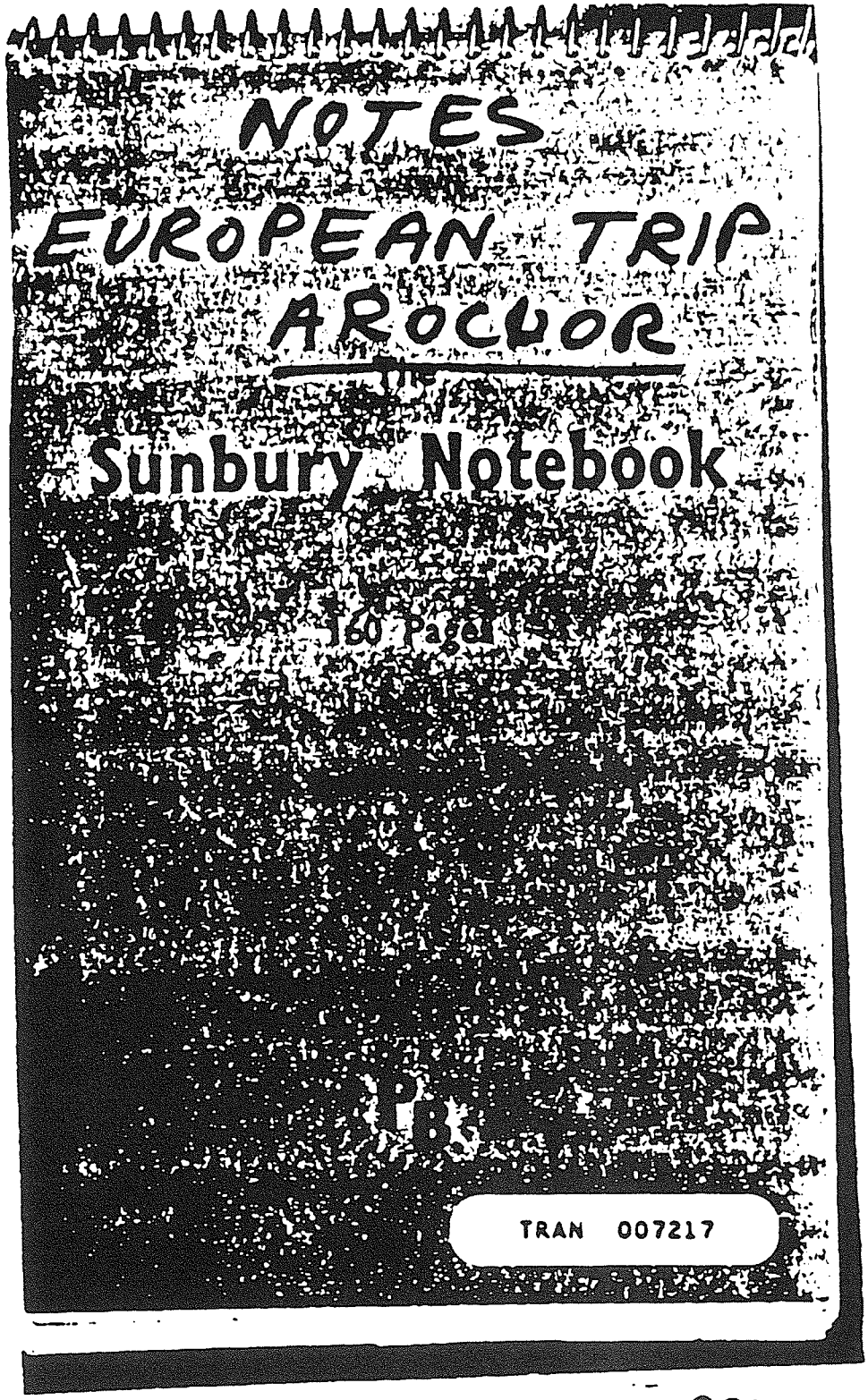
# ITINERARY - E. P. Wheeler
## Medical Department

| | | | | |
|---|---|---|---|---|
| Lv. St. Louis | Thurs | April 24 | 4:40p.m. | TWA #704 |
| Ar. London | Fri | April 25 | 9:05a.m. | |
| Lv. London | Sun | April 27 | 8:20p.m. | Train |
| Ar. Pitlochry | Mon | April 28 | 5:40a.m. | |
| Lv. Pitlochry | Mon | April 28 | early pm.? | Car |
| Ar. Edinburgh | " | " | ? | |
| Lv. Edinburgh | Mon | " 28 | 5:05p.m. | BE #5401 |
| Ar. London | " | | 6:20p.m. | |
| Lv. London | Tue | " 29 | 8:40a.m. | Train |
| Ar. Sittingbourne | " | | 9:33a.m. | |
| Return to London in afternoon | | | | |
| Lv. London | Wed | " 30 | 8:00 a.m. | Train |
| Ar. Stoke | " | | 10:07 a.m. | |
| Return to London in afternoon | | | | |
| Lv. London | Thurs | May 1 | 5:35 p.m. | BE 474 |
| Ar. Brussels | " | " | 6:25p.m. | |
| *Lv. Brussels | Sun | May 4 | 12:10 p.m. | Sk#558 |
| Ar. Stockholm | (Arlanda Airport) | | 3:25p.m. | (SAS) |
| *Lv. Stockholm | Thurs | May 8 | 4:10 p.m. | Sk #553 |
| Ar. Amsterdam | | | 7:05p.m. | |
| (need Hotel) | | | | |
| *Lv. Amsterdam | Sun | May 11 | 5:20 p.m. | KLM #233 |
| Ar. Dusseldorf | " | " | 6:00p.m. | |
| (need Hotel) | | | | |
| *Lv. Dusseldorf | Mon | May 12 | 8:15 p.m. | Air France |
| Ar. Paris | | | 9:15 p.m. | #765 |
| (need Hotel) | | | | |
| Lv. Paris | Thurs | May 15 | 4:00 p.m. | TWA #801 |
| Ar. New York | " | " | 7:00p.m. | |
| Lv. | " | " | 11:15p.m. | TWA #3 |
| Ar. St. Louis | " | " 16 | 12:49 a.m. | |

TRAN 058748

15°

STLCOPCB4027109

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DEPOSITION
EXHIBIT
Plfs 287
6-11-92

4-24-69

Dick Baxter - Opening Remarks

Tom Light - Guide Lines
Newport, Ruston

Tom Gray - Thermal Analysis
(DTA) Study of physical and chemical changes through temperature. DTA and TGA.
DTA Equipment - Schematic.
Du Pont
measured accuracy -
(TGA) Du Pont 950 Thermogravimetric analyzer
(Calcium Oxalate - $CO$, $CO_2$ Calc...
* Penetrometer Attachment
Du Pont Equipment
for penetration measurement.
Neoprene - Application.

TRAN 007218

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STLCOPCB4027111

Roberts - Ruabon

Atomic Absorption

Perkin Elmer 303 equipment currently used. Getting underway nicely.

Application at Ruabon -

Metals in rubber chemicals

V, Fe, Ca, P in sulfuric acid.

220 man hrs. / annum savings ~~[illegible]~~ at Ruabon

Digital concentration readout.

TRAN 007219

COMPANY CONFIDENTIAL

Doug Casey - Newport

Phillips XRF — Metals and non-metals in petroleum additives.
Ba, Ca, Zn, Br
Required 2 yrs. ago to replace more costly wet chemical analysis.
Two hr. results now vs 4 hr formerly.
Phillips PW-30 Semi-Automatic Sample Equipment — Would now choose complete automatic equipment.
Cr and W Tubes — 2 Kw.
Several analyzing crystals. From atomic nos. 30 (zinc) to 92 (uranium)
Prefer to use sample in form of liquids. Solids (metals) can be examined.
Count for several minutes

TRAN 007220

COMPANY CONFIDENTIAL

Low level chloride detn
Pt detn in cat.
- also being explored.
Cost of developing new method
50-150 lb.
Other applications - silicon
in silicon products.
Metallic element in Lactex.
Detection limit -
500 ppm down to calcium.
750-1000 " below calcium.
Relative accuracy - 0.1% level
- 0.5% of amount present.
Advantages - speed, non-destructive,
specificity,
Disadvantages - precision down,
sensitivity poor, high
capital cost.
* Send comparative AA/XRF
data for petroleum
additive analyses.

TRAN 007221

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STLCOPCB4027114

0.5 mg/in²
200 ml
100 sq. in.

Analytical Problems in the Toxicological Area

Mr. Moquand, R&D, Wingles (French) (Langs)

Dr Polystyrene ⇒ Acrylonitrile is very toxic. Styrene is also a problem. Food is wrapped in these products.

GC — sample dissolved in ~~[illegible]~~ solvent, ppt'd in alcohol. Impurities are examined by GC.

0.7 ppm Styrene in polystyrene can be determined.

TRAN 007222

Oils & Similar Products

Can't use GC because of interferes. Extract solvents to be analyzed with $CH_3OH$.

COMPANY CONFIDENTIAL

STLCOPCB4027115

[redacted]

Prof. Lewis - Bangor Univ.

Swiss test - used as extraction parameter, -

Pyrolysis / Gas Chromatography - Mrs J. Ellis

Polymer analysis. Sample coated on wire.

Can trap out certain gases or pass all to gas chromatograph

Rubbers - quantitative analysis of mixtures.

"Curie Point Pyrolysis" System

Not many published chromato programs. Previously difficult to get reproducibility between labs. - not now such a problem.

TRAN 007223

COMPANY CONFIDENTIAL

J. Newton - Nuclear Magnetic Resonance
Perkin - Elmer
R-10? NMR Spectrometer
High Resolution Instrument

Phenols - ~~Lead~~ Our NMR publication ~~to above~~ was referred to by Newton. - Did not work apparently for specific application due to impurities etc. in actual sas. vs. synthetic mixtures.

TRAN 007224

COMPANY CONFIDENTIAL

STLCOPCB4027117

- Fewley

Gary Fleet - Gel Permeation Chromatography of Polyethylene & Polystyrene

Have Waters Associates 100 GPC Instrument - purchased about 2 yrs. ago.

Computer Program Described Polystyrene -

Parameters

Column configuration

Toluene, Nonyl Acetate Solvents

Concentration - distortion can develop

Sample Volume

TRAN 007225

COMPANY CONFIDENTIAL

STLCOPCB4027118

F. J. Smith - Scanning Electron Microscopy
— Hydrocarbon Polymer —
Cambridge Instrument

Disadvantages — —
(1) Image seen - not photographic image.
(2) Operation - personal touch to get optimum settings.

TRAN 007226

COMPANY CONFIDENTIAL

John Davies — UV Lighting in the Study of Vulcanization

Natural rubber) —
Accelerator System
conventional
semi EV
EV

illustrates how application of simple tech. can be used for complex system.

John D. White — Information Data Retrieval and Storage.

(3) Centers
Reading
London
Newport

TRAN 007227

COMPANY
CONFIDENTIAL

STLCOPCB4027120

Function - to provide technical information.

Specialized Services
Weekly Publications -
Rubber Chemicals

Sample -
Physical Methods Bulletin

Trying to get up-to-date coverage for all of Europe
Cost of preparation -
36¢/abstract for master
= 2-10-D per year per recipient.

TRAN 007228

Index - System for method retrieval for this information. Trying to make service to information service specialist.

COMPANY CONFIDENTIAL

[redacted]

individuals would abstract
forwinds and -- provide inputs
for Gulletin.

TRAN 007229

COMPANY
CONFIDENTIAL

4-25

Angus Jones - Aspirin
Process Development

Exchange of info regarding McCrones work agreed on. MCL plans to spend about $2500.

Have purchased Swift PD microscope via McCrones recommendation. They are using our hot melt ~~test~~.

TRAN 007230

COMPANY CONFIDENTIAL

4-25-69 Discussions

UV atlas of Organic Compounds
Butterworths, London 1966
Published in Collaboration
with the Photoelectric
Spectrometry Group, London
and the Institut für
Spektrochemie und
Angewandte Spektroskopie
Dortmund
Library of Congress Catalog
Card Number 66-21542
Printed in Germany

G.C.
Tattor - Thermionic Micro
Detectors - Prepare
micro disc - put on
top of jet - 50/50 mixture
of cesium Bromide / ?
Thin Layer / GC - Flame
Detectors.

TRAN 007231

COMPANY
CONFIDENTIAL

STLCOPCB4027124

[redacted]

[redacted]

MCL - Punched Tape with Read Out Will Be Way Rusbon Will Go. - Don't have computer per se on site.

Attempt to get advisory panel to recommend purchase of instruments - a problem.

TRAN 007232



COMPANY CONFIDENTIAL

STLCOPCB4027125

[redacted]

Monday, April 28
Mr. A.V. Holden
Freshwater Fisheries Laboratory
Pitlochry, Scotland
Monsanto - Messrs.
D.V.N. Hardy and
R.E. Keller

Evidence for DDT degradation does not explain all chlorinated byproducts.
Could not use Jensens selective pesticide degradation procedure.
80-200 mesh
2g columns
Hexane partition system gives emulsions - use alumina column - gets rid of ~~pesticides~~ fats.
Hexane → silicic
Dry 120°
Add 5% water
Hexane 10% ether
PCB's come out fast. Gives 8-10 pesticides

COMPANY CONFIDENTIAL

TRAN 007233

STLCOPCB4027126

1 g. tissue
5 g. anhydrous sodium sulfate
↓ Grind Mortar + Pestle
extract Soxhlet Extractor
1/2 hr.

Gives complete recovery.
Spike tissue gives complete recovery.
Radioactive DDT used
to check recovery. Couldn't pick
up residual $C^{14}$ DDT. Not conclusive

GC - isothermal 200°C
electron capture
chromosorb W
80-100 Mesh
Acid Wash

Published by FDA 8-5 yrs ago.

One column: Coated 10% DC 200 (12,500 centistokes)

TRAN 007234

Other column: 5% DC 200
7 1/2% QF-1

COMPANY
CONFIDENTIAL

STLCOPCB4027127

must have 1500 plates –
" not breakdown NOT
" " have tailing
of peaks.
Cannot use nickel detector
– ~~go~~ peaks overshoot – difficult
to read.
Not using thin layer chromatography plate.
unacceptable level in water.
$\frac{1}{1000}$ ppm = conc. 1 ppm in
fish.

TRAN 007235

6 weeks exposure will
kill fish at slightly
higher level.
Threshold level – 0.001 ppm
Safety Factor 0 = 0.0001 ppm
Fish Muscle > 1 ppm
Steady Guild up over period.

Pain Boat Trout

COMPANY
CONFIDENTIAL

STLCOPCB4027128

[redacted]

Alan Hellon

Done ~ DDT and Dieldrin
- not done with PCBs.

Do not know if lower conc. for 12 mos. would be critical.

Do not know if others have done this with PCBs -

Other toxicological work?

Sensitivity of fish - more sensitive than others (Birds & mammals).

PCB toxicity to fish - called London - Govt Labs to find out.



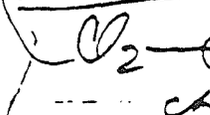
TRAN 007236

COMPANY
CONFIDENTIAL

STLCOPCB4027129

Crux of problem –
toxicity of PCBs –
Widmark + Jensen (Reference for Toxicity)
Chlorinated Biphenyl + Related
Cpds – Hubbard
Vol. 5 – Encyl. Article.

Produced in commercial products:



Tetrachloro biphenyl
dioxane.
van Genderen – Holland
$Cl_2$ – $\phi$ ~~[illegible]~~ $\phi$ – $Cl_2$
chloro-acne
chick-ne-adema } caused by above.
Sep can go from chlorinated biphenyl

Not likely that
degradation from
pesticides (DDT) occurs
to give PCB products.
conference
OECD – PCB's
mid-Sept Closed Conference
industrialists as OB.

TRAN 007237

COMPANY
CONFIDENTIAL

STLCOPCB4027130

Proposition for meeting

Birds / Fish - show 2 population levels chemically - one as normal,

Biologists - shows meaningful difference between levels. Set residue level on this basis.

Can't just accumulate data and not know significance.

Don't know how to set meaningful core levels.

Pay biological consequences to real world core. levels.

Will be put up as a proposal.

TRAN 007238

COMPANY CONFIDENTIAL

4-29-69

Nature - March 22 issue
VIC-221

Eichelberger, M. (1963)
J. Ass. Off. Agric. Chem.
46, 182

Aroclor 1254

Perkin-Elmer Application
No GC-DS-011
Analysis of Chlorinated
Biphenyls with Open
Tubular (Golay) Columns

Heron Eggs - GC/Electron
+ Oils Capture
More PCB than pesticides

VK
Gould has found 400 ppm in
PCB.

TRAN 007239

COMPANY
CONFIDENTIAL

STLCOPCB4027132

1 million gal of high pressure oil (lubricant) used in UK per year may explain higher ones. compared to pesticides.

3-4% of this volume probably PCB related.

1000 tons of DDT used in Kent since war.

Main DDT useage - on fruit.

Could be chlorinated waxes, naphthalenes, hexachlorobenzene (fungicide) -

TRAN 007240

Convert DDT to phos derivative - want direct measure not by difference.

COMPANY CONFIDENTIAL

Dick Baxter will [illegible] if
Aroclors are [illegible].
Confirmed 8-12 peaks
via GC - convinced PCBs
involved.
Shell used racing pigeons.
Results show no change
c̄ 10-12 ppm Dieldrin
in eggs.
100 ppm aroclor 1254 fed to
chickens - fed one year
- no mortality - egg
laying not effected
Shell thickness not
effected pcb.
Need residue levels
from such studies.
~~[illegible]~~

Alabaster - Govt. Labs.
Near Trafalgar Square
Whitehall Place.
↳

TRAN 007241

COMPANY
CONFIDENTIAL

STLCOPCB4027134

Note state of sample used for reporting ppm pesticides etc.
Concs. in reports may be quite selective - probably not found in other mats.
6 out of 20 birds had insecticides reported by Kolman.
Hydrolyze tissue with alkali to obtain extraction - for recovery expts.
Use MS-9 mass spectrometer.

Robinson
Richardson

TRAN 007242

COMPANY CONFIDENTIAL

Tatton

'53 - Noted these PCB spds being used in food phy in govt. annual reports.

'63 - Pesticide GC w/ R. showed peaks not organochloric pesticides associated c̄ wildlife interference - used [illegible] + GC Techniques. Not sure if breakdown [illegible] of what. Jensen in Sweden pointed out type. Reported this in paper - found in wildlife, eggs. Not in foods, rare in human fat. 0.01 ppm PCB detection limit. 2-8 ppm pesticides DDT in human fat (Britain) USA. No PCB found.

COMPANY CONFIDENTIAL

TRAN 007243

STLCOPCB4027136

Have found traces of PCB in human lung, $1 \times 10^{12}$ ppm DDT in air normally.

$\frac{1}{1000}$ PPB  American PPB $10^{7}$
British " $10^{12}$

Nature - 1966 - ~~[illegible]~~ Tatton

100-1000 liters - Don't find toxaphene etc - don't use in Britain. Very high DDT content in London - sticks to carbon in air.

Curious about what PCB's are used for in Britian - how do they get to birds - do it get to man or to domestic animals.

TRAN 007244

COMPANY CONFIDENTIAL

STLCOPCB4027137

Heron lives - 200 ppm
PCB found. Can't estimate
accurately. Nature Conservatory
sends livers, eggs + Brains
PCB Peaks in birds represent
only a portion of those
in cpds. - Won't comment
on higher chlorinated
species.

These cpds are not
pesticides - interest
as interferences. Attention
to existence has been brought
up c̄ ministry of Food.

Uses - 50% as (1) Dielectric
fluids in sealed containers
life 15 to 20 years.

(2) Heat transfer fluids

TRAN 007245

Concorde - blows
out chlorinated biphenyl
- we don't think so!

COMPANY
CONFIDENTIAL

q - what about uses as fixtures for pesticides?
- as plasticizers? - have tried to promote as plasticizer extenders. Chlorinated rubber paints. Hot melt adhesives.
- Have found PCB in terrestrial Hawk type birds as well as sea birds.
- Chemical degradation of pesticides carried out - could not produce GC chromatogram?
10 peaks - correlate with 54 Aroclors - suggest that bird recently had meal.
Owl, sparrow hawk
Kingfisher, highest PCB feed wholly on ~~from~~ fresh water fish.
No identity of source of bird.

TRAN 007246

1

COMPANY CONFIDENTIAL

STLCOPCB4027139

Dr. N.W. Moore
Marks Wood Expt Station
Huntington
- than who can identify
source of birds.

Jensen - peaks represent
Bayer product and not
Aroclors. Storage in
human fat pointed
to in plant workers?
Mothers milk - high
pesticide content.
ICI - polychlorinated
napthalenes - have not looked
out - a possibility.
Tatton has never looked
at our individual Aroclors.
Have looked at all 6
of our Aroclors. Jensen
has separated some by
prep G.C. Will sep
& isolate + use mass

TRAN 007247

COMPANY
CONFIDENTIAL

STLCOPCB4027140

within month
Sometimes find traces PCB
in milk, beef fat
Fluorsil column cleanup
- costly don't use in Britain
lose PCB's - down
sink,
Jensen & Shell
Van Genderen Only [illegible]
in Europe using
mass.
Have never achieved
more than 10% improvement
in peaks beyond 10 to 12.
Confirmed no one else
in UK other than Shell
+ Holden who are working
on problem,

TRAN 007248

Significance - Residue problem
primarily ≡ aquatic birds +
fish - not domestic animals
+ humans.

COMPANY
CONFIDENTIAL

STLCOPCB4027141

No PCB's found in rivers, waters of UK. Parts per 1000 million level.

Tatton will send Annual Reports etc. through Dr. Hardy.

Concerned primarily as interference to jet Composition of [illegible] gases.

We will provide GC data for Tatton et al as developed.

Lancet publication — will be included.

Mr. Collins Works
Mr. Tatton for
J.O.G.

TRAN 007249

1

COMPANY CONFIDENTIAL

STLCOPCB4027142

Widmark -

1964 - Grants for pesticides
Jensen started working at
this time. Found PCB
OECD program - Mr + Mrs
Stickels representatives -
Dr. Jensen gave lecture
and showed presence
of PCB. Widmark was
not eager to have results
published at this time.
Hard decisions. Group
of ecologists Johnels museum
- worried about organs
mercury in cpds.
End of March 1969 - Stockholm
meeting - most of people
doing anal. work were called.
DDT, DDE & PCBs 7 ppm increasing
levels were found -
heads of fish (salmon)
eaten by seals

TRAN 007250

COMPANY
CONFIDENTIAL

STLCOPCB4027143

Widmark refused to sign - fish taken from Baltic - some taken near Stockholm. Sas collected by group of ecologists + sent to Institute for Analysis.

PCB levels also reported

* Very difficult to quantify PCB results - too many isomers - don't know which group working with - food chain - some might be metabolized.

Holland fish contain less PCB's than those in Sweden.

Do not find chlorinated naphthenes.

TRAN 007251

COMPANY CONFIDENTIAL

STLCOPCB4027144

Naphthenes are [illegible] metabolized compared to PCB's. * in Stockholm Lab. Only 2-3000 was analyzed. Not enough. No summary of information - more of finding by one single man.

* 5, 6, 8 chlorinated biphenyls are not metabolized - theory. Not many below 5 are found

German chlorinated 50% biphenyl used as a standard.

Fig 5 Peak 3 diminishes through food chain - peak 10 (5 chlorine) increases. This is evidence for metabolism.

TRAN 007252

COMPANY CONFIDENTIAL

Toxaphene + $H_2SO_4$ [illegible]

PCBs

* American eagles gave PCB chromatogram after $H_2SO_4$ treatment than reference German chlorinated biphenyl.

American OECD group - Dr. Hickels - Pantanal will report analytical results. 5 sas. circulated to all 12 labs. There will be extreme differences between labs. Representative portions of homogenized sas. will be sent. Each lab. will use own method. Wants to emphasize anal. difficulties.

Sept meeting - scheduled 6 months - for results.

TRAN 007253

COMPANY CONFIDENTIAL

STLCOPCB4027146

List of OECD representatives is available —

Paris — 1st OECD — fight

Sweden/US against UK, Holland etc

Finally only UK against forming ~~again~~ ecology group for wildlife chlorinated residue studies.

All of labs. should have reported results.

Background of build up in birds + fish in non-industrial areas. World wide picture will be obtained - form basis for future action.

TRAN 007254

Don't know where sas. are collected.

No toxilogical work has been done which he can understand.

(1/2 Ecologists, 1/2 Chemists)

COMPANY CONFIDENTIAL

STLCOPCB4027147

Only very few toxicity studies being done in Stockholm. Too little done.

Very little hope to combine analytical/ toxicology activities in Sweden.

OECD group not really considering toxicological results. Will probably come later.

USA - OECD Representatives

* Dr. J.L. Buckley - Director of the Office of Ecology, US Dept of the Interior, Washington DC 20240

Mr. W.H. Stickel + two - Patuxent Wildlife Research Center, Bureau of Sport Fisheries and Wildlife, US Dept of the Interior, Laurel, Maryland 20810

TRAN 007255

COMPANY CONFIDENTIAL

Mr. W. L. Reichel - Patuxent Wildlife Research Center, Bureau of Sport Fisheries and Wildlife, U.S. Dept. of the Interior, Laurel, Maryland 20810

Mr. S. Hall - Chief, Pesticides Chemicals Research Branch, Entomology Research Division, Agricultural Research Service, Beltsville, Maryland 20705

Sweden - Prof. G. Wilmark

Prof. A.G. Johnels - Swedish Museum of Natural History, Stockholm 50

Dr. S. Jensen - Institute of Analytical Chemistry, [illegible] 90, Stockholm 50

TRAN 007256

UK - Mr. A.V. Holden

Dr. D.C. [illegible] - Lab. of Plant Chemist

Mr. J.O.G. Tatton

Dr. N.W. Moore - National Conservancy Board [illegible]

COMPANY CONFIDENTIAL

in Vienna

IAEA - Radioactive methods for food studies discussed. Radio-active cpds. used to follow metabolic fate.

PCB/Environmental Problem - No single method able to give how. Must have very carefully planned expts. Not for environmental studies. Could work as well with PCBs - chlorinate with enriched $^{37}Cl$ and use as isotope dilution method.

Exit gases of auto cars - chlorine use to bleach cellulose - large no. of chlorinated aromatic compds generated - what are they?

TRAN 007257

COMPANY CONFIDENTIAL

STLCOPCB4027150

chlorinated phenolic cpds - phenolic group removed. No evidence hexachlorophenyl + pentachlorophenyl - digestion. Too few studies have been allowed to be done.

Prof. Widmarek not allowed to be paid personally - taken from university.

Must know - * where do PCB come from

* Must know more about toxicological levels

* Can contamination process be prevented. (smaller amts under) mostly 5 + 6 chlorinated biphenyl, no 7 + 8, trace 10, 11 on nothing. (no chlorinated terphenyls).

TRAN 007258

COMPANY CONFIDENTIAL

STLCOPCB4027151

mass sensitivity limits detection.

Should pursue picture on Aroclor 1254 - other

4 — 8 calorimetry + documented by mass spectroscopy

Not too many materials have enough PCB for mass identification.

Not impressed by analytical ability of Research.

How to quantify a problem.

Tell customers not to throw away product - will probably solve problem.

Thinks partly matter of education to limit exposure.

TRAN 007259

COMPANY CONFIDENTIAL

Jensen - going to be ecologist.

Society will ask for bio-degradable cpds in the future.

Who raises question of toxicological position:

Mat. Balance of chlorine cpds? - can't account for total chlorine cpds. Done with extractable part. Do not check residue.

- what's to come

Mass ~~spec~~ - Repetitive scanning - CAT - to be used - on line computer

TRAN 007260

Have actually used Monsanto Aroclors as references - similar - not exactly. Can't be used as basis for identifying source or supplier.

COMPANY CONFIDENTIAL

Can show different mfg. process. Said our product contains non metal materials. Will send GC chromatograms of Monsanto + Competitive products.

Ecologists - don't want chemicals in nature - don't understand chemicals. In about 2 years will have a better feeling about whole problem.

Wisnuk - promotes dropping PCB and substitute product. Thinks it premature to support position.

High 2 weeks PCB users were contacted regarding transformers.

TRAN 007261

COMPANY CONFIDENTIAL

Replied they had one accident with one man - fat & blood checked - not as high. Not able to start studies with humans - could not get govt. funds. Food handled by others such as FDA. Museum sas. sewage (sludge) - after biological treatment mud - source of PCBs. - similar chromatograms. Particular towns in Sweden are not under control.

$H_2SO_4$ acid treatment removes pesticide interferences.

Advice - Program

(1) Restrict PCBs to closed systems.

TRAN 007262

(2) Find substitute for PCB for other applications.

(3) Come up with positive analytical data so that biologist can't missue.

COMPANY CONFIDENTIAL

STLCOPCB4027155

Wilmark - does not want to bring info out in public. PCB discovery fine. Toxicity related thereto is nonsense.

---

Afternoon - contacts suggested

* (1) Dr. Jensen - ✓
* (2) Dr. Johnels - Museum of Nat. History ✓
(3) Karolinska Institute - ✓

* (4) Dr. B. Aberg - Fil. D., Professor, Royal Agricultural College, Dept. of Plant Physiology
Should see to set up contact for future.

TRAN 007263

Paper prepared for Nature - distribution of sas. & results. East, south west visited areas of Sweden being sampled.

COMPANY CONFIDENTIAL

STLCOPCB4027156

5-9-69 Prof. Van G.

Veterinary + Pharmacology Institute

Prof. VG.

From Public Health Institute

Concerned with pollution of environment — at sc. level not political level

TRAN 007264

Chairman on one of pesticide pollution committees acts as advisor to Public Health Institute.

Have veterinary students — not too many interested

Teaches pharmacology etc. Has contact with biologists — (10 students) — choose Ph.D projects — biological/toxicological type. Try to [illegible] students with problem of society as well as [illegible]

COMPANY CONFIDENTIAL

STLCOPCB4027157

Have gone into anal. work with wild animals. Diminishing birds was of concern - thought might be poisoned.

Problems

~~Pol~~ Chlorinated Hydrocarbon
Mercury Cpds
Eddie Duck } serious problem
? Bird }
- not sure if polychlorinated biphenyls are contributors.

Chorinated Biphenyls in Fish Mussels and Birds from the River Rhine and the Netherlands Coastal Area
Nature 221, March 22, 1969

TRAN 007265

COMPANY CONFIDENTIAL

STLCOPCB4027158

Chlorine atoms as low as 3 but as ~~highly~~ normally as 1260.

Broad findings on PCB in environment by Prof V.G et al.

Can expect emphasis on PCB's.

Have used Monsanto + European suppliers as sources for reference materials.

No work on domestic animals + humans.

Feel from water environment. Only matter of time for humans. Highest in birds. Higher figures for Rhine Than sea. Galiene contains coming from Germany.

TRAN 007266

COMPANY CONFIDENTIAL

No work on conc PCB in Rhine – only lower chlorinated PCB's – probably escape from water. PCB's are higher in aquatic life than pesticides.

Aroclor Sample Identification –

Aroclor 12~~40~~ –
1st par Aroclor 1254 –
Aroclor 1260 –
" 1268 –

Invoice June 11, 1968
No. ME-7679/04

Lot AK-3

Dispatched June 12, 1968

Monsanto Europe

Sent To Drs. J. Koeman
Institute [illegible] Veterinary

TRAN 007267

COMPANY CONFIDENTIAL

STLCOPCB4027160

Hex Chlorobenzene worse than European PCB in toxicity studies - no work c̄ other pesticides.

Varian Atlas separator was used. Reference (7) isothermal used c̄ GC/MS hook-up.

* Would like any s.s.c. of pure isomers we prepare for their studies.

M.C. Ten Noever De Brauw
↳ involved with MS work.

TRAN 007268

COMPANY
CONFIDENTIAL

STLCOPCB4027161

5-12-69 Bayer

Dr. Knust - Technical Service for Electrical Industry
[redacted] Comments
sales of products

Dr. Frazer - Pesticide Problem

Dr. Joachim George - Manufacturer of Products.

Erhard Martin - Marketing

EM - Any concern on part of PCBuster.
Dr. K First confronted with Jensen work. Agreed that anal. work valid.
Toxicity - acute only considered.
Suggestion - study concs. in factory workers with long exposure.

TRAN 007269

COMPANY CONFIDENTIAL

STLCOPCB4027162

Will proceed as first step and give us date.
If no decomposition in body no toxicological effect via Widmark.
Ships have been painted c̄ chlorinated biphenyls, napthalenes etc mentioned.
Some use as lubricants. - use has been suppressed.
Put fish in Casin painted c̄ chlorinated biphenyls.
DDT + pesticides in spite of toxicological ~~problem~~ [illegible] are being banned. Must control useage so no detectable contamination of environment.
Prob. - Toxicology is not sufficiently an exact science.

TRAN 007270

COMPANY CONFIDENTIAL

STLCOPCB4027163

mixtures are a problem in nature? must be sure no detectable DDT in PCB per se studies.

Find source

What can be done to continue use

must be approached while pursuing toxicity problem - Monsanto will attempt to show no effect level.

Dr. Kurst - suggest they might study effect on fish.

Higher chlorinated biphenyls suggests electrical products are not involved.

Will do statistical work about workers in PCB over period of years.

No actual analytical work done yet by Breyer.

TRAN 007271

COMPANY CONFIDENTIAL

STLCOPCB4027164

Dr. Martin - started out to show PCB content in liver + compare to illnesses/effects.
Could scrape paint from ships, road markers, paint residues in tins - higher chlorinated ones most present.
Best we could use for closed system - heat transfer, electrical fluids etc. Develop profile
Lubricants, paints main source of contamination in nature.
Get info on painted reservoirs.
Advice - get toxicological data as soon as possible.

TRAN 007272

COMPANY CONFIDENTIAL

STLCOPCB4027165

Work likely with Organic Chemicals Division.

[illegible]

Intercontinental Hotel
Frankfurt
Ludwigshafen

Mr. Bilger, Stevens

Dr. Helmut Frehse
Scientific Crop Protection
Department
Residue Laboratories
509 Leverkusen - Zayerwerk
Abt. Pflanzenschutz
Wissenschaft

Car will pick up at hotel
at 9:00 in morning.

TRAN 007273

COMPANY
CONFIDENTIAL

STLCOPCB4027166

5-13-69 Dr. Koppmann

Letter of intent was received.

I:- No comparison to Res. Anal. Group at BASF will apply method. Agreement from BASF.

TRAN 007274

II. Write down - very seldom used by BASF, Not necessary for plant control. Should omit method - not necessary to look for this.

Quite low 10 ppm - if stopped further analysis - did it [illegible] customers asked for it.

COMPANY CONFIDENTIAL

III 1. OK

2. agreed ~~at~~ on May 27 meeting c̄ Bilger / Stevens — ~~[illegible]~~ to 90 minutes @ 250°C.

Temp. same — time difference only difference.

3. Cant say both tests are equivalent [illegible] get poor sulfuric acid test + good heat test. Not included in specification tests.

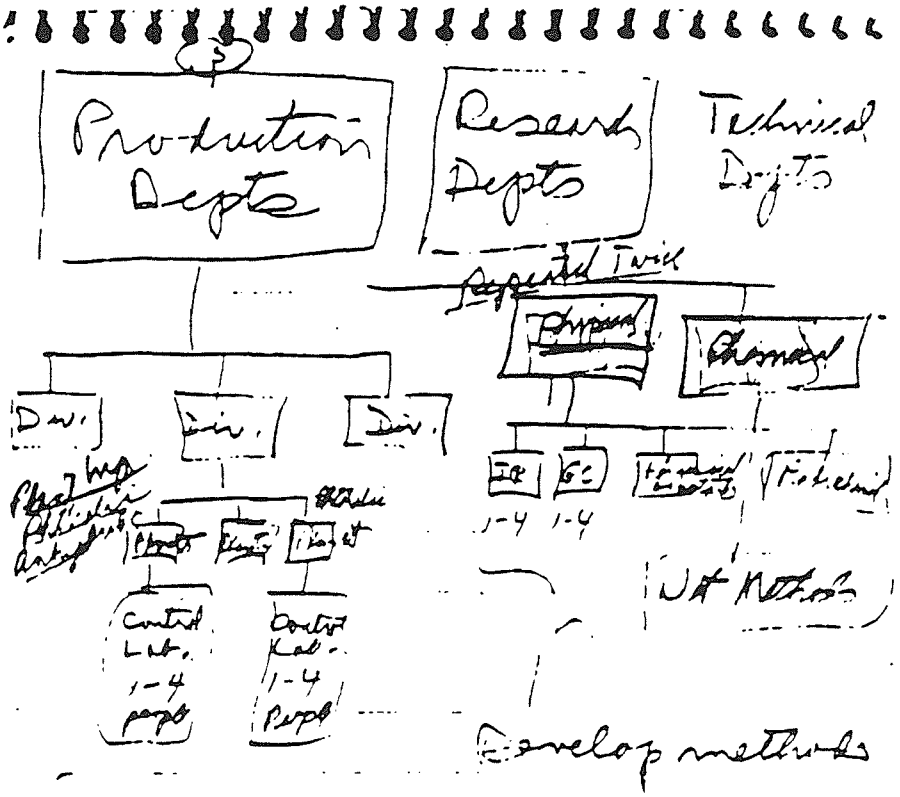
TRAN 007275

4. OK

5. Procedure developed in anal. dept. & set up in control lab.

COMPANY CONFIDENTIAL

STLCOPCB4027168




5 b) Anal. people didn't calibrate sufficiently low lead. No problem sensitivity-wise. Extremely low figures will be obtained.

c) Apiezon - misspelled

TRAN 007276

COMPANY CONFIDENTIAL

STLCOPCB4027169

5 d.) Dr. Koppenaal will send chromatogram within 2 weeks.

6. a.) OK

7. a.) OK

8. Assay should be 99.7% — difference figure O.K. in this case.

Crude product - still relatively high 97-78% - different method still O.K.

(50-70% of A)

Topping & tailing products - lower conc. - could take titration method which includes all acids. No problem with this figure to date.

TRAN 007277

COMPANY CONFIDENTIAL

Dr. K. will discuss with other plant mgr. to detn if significant.

7 a) Comparison table shows that Monsanto ash figure is higher - perhaps giving support to concern about high temperature.

* How did Monsanto carry out ashing procedure. Method No. 20 A, B, C, D - Ash Detn - followed exactly.

TRAN 007278

BASF will check on ash methodology to detn if low answers are obtained by direct thermal ashing.

Further check at meeting shows that analysis not carried out by BASF

acca

COMPANY CONFIDENTIAL

STLCOPCB4027171

Is ash in specification?
- Phone call made.

If in, BASF will indicate the sensitivity (detection limit) of their procedure

VI. 1. Get good yields by this method - once per week for each reactor. No critical parameters on the design of collection system.

TRAN 007279

Could use continuous GC method - yes, but necessary with smooth process operation

yield - on line burner to $CO_2$ - BASF doesn't do it.

measure benzoic acid, toluic acid

BASF has done no on line process monitoring - not necessary in their opinion.

COMPANY CONFIDENTIAL

STLCOPCB4027172

IR on line analysis for oxides of nitrogen - did not work successfully - ~~plotting of lines~~ mechanical problems. Subsequently dropped. No on-line instrumentation involved with the process.

III 1. Don't know if there is a sampling problem - non representative portion of solids - PA for example - no problems homogeneous samples encountered.

TRAN 007280

2. Open ended - yes perhaps could be ~~alternate~~ method. For waste water treatment purposes only.

COMPANY CONFIDENTIAL

This is not done continuously - perhaps every few months. Upon request.

VII 2. B) - Will provide conc. levels in BASF plant - very low - 160 kilograms org. mat / 70 cu. meters of water.

TRAN 007281

VIII. Only done on request. Art category.

Opinions - BASF has not put in spec. Don't know if Monsanto requested opinions of most concern with our manufacturing process. To best of knowledge - none are available - will provide info on what has been done from BASF

COMPANY CONFIDENTIAL

STLCOPCB4027174

Free Phthalic acid Solubility

| T° | Conc. |
|---|---|
| 30 | 0.79% wt/wt. |
| 40 | 1.1% |
| 50 | 1.7 |
| 60 | 2.5 |

TRAN 007282

COMPANY
CONFIDENTIAL

5-14-69 - Prodelec
marketing
Subsidiary of 50%
Owned By Prodjel

Mr. Lombre - In Charge of Analytical Process Work.

Grundig EN3 - Tape Recorder
Made in Germany

OECD - Organization Economic Commercial Development

TRAN 007283

Reactions to Jensen - via visit.

Lower chlorinated biphenyls - have we done any work to detn if lower chlorinated species are degraded - if not due to source - eliminates capacitor industry - 3 chlorine.

COMPANY CONFIDENTIAL

STLCOPCB4027176

Hypothesis - Germany ~~[illegible]~~ installation ~~factories~~ ~~[illegible]~~ ~~bombed~~ were bombed - Germans substituted Aroclor for oil.

Purification done with Fullers Earth - Bayer probably discarding into Rhine river.

Don't know conc. of PCB in ~~treatment~~ plant.

TRAN 007284

Prodigl wastes will end up at Marsilli in the Mediterranean

Jensen Visit - Found by electron capture GC and mass spectroscopy. Related full bird story. Found 50X PCB content in liver of salmon [illegible]

COMPANY CONFIDENTIAL

STLCOPCB4027177

to other fish. Salmon farm - found out tank walls had been painted with PCB paint.

Done some work - with std. chlorinated biphenyls - flame detectors.

Visited on April 23.

We visited on May 5.

Jensens -

TRAN 007285

Thought first source was capacitors industry - found high conc. of PCB in pine needles near factory. Found PCB in hair day after they visited factory.

Sievert - use any 3 chlorine biphenyl. Supprising that higher chlorinated only found.

COMPANY CONFIDENTIAL

STLCOPCB4027178

- Boats - found many ships painted - mussels cling to ships - fish eat mussels - birds eat fish.

Transformers - no markets for PCB - should not be a source of pollution - PCB bound 5.1 ppm in livers of fishes - One kilogram of Chlorinated Biphenyls with contaminated one million fishes.

Not a man of humor - rough man / sad.

One sheet - giving chromatographic details (electron capture) with Aroclor retention times given to Project Mr. Lombre

TRAN 007286

COMPANY CONFIDENTIAL

STLCOPCB4027179

No program set up yet - waiting for our meeting for decision.

First thought of looking around factory first.

How much toxicity fore will Monsanto put on this problem - consultant Bio-Test will maybe have 10 men.

TRAN 007287

→ * Send methods + details - on all of Widmark/Jensens analytical work to Pierre Jay.

Will coordinate future analytical studies.

→ Need to firm up lends of higher chlorinated species.

COMPANY CONFIDENTIAL

STLCOPCB4027180

Will do mass work outside + no inside facility.

Spill way levels in dams suggested possibility ~~as~~ lubricant contamination for rivers.

TRAN 007288

Will arrange anal. procedures - will look at factory. Would like to exchange samples.

Breyer uses antimony as cat. instead of iron used by Prodelec + monsanto. Relative heights IR are different with German product.

Not as concerned with PCB's as we are - will not touch capacitor industry in France. Use is so

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STLCOPCB4027181

important - just
Doesn't think authorities
will eliminate use of
capacitors. Most
attack will be on
plasticizers - where other
products can be used.
Sales in this area not
too high with Prodelec.
Stability of chlorinated
naphthalene.
Toxicology program
- nothing going.
Have never done any
toxicity work - have
relied on old German
papers.

TRAN 007289

Work on phosgene
on arcing of Askarel.
Will buy analytical
equipment for analysis.
May exchange samples.

COMPANY
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STLCOPCB4027182

Dr. Suter
Badische Aniline Soda
Fabrik
Ludwigshafen, Germany

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R. Keller called.
Will be at Intercontinental
Hotel, Frankfurt tonight.
9:00 at hotel

TRAN 007290

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COMPANY
CONFIDENTIAL

STLCOPCB4027183

Notes from trip of European T/P

ROUGH DRAFT - 6-12-69 Dictated by R. E. Keller

April 28th: Mr. A. V. Holden, Dept. of Agriculture and Fisheries, Fresh Water Fisheries Laboratory, Pitlochry, Scotland. Mr. Holden is a very rational and understanding person on the question of pesticides and PCB's appearing as residues in wildlife. In fact, he responds more like a Monsanto employee than a government laboratory representative. He discussed very openly and willingly all details of his work and exhibited very cooperative attitude for future work of any kind that might develop either between ourselves and his laboratory or ourselves and other laboratories in the United Kingdom. The following pieces of random information were picked up during the course of our conversation with Mr. Holden.

When asked about the possibility of pesticide degradation and the production of chlorinated biphenyl products, he commented that there is evidence for DDT degradation but that it does not explain all the chlorinated products being found as interferences in work directed toward pesticide residue measurements. His prime concern has been from the interference standpoint of PCB's in measurements directed toward pesticides. He has not made an exhaustive, or for that matter, preliminary study of the type of interferences being found. His laboratory is one equipped quite well with gas chromatographic equipment but it has no equipment such as mass spectrometers needed for final identification of residues separated by gas chromatography. He commented that he could not use Jensen and Widmark's selective pesticide degradation procedure which they reported in their earlier work. The hexane partition system gives emulsions and he, instead, uses an alumina column which gets rid of fats. This is a column composed of 80 to 200 mesh alumina in 2 gram amounts in the columns. The silica gel dries at 120°C and to which 5% water has been added back has been used with hexane and hexane containing 10% ether to chromatograph residues containing pesticides. With this procedure the PCB's come out first and 8 to 10 pesticide peaks are also eluted in this manner but at a later date in the chromatograph. Tissue

DEPOSITION EXHIBIT P/FS 288 6-11-92

TRAN 022096

preparation involves taking one gram of tissue, 5 grams of anhydrous sodium sulfide, grinding ~~an~~ mmorter in pestal and extracting with a soxhlet extractor for 30 minutes. He apparently obtains complete recovery and this has been demonstrated by spiking the tissue and carrying out recovery procedures. When asked how do we really check the recovery with biological tissue, he commented that radioactive DDT has been used to check the recovery, however, those doing the work could not pick up residual carbon$_{14}$ DDT and the results were not conclusive. The gas chromatographic procedure he commented on is one published by the FDA 3 to 5 years ago. The conditions are isothermal 200°C, electron capture, Chromasorb W 80 to 100 mesh, and acid washed. One column is coated with 10% DC 200 and a second column with 5% DC 200 and 7-1/2% QF-1. He commented the columns must have 1500 plates, must not break down DDT and must have no tailing of peaks. A nickel detector cannot be used because peaks overshoot making it difficult to read. No work has been done by Holden on thin layer chromatography.

Holden feels that an unacceptable level of DDT and Dieldrin in water is 1/1000 part per million. Experiments have been carried out where concentrations at this level have resulted in subsequent concentrations in fish at 1 part per million levels after six weeks exposure. At our levels, above 1 part per million, the fish died. This work was done using Rainbow trout as the fish. On this basis, the threshhold level for pesticides is 1/1000 of a part per million and with a 10X safety factor this becomes 1/10,000 of a part per million which would be a tolerable level regarding waters containing fish. The evidence showed that there was a steady build up of these pesticides in the tissue of the fish over the six week period. He did not know if lower concentrations for 12 months would be critical. He thought that work of this type had been done with PCB's, also in the UK government laboratories, but was not certain. At this point, he called London and the party on the other end said they would report back after they had searched the files. This call was not returned during the course of our visit and by arrangement with Hardy a follow-up will be made later to determine what was found at on



STLCOPCB4027185

this point. Holden commented that he felt that fish were more sensitive than other experimental species such as birds and mammals to toxicity studies of this type. When asked about where the PCB toxicity information was being obtained, he commented that Widmark, Jensen and others are referring back to the encyclopedia article [illegible] prepared by Mr. Hubbard on chlorinated biphenyls several years ago. This is Volume 5 of this encyclopedia. This work, of course, contains no real information toxicity-wise that's of value at the levels and under the conditions of concern related to the PCB environmental contamination. He commented that the OECD of which Ag is the secretary, planned a conference in mid-September. This would likely be a closed conference where industrialists would not be invited. His proposition for this meeting was to take birds and fish and show two levels of residues which could be considered as normal and abnormal and present this to the biologists and ask to show a meaningful difference between the levels and on this basis set a residue level. He feels that we just can't accumulate data and not know the significance. He recognizes the complexity of the problem and how to get meaningful concentration levels but emphasizes that the biological consequences to the real world situation must be clarified to know the significance of the residues being found. It will be interesting to see if there is really a proposition of this type and resulting follow-through coming out of the September meeting.

The following three reprints of publications were given to us upon our departure:

1. Cleaning of Tritium Foil Electron Capture Detectors published in the Journal of Gas Chromatography, July 1967.
2. The Examination of Surface Waters and [illegible] Sewage Effulence for Organo Chlorine Pesticides, published in the Journal and Proceedings of the Institute of Sewage Purification, Part 3, 1966.
3. Organo Pesticides in Seals and Porpoises published in Nature, Volume 216, No. 5122, pp 1274-1276, 1967.

TRAN 022098

STLCOPCB4027186

April 29: Tunstall Laboratories, Shell Research, Ltd., Sittingbourne, Kent. Those present for Shell were Dr. J. Robinson, biologist, Mr. A. Richardson, analyst, Dr. Stevenson in charge of experimental animal house, Mr. DeCorsen, laboratory administrator - and for Monsanto, R. E. Keller, E. P. Wheeler and D.V.N. Hardy of NCL. Shell commented that their analytical work was, and a part of it is still, based on work reported by Eidelman, M., in the Journal of Association of Official Agriculture 2 46 182 (1963) with Open Tubular (Golay) column" ~~chlorinated (Goulet)~~ (column) in Perkin-Elmer Application No. GC-PS-011. (Another reference was "Analysis of Chlorinated Biphenyls" Shell's work was directed toward the pesticide problem in its early stages. Later they ran into unexplained interferences and it wasn't until the work of Widmark and Jensen that they really knew the identity of these interferences. Their work had been concentrated, for example, on the analysis of Heron eggs and oils, using the gas chromatography electron capture procedure. During the course of this work, they ~~have~~ found a higher concentration of PCB's than pesticides. They commented that the government laboratories in the UK have found as high as 900 parts per million PCB's in such products. They also commented that the use of one million gallons of high pressure oil as lubricant is used in the UK per year and this may explain ~~our~~ high concentrations of this material compared to the pesticides found. About 3 to 4% of this total volume of high pressure oil would be PCB related material. They also mentioned that one thousand tons of DDT have been used in Kent since the war. The main DDT usage being on fruit. Shell has confirmed 8 to 12 peaks found by gas chromatography as PCB's and more specifically as those related to our Aroclor 1254.

Shell used ~~raising~~ racing pigeons to carry out toxicity studies. Results show no change where 10 to 12 parts per million Dieldrin were found in eggs. Other feeding studies involved feeding 100 parts per million of Aroclor 1254 to chickens for 1 year with no mortality and

with no effect on egg laying. Also shell pigment of the egg was not effected. A missing link to this type of study was the PCB residue levels related to the studies and this should be determined in the future. Shell has a very well equipped laboratory with an MS MF-9 mass spectrometer and personnel who are highly competent and very capable of carrying out studies of the type of Widmark and Jensen. They were very cooperative in their discussions and left the door open for future follow through, whatever it might be on this common problem. Their interest, of course, is primarily insecticide related and not from PCB viewpoint. Shell provided 3 bar graphs of mass spectrum for Aroclor 1254. In addition, the following publications were provided: a working paper on Mass Spectrometry of Organol Chlorine Compounds and Attempts to Reach Positive Identification which was read before the ~~XXXXXXXXXXXXX~~ meeting by ~~XXXXXXXXXXXX~~ Widmark in October, 1968 at the International Union of Pure and Applied Chemistry, Applied Chemistry Division, Pesticide Section, Commission on Pesticide Residue Analysis. The second paper is entitled, Some Recent Analytical Developments in Respect to Organo Chlorine Compounds in the Environment, by A. Richardson, Shell Research, Ltd., Tunstall Laboratories, Sittingbourne, Kent, England.

May 1: Laboratory of the Government Chemist, Monsanto House, London, England. Mr. J. O. G. Patton and G. B. Collins of the Laboratory of the Government Chemist, and Messrs. R. E. Keller, E. P. Wheeler and D.V.N. Hardy met and discussed the problem of PCB and pesticide residues. Mr. Patton reviewed the total problem as they had experienced it starting in 1953 when it had been noted that PCB compounds were being used in food packaging in government annual reports. In 1963, pesticide GC work in the governmental laboratories showed peaks which were not organochlorine pesticides related and this was also found in wildlife analyses. At this time they were not sure if the peaks being observed in gas chromatographic techniques were the result of breakdown products. Jensen in Sweden pointed this out in his report mentioned earlier. Overall, the PCB has been found in wildlife, especially aquatic wildlife, and eggs. It has apparently not been detected in foods and only rarely at very low



STLCOPCB4027188

levels in human fat. The detection limit is 0.01 parts per million PCB. The amount found in Britain in human fat is 2 parts per million and in the United States 8 parts per million, most of this being pesticide or DDT related. Traces of PCB have been found in the human lung. $1 \times 10^{-12}$ parts per million DDT has been found in the air. For clarification, American notation of parts per billion is 10 to the 9th, and the British notation is 10 to the 12th. Patton commented that there is a very high DDT content in the London air and this is apparently the result of the pesticide sticking to carbon in the area.

Patton was curious about what PCB's are used for in Britain and about how they get to birds since they apparently don't get to man or domestic animals. Again, it was mentioned that the Heron liver contains 200 parts per million PCB. He commented and qualified the statement that this level cannot be estimated accurately. The Natural Environmental Research Council conservatory in the UK sends liver, eggs and brain tissues to Patton for analysis. PCB peaks in chromatograms from bird tissue represent only a portion of those found. He felt that he could not comment on the entire chlorinated species simply because he didn't know what these might be. The question about Concord jet aircraft blowing out chlorinated biphenyls was brought up and Monsanto was asked about what we know on this point. To our knowledge, no uses of this type exist except for a very nominal amount. Patton has found PCB in terrestrial hawk type birds as well as sea birds. Chemical degradation of pesticides have been carried out in their laboratories and they could not produce GC chromatograms of the type related to PCB compounds. Again, Aroclor 1254 was associated with the type of chlorinated residues being found. Traces of PCB have been found in milk and in beef fat. Patton knows of no one else in the UK other than Shell and Holden who are working on the PCB or pesticide PCB related problem.

A significant part of the conversation revolved around the point that residues from PCB are higher in aquatic birds and fish than domestic animals and humans. No PCB's have been found in the river waters of the UK. This detection would be at parts per 1/1000 million level.

TRAN 022101

government
Patton will send annual/reports to Dr. Hardy at MCL who in turn will forward pertinent information back to the States. Again, they are concerned primarily with intereferences and not with measurement of PCB. It was obvious that a coordinated project has not yet evolved in government laboratories where the source of tissues in birds could or would be correlated with residue amounts. However, it is safe to assume that in time a special effort will be made to provide follow through on a program of this type in the government laboratories. It was agreed that we would be happy to provide GC data for Patton, et al, as we develop this in our laboratories. In conclusion, while this work has been very informative relative to the gas chromatographic separations, it is not worth involving positive identification through mass spectroscopy as has been carried out by Widmark.

May 5: Institute of Analytical Chemistry, University of Stockholm, Stockholm 50, Sweden. Messrs. Keller, Wheeler and Digby of Monsanto and Richard of Monsanto met with Prof. Gunnar Widmark and discussed the problem of PCB's and the environment. Widmark, while basically an analytical chemist, has considerable know-how about the total problem related to environmental contamination and the ecological system in general. Widmark is a rational person who exercises great caution in work that is done and published. He has regretted that the course has gone the way it has resulting from publication of their work. He commented initially that his colleague, Jensen, was no longer with him or a part of the Institute of Analytical Chemistry. Jensen subsequently could not be located as he was out of town and not available. It is our understanding that he will become a participant either in academic or government related coordinated projects more from the viewpoint of ecological system involvement as it relates to PCB. The following comments cover our discussions with Widmark today, May 5, and subsequently by R. E. Keller with Widmark on May 7.

Widmark surveyed the development of events leading to their work in publications involving PCB. In 1964, they had grants for pesticides



STLCOPCB4027190

and Jensen who was with the Institute of Analytical Chemistry, started working on the problem at this time. During the course of this investigation, they found interferences which in turn led to additional studies resulting in the identification of PCB's by mass spectroscopy. Out of this came the now famous lecture referred to by many in the States and in the UK and Europe as the first insight as to the identity of interfering components. Widmark was not eager to have these results published by Jensen at this time. At the end of March 1969, there was a meeting in Stockholm of most of the people presumably in Sweden doing analytical work. It was found that DDT, DDE and PCB's were increasing in concentrations in fish and aquatic wildlife. Widmark refused to sign any endorsement or recommendation out of this meeting. His position being that it is very difficult to quantify the PCB results because of the many isomeric species, and that we really don't know which group we are working with under these conditions. Furthermore, at this time, the metamollic processes in terms of stability of PCB type compounds was not known. The comment was made that Holland (?) fish contained less PCB than those in Sweden. Chlorinated naphthenes are not found. Naphthenes are probably metabolized in a manner similar to PCB's. About 2,000 to 3,000 samples, presumably wildlife, have been analyzed in the Stockholm laboratory of Widmark. In his opinion, this is not a sufficient sampling to provide a summary of the information obtained. His theory on findings observed is that the 5, 6, and 8 chlorinated biphenyls are not metabolized because not many below 5 are found. For his work, the German chlorinated 50% biphenyl was used as a reference standard. In figure 5, peak 3 diminishes through the food chain whereas peak 10 related to 5 chlorines increases. This is evidence for metabolism. Toxithene treated with sulfuric acid does not give PCB's. American eagle residues gave PCB chromatograms after sulfuric acid treatment that were different than reference German chlorinated biphenyl compounds. Dr. Stickles, at the Pawtuxet Laboratory in America, who is our representative with the OECD group is participating in a joint analytical program with Widmark. Apparently, 5 samples have been circulated to 12 laboratories. No common method has been circulated such as for round robin testing



STLCOPCB4027191

but rather each laboratory is to use its own procedure and make determination of PCB's and presumably pesticides in these common samples. Representative portions of homogenized samples were sent to each laboratory. One objective of this exchange is to determine analytical difficulties between laboratories and different procedures. Results are to be compared in about six months at a September meeting of the participants. One objective out of this work is to build a background of information about PCB's in residues in birds and fish in non-industrial areas. A worldwide picture will be obtained to form a basis for future action. To date, no real toxilogical work has been done in Widmark's opinion which he, at least, can understand. Only a very few toxicity studies are being done currently in Stockholm. In his opinion, too little is being done. He believes that there is little hope that analytical and toxilogical activities in Sweden will be combined. He believes that the OECD group is not really considering toxicological results and that this will follow later. The question of composition of exit gases from automobiles was brought up and discussed briefly. There is apparently no evidence for degradation of pentachlorophenol or hexachlorophenyl compounds. Widmark takes the position that we must know where the PCB compounds come from and more about the toxilogical effects. He apparently has not been impressed in his words by the analytical ability of Risebrough at the University of California. He thinks that society will ask for biodegradable compounds of the type we provide as Aroclors in the future. Widmark has work planned related to repetitive mass scanning with a CAT for more sensitive and perhaps specific determination of isomeric species being found in residues. Differences in Monsanto Aroclors and those manufactured in the UK and Europe are not sufficient to permit identification using mass spectroscopy techniques. Widmark agreed to send GC chromatographs of Monsanto and competitive product. He promotes the dropping of PCB as a product and in turn the offering of a substitute product. He thinks it is expensive to support the position we may have to take or prove or disprove the use of Aroclors. PCB users, presumably those involved mostly with transformers, in Sweden were contacted. ~~Most replied~~ they had one accident with one man in which the fat and blood was checked. The results from this apparently



STLCOPCB4027192

were not conclusive. No further work was done involving humans primarily because government funds were not available. When asked what he would do if he were in a manufacturer's position involving PCB, Widmark commented as follows:

1. Restrict PCB's to closed systems.
2. Find a substitute for PCB.
3. Come up with a positive analytical data so that biologists ecologists cannot misuse it.

Widmark provided the following reprints of publications for our reference: "Determination of the Number of Compounds which can Result from the Chlorination of Biphenyl and Development of a Simple System by which these may be Codified," "Methods of Analysis and Examples of Chromatograms," the OECD study of analysis of PCB, December 1968 and "Results Obtained by Analysis of the OECD Exchange Samples 1, 3 and 4." Our methods that we either are using or plan to use for the gas chromatographic work and mass spectrometry work were reviewed with Prof. Widmark. Comments from these discussions are noted directly on the reference attached copies of the individual procedures. He feels that the chromatographic and mass chromatographic procedures should be similar for direct comparison as opposed to having separate conditions such as isothermal vs. programmed temperature separations.

May 9: Professor H. vanGenderen, Veterinary Pharmacology and Toxicology, University of Utrecht, Biltstraat 172, Utrecht (Netherlands). Messrs. Wheeler and Keller discussed with Prof. vanGendern and Drs. J. H. Koeman, biologist, W. L. van der Maas, chemist and T. G. Vos, veterinary pathologist, the problem of PCB and environmental contamination. Prof. vanGenderen is concerned with pollution of the environment at the scientific level and not at political levels. He is chairman of one of the pesticide pollution committees in the Netherlands and acts as an advisor to the public health institute. He teaches pharmacology at the university, has contact with biologists and uses students on PhD projects related to biological toxicological studies. He tries to involve students with problems of society as well as scientific



STLCOPCB4027193

direction only. The problem of the diminishing birds and wildlife species has been of concern with the thought that this may be caused by poisoning. Chlorinated hydrocarbons, mercury compounds and perhaps polychlorinated biphenyls are contributors to the demise of certain species. For this reason, they embarked on a program to make a study of fish and birds from the Netherlands area and the resulting work was published in Nature ~~xxxx~~ entitled "Chlorinated Biphenyls in Fish, Mussels, and Birds from the River Rhine and the Netherlands Coastal Area", the reference being Nature, Volume 221, pp 1126, March 22, 1969. Monsanto and European products have been used as reference PCB material. No work has been done on domestic animals and humans in terms of analysis for residues. It will be only a matter of time until human~~s~~ studies of this type are made according to vanGenderen. The highest residue levels appear in birds and the higher figures are related to the Rhine as compared to the sea. On this basis, vanGenderen believes that the contamination dateline(?) is coming from Germany. No work is underway on the concentration of PCB's in the water of the Rhine. He believes that it is only the lower chlorinated PCB's that escape from the water. The PCB's are higher in aquatic life than pesticides. Their toxicity studies indicate that hexachlorobenzene is worse than European PCB's and toxicity studies. There has been no work with other pesticides. The mass spectroscopy work for their studies is carried out at a separate laboratory on a contract basis; a facility which is similar to the Battelle Institute in the United States. The mass spectrometer is a Varian Atlas CH4 instrument. Upon questionning the members of this group more closely about the mass work, it became apparent that this work was done elsewhere. At this point, vanGenderen and the others volunteered to take us to deBrauw, who actually did this work. After visiting with deBrauw, it became apparent that they have all the expertise and gear needed to do a completely competent job such as that by Widmark and Jensen. deBrauw is an expert in mass spectroscopy having installed 3 or 4 instruments in the States for the Varian Atlas Company. Comments from discussion with him are noted on the attached copy of our method write-up and will be recorded in this manner.

Notes on other parts of this trip are reported in call reports by J. Haggart and a special report by Hardy.

TRAN 022106

MONSANTO CHEMICALS LIMITED

# PRODUCT TOXICOLOGY - AROCLORS

Report summarising present knowledge concerning chlorine-containing residues in wild life and visits made to organisation implicated therewith during the period April 28th - May 1st, 1969.

by: D.V.N. Hardy*

Circulation

J.W. Barrett, London.
R.A. Baxter, Ruabon.
H.A. Voddon, Ruabon.
H.R. Newman, Newport.
A.C.W. Pemberton, Newport.
W.J.S. Budd, London.
J. Herefousse, Brussels.
D. Wood, Brussels.
R.E. Keller, St. Louis.
E.P. Wheeler, St. Louis.
R.E. Kelly, St. Louis.
W.R. Richard, St. Louis.
H.S. Bergen, St. Louis.

*Formerly Research Services Manager, MCL., and now Consultant to MCL in the area of Product Toxicology and Safety in the Use of the Company's products.

TRAN 005818

DEPOSITION EXHIBIT
Plf. 289
6-11-82

STLCOPCB4027195

INTRODUCTION

The wide agricultural usage of insecticides in the post-World War II period and consequent effects on wild life have led in certain circles to apprehension concerning the overall effect on man. The situation has been under study for many years and Government agencies and manufacturers of insecticides have been implicated in the various aspects, particularly in the assay of such residues in many species of wild life. Fish, fish-eating birds and mammals have been subjected to wide ranging study, and it became clear that terrestrial usage of insecticides could have far reaching and hitherto unsuspected effects on wild life, part of it making an important contribution to the diet of man and domestic animals.

The size of the wild life specimens and the minute concentrations of insecticides therein demanded highly sensitive methods of assay, which have been progressively refined. Gas liquid chromatography using electron-capture or microcoulometric detection has become established as the most suitable technique. It appears that the chromatograms of the residues showed not only the peaks corresponding to the insecticides and their known metabolites, but additional peaks of unknown chlorine-containing substances.

Towards the end of 1965 these unknown substances were identified by Sören Jensen, a research worker at the Institute for Analytical Chemistry, University of Stockholm, under the direction of Gunnar Widmark. Using a combined gas chromatograph mass spectrometer (LKB-9000) it was shown that the peaks are due to polychlorinated biphenyls (PCB's). The evidence and reasoning are available and the conclusion can hardly be in doubt.

Unfortunately the announcement was coupled with sensational statements concerning the poisonous nature of the PCB's, which were calculated to cause misgivings both to public authorities and to the general public.

Jensen and Widmark found PCB's in specimens of pike from different parts of Sweden, fish spawn, a dead eagle and in human hair, and even on fir cones! The conclusion reached was that the PCB's originated from burning industrial wastes and were washed down by the rain. Examination of feathers from museum specimens of sea birds suggested that contamination commenced in 1944.

The original work of Jensen and Widmark has been extended and generally confirmed, notably by workers at the following establishments and much of the detail has been published:-



STLCOPCB4027196

Great Britain

1. Laboratory of the Government Chemist,
   Cornwall House,
   London, S.E.1.
   (Notably Mr. O'G. Tatton)

2. Freshwater Fisheries Laboratory, (Ministry of
   Faskally, Agriculture, Fisheries
   Pitlochry, Scotland. and Food)
   (Mr. A. Holden)

3. Tunstall Laboratories,
   Shell Research Limited,
   Sittingbourne, Kent.
   (Dr. J. Robinson and Mr. A. Richardson)

4. Monks Wood Experimental Station, (*Nature Conservancy)
   Abbots Ripton,
   Huntingdonshire.
   (Dr. N.W. Moore)

*Established by Royal Charter to (1) provide scientific advice on the conservation and control of the natural flora and fauna of Great Britain, (2) establish, maintain and manage natural reserves in Great Britain including maintenance of physical features of scientific interest, (3) develop research and scientific services related thereto.

At 30th September, 1963 there were 144 scientifically qualified staff and in 1963-4 there was a Government grant-in-aid of £701,000.

Holland

5. University of Utrecht
   (Professor H. Van Genderen)

U.S.A.

6. University of California
   (Drs. R.W. Riseborough, P. Rieche and S.G. Herman)

7. Cornell University
   (D.B. Peakall)

8. San Diego Natural History Museum
   (M.N. Kirven)



STLCOPCB4027197

This report is written following discussions with staff from Establishments 1, 2 and 3 in which Dr. R.E. Keller and Dr. E.P. Wheeler (1 and 3 only) were associated. Drs. Keller and Wheeler are proceeding to visit 5, University of Stockholm, to see Jensen and Widmark. They are also visiting Bayer to ascertain its reactions as manufacturers of PCB's (Clophens).

SUMMARY

1. There can be no doubt that traces of PCB's (Aroclors) are to be found in wild life, fish and certain items of human diet.

2. There is little evidence to show how serious this minor contamination will prove to be, but what evidence there is is reassuring.

3. There is little evidence to show how the PCB contamination arises. It may be by run off of wastes into rivers, or by burning of industrial wastes with consequent washing down by rain. It is remotely possible that the PCB's might arise by biochemical action on insecticides and other chlorine-containing materials which are similarly discharged into the rivers, seas and oceans. It could be that among the vast quantities of materials destroyed on land and at sea during the 1939-45 war, significant quantities of Aroclors and Aroclor-containing materials found their way into the rivers, seas, etc., and are being slowly released by corrosion, extraction, etc. (see footnote at end of report).

4. A considerable Monsanto effort will be necessary to obtain the necessary analytical and toxicological information for dealing with this threat to our commercial operations.

A. Visit to Freshwater Fisheries Laboratory, Pitlochry*
Monday, April 28th, 1969.

Mr. A.V. Holden - Officer in Charge.
Dr. R.E. Keller - MC
Dr. D.V.N. Hardy - MCL

This visit was made not so much on account of contributions made to the subject but because Mr. Holden acts as Co-ordinator

*Dr. E.P. Wheeler was prevented from taking part in this visit due to the sudden indisposition of Mrs. Wheeler.



STLCOPCB4027198

for the programmes of the O.E.C.D. countries concerned with the influence of biocides on natural environments, wild life, etc. He might therefore be expected to have a wider appreciation of the overall problem and of its assessment in terms of the hazard to man. On this latter aspect he was of the opinion that there had been a tendency to overplay the hazard to man of insecticides and PCB's, and that it was very doubtful whether the toxicities of PCB's were greater or similar to those of the insecticides. Experiments with rainbow trout in water containing 1 part of DDT in $10^9$ of water had shown that death occurred in six weeks after which the concentration in the fish muscle was 1 in $10^6$. In other words there was a concentration factor of 1000 in 6 weeks: this would take place mainly by absorption in the gills. He believed that some similar experiments with Aroclors had been carried out in the Ministry's London laboratory, and phoned for confirmation but was unable to get this during our visit. I am therefore endeavouring to get the answer to this perhaps significant experiment.

Mr. Holden's analytical work has been concerned largely with extracts of seals and fish taken in Scottish waters, and he has observed similar peaks to those found by Jensen and the Government Laboratory. Extracts from freshwater and seawater fish show fairly low amounts of PCB's, while seal blubber contains considerably more. Reprints of relevant papers were given to Dr. Keller and there was a discussion on analytical work, much of which was carried out for other bodies, e.g. the Nature Conservancy. Much of the work remains unpublished and he saw little chance of rectifying the situation.

B. Visit to Tunstall Laboratories, Shell Research Ltd., Sittingbourne, Kent.
Tuesday, April 29th, 1969.

Dr. J. Robinson - Biologist
Mr. A. Richardson - Analyst
Dr. Stevenson - In charge of Experimental Animal House.
Mr. De Corsen - Laboratory Administrator.

Dr. R.E. Keller - MC
Dr. E.P. Wheeler - MC
Dr. D.V.N. Hardy - MCL

Shell has naturally been concerned with the problem of insecticidal residues from the outset and the cost of the analytical effort and its interpretation must have been very considerable. To Shell the PCB aspect has been simply one of analytical complication, and although at times there have been suggestions from their commercial people that the focus of



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toxicological interest might be transferred from insecticides to PCB's, there has been no attempt to do so. In fact papers by Robinson do not refer to PCB's but make a very sound approach to the insecticides problem and to its true perspective. Nevertheless they had collected some information on possible usage of Aroclors, for example, they had noted that the Coal Board disposes of 1,000,000 gals. of high pressure lubricant each year. Did this contain Aroclor? We thought it unlikely but questions concerning safety in use of Aroclors had certainly been referred to MCL by the Coal Board and it was therefore likely that some minor uses have been developed. There might conceivably be a small content of Aroclors in the high pressure lubricant. Usage of insecticides was of a high order, and in Kent alone 1000 tons of DDT had been consumed since its introduction after the 1939-45 war. Reference was also made to usage of Aroclors in cutting oils; the Government Laboratory had suggested that this was a possible source of contamination. Cutting oils are normally sold as proprietary formulations and it would be difficult to estimate how much Arcclor is consumed in this form.

Shell would much prefer to have a direct method for estimating insecticides. Now, owing to the stability of PCB's, it was necessary to estimate insecticides and PCB's, then PCB's by destroying or removing the insecticides and getting the insecticides by difference.

Aroclor 1254 had been fed to chicken at 100 parts per million of the diet and no adverse effect observed over a period of 12 months. The Nature Conservancy has carried out feeding tests on Bengalese Finches with DDT and found no adverse effects, and similar results have been with chicken and quail fed on 10 and 20 parts per million of Dieldrin. Other workers found some effect on mallards and chicken. At the Government Laboratory it has been shown that human fat (from mothers' milk) may contain PCB. Reference was also made to the work of Mr. Alabaster, Ministry of Agriculture, Fisheries and Food, on toxicological levels with fish.

It was agreed that many of the specimens submitted for analysis were not representative of the total population, e.g. predators were heavily weighted, and of the sick and dead birds, were most frequently collected. There must be large numbers of each predator much less contaminated than those submitted for analysis.

Samples of fish oils and margarine (supplied by Unilever) have been examined, and while PCB's can be found in the oils, they disappear during the hardening process so that margarine is free.

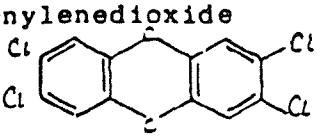


STLCOPCB4027200

The decrease in bird populations which is made much of by the Nature Conservancy and others cannot be attributed wholly to organic chlorine compounds. Factors such as exceptionally cold winters (e.g. 1963-4) and the decrease in hedgerows (10,000 miles/year are eliminated in U.K.) must have an important effect.

Reference was also made to wood preservation with PCP and to the possible effects of waste discharging, but this seems an unlikely source of PCB's.

There was some discussion of the chick oedema factor which has been attributed to the use of trichlorethylene as extractant. Here Dr. Wheeler spoke of the highly toxic tetrachlorodiphenylenedioxide




with which the Germans and MC have had experience.

We outlined the main uses for Aroclors as being:-

Dielectrics for condensers, transformers and switchgear,
Industrial hydraulic fluids,
Heat transfer fluids,
Extreme pressure lubricants,
Hot melt adhesives,
Chlorinated rubber paints,

and played down suggestions that they are used as media for insecticides and in jet engine lubricants. No reference was made to N.C.R.

Finally Shell made reference to some work to ascertain whether PCB's might arise through degradation of insecticides. The degradants were chemical agents and no success was obtained but it is still possible that some biological process might be able to bring about such transformations.

We were given a brief opportunity to view the Animal Laboratory where much work was in progress on inhalation studies. Long term tests were in progress with benzene vapour on human volunteers, 8 hours a day with full monitoring of all appropriate factors. This is likely to become the standard reference for safety in factor operations.



STLCOPCB4027201

C. Discussions with Representatives of the Government Laboratory at Monsanto House
Thursday, May 1st, 1969.

Mr. J.O'G. Tatton - Laboratory of the Government Chemist
Mr. G.B. Collins - " " " "

Dr. R.E. Keller - MC
Dr. E.P. Wheeler - MC
Dr. D.V.N. Hardy - MCL

Mr. Tatton said that the possible danger of Aroclors used as plasticizer (in food packaging and in lacquers for cans) had been considered quite early on and reference is made thereto in the Annual Report of the Government Chemist 1953-4. He will send me a photostat copy. As regards residues in wild life peaks not due to pesticides were observed in 1963-4 and this caused trouble in view of the complicating effect on insecticide determination. Attempts to identify the disturbing material failed until Jensen showed their nature. Jensen's work has been fully confirmed and extended and PCB's have been found in eggs, predatory birds, rarely in human food, and traces in cod liver oil. Human fat seems to be free from PCB but there may be 2 - 12 parts/$10^6$ of insecticide. Some PCB found in human lung samples. Work has been carried out on air and on circumstantial evidence PCB must be present. 1 part DDT compounds/$10^{12}$ parts of air has been found. The analysis is complicated by the presence of hydrocarbons. Tatton will supply references to this work which was carried out on sample of about 1200 litres. The content of insecticides in air and water is variable, generally higher in London due to soot particles acting as absorbent, and also depending on when spraying is in progress. Dr. Kroll (Water Research Association) has failed to find PCB in river waters at parts per $10^9$.

Many samples are sent to the Government Laboratory by the Nature Conservancy, but having carried out the analysis nothing more can be done as the source and significance of the sample are known only to the Nature Conservancy.

The peaks seen in the chromatogram represent only part of those observed in commercial samples of Aroclor 1254. Probably the middle components are favoured by the analytical procedure: certainly peaks due to higher boiling material can be obtained by raising the operating temperature of the column.

Recently high PCB figures have been found on herons (several hundred parts per million in the liver), kestrels, owls



STLCOPCB4027202

and kingfishers. Dr. N.W. Moore (Monkswood Experimental Station) might be prepared to discuss their significance and the geographical factor. Tatton considers that Jensen's peaks are more in line with Bayer's Clophen. He thinks that some of the bird samples are representative as they were taken from colonies.

Tatton also referred to possible traces of PCB nature in occasional food samples, e.g. milk, mutton and beef fat. He will supply me with copies of all relevant papers, including a report on some new detectors for VPC analysis. Dr. Keller will reciprocate by sending chromatographic curves for the various Aroclors.

I referred to MCL work now starting on the biodegradation of Aroclors and suggested that our workers should contact him on the analytical techniques. He would be most happy to do so.

D. Discussion with Representatives of Cremer & Warner, Consultants, 140 Buckingham Palace Road, London, S.W.1. Thursday, May 1st, 1969.

| | | |
|---|---|---|
| Sir Frederick Warner | - | Cremer & Warner |
| Dr. David Train | - | " " |
| Dr. R.E. Keller | - | MC |
| Dr. E.P. Wheeler | - | MC |
| Dr. D.V.N. Hardy | - | MCL |

This was a lunch discussion arranged to determine the possible value of this firm of consultants in the assessment of the PCB problem.

Cremer & Warner have contracted with the Port of London Authority (and possibly with others too) to study the effect of certain contaminants in the Thames. They are concerned specifically with detergents and Mr. Cremer is Chairman of the Government Committee which has been advising on the problem due to hard detergents. Cremer & Warner are also advising on atmospheric pollution due to sulphur dioxide and smoke, and on the design and interpretation of data from the recording stations.

They have also acted as consultants for the new potash project in relation to the effluent disposal problem. They have advised that the effluent should be discharged 1 mile from the shore at a minimum depth of 75 feet.



STLCOPCB4027203

They were interested broadly in our problem and fully appreciated that after all the analytical work had been done it would be necessary to interpret the data and to correlate it to safety standards in a way which would carry conviction with the authorities. They seemed confident that this area was one of their strong suits, but it is far too early to assess their ability to contribute to the present problem.

D.V.N. HARDY

JWB/HMH
6.5.69.

Footnote (See p.4 - Summary, Item 3)

Compare Jensen's findings that no contamination was observed prior to 1944.



STLCOPCB4027204

PUBLIC RELATIONS DEPARTMENT

PRESS QUERY

(xxx) Divisional
( ) Financial
( ) Corporate

To: St. Louis Bulletin Boards
New York Public Relations
Lloyd Cole
Howard Minckler
Howard Bergren
Elmer Wheeler
Jim Springgate
Bill Richard/Bob Keller

Date: March 6, 1969 Time:

QUERIED BY (Reporter and Media)

Bob Sanford, St. Louis correspondent for TIME

QUERY

TIME's science section is doing story on alleged contamination of wildlife by polychlorinated ~~poly~~biphenyls. Story based on work of Dr. Risebrough at University of California which appeared in NATURE, a U. K. publication.

Q. Doesn't Monsanto produce chlorinated polybiphenyls?

A. Yes.

Q. Aren't you the only U. S. producer?

A. Yes. There are other producers around the world.

Q. Can you give me Monsanto's position on this matter?

A. (Sent him company statement dated March 3, 1969.)

Q. (After receiving material.) Can you better describe use of PCB in "plastic-type" applications?

A. PCB used as a modifier or plasticizer in several areas. One is rubber-based adhesives used for such items as industrial tapes. A second is surface coatings for specialized uses such as traffic paints and concrete curing. A third is in elastomaric sealants for construction and automotive uses.

Q. Is PCB used in automotive tires or vinyl plastic?

A. To our knowledge, no.

Q. Any other comments on Risebrough's work?

A. Looking at his report in NATURE, we have some reservation about the absolute identification of the materials he worked on. Gas chromatography alone will not give absolute identification. A mass spectrometer or other means would be needed for absolute identification of components.

SOURCE OF ANSWER (List All Sources)

Howard Bergren
Bob Keller
Cumming Paton

NAME (Staffer Handling Query)

Tom Ford

TRAN 058656

DEPOSITION EXHIBIT
PIFs 290
6-11-97

STLCOPCB4027205

ROUGH DRAFT

R. E. Keller to ( ) R. Richard per phone by ( ) Stanley - 2/28/69

Publications in hand (1,2) show that Risebrough has not made a positive structure identification to presence of chlorinated hydrocarbons (Biphenyls) let alone Aroclor products in material he has worked with. His approach is not an absolute identification procedure. It's based on similar retention times for 10 components eluted through a gas chromatography column. This measurement is not specific for chemical compounds in unknown mixtures. Furthermore, the electron capture detector used to monitor separated component is not specific for chlorine or even halogens. Work in our labs shows at least 13 components in Aroclor 1242, 17 in Aroclor 1254 and 14 in Aroclor 1260 with different retention times than those reported by Risebrough. More information is needed about un-reported experimental chromatographic conditions used by Risebrough to resolve differences between labs.

Risebrough has based his conclusions implicating chlorinated biphenyls on work by Holmes (3), Holden (3a) Widmark (4), and Jensen (5). Holmes and Holt obtained similar gas chromatography containing 11,13 and 7 peaks respectively for commercial polychlorobiphenyl resin. Holmes and Holden also did not make a positive structure identification.

Widmark and Jensen in Europe (Sweden) obtained a similar gas chromatography for eagle extracts. Chromatographic fractions from this work were identified structurally by Mass Spec as higher chlorinated biphenyls. Risebrough, in his reported work has apparently made no positive identification of this type. His implication of chlorinated biphenyls appears to be by reference back to above work through similar chromatograms. To the best of our knowledge, no one has identified components structurally in biological systems as chlorinated biphenyls in U.S.A. Work is underway in our labs on an unequivocal structure identification of unknown organic species in national products using gas chromatography and mass spectroscopy techniques.

REFERENCES:

(1) Risebrough , R. W., Chem. Fall Out, First Rochester Conference on Toxicity, in the ~~Press (1968)~~

(2) Risebrough, R. W. Nature, Draft of Paper in Press

TRAN 007918

DEPOSITION EXHIBIT
Plfs 291
6-11-92 889

STLCOPCB4027206

ROUGH DRAFT

REFERENCES - Cont'd.

(3) Holmes, D. C. & Simmons, J.H., Tatton, J., Nature 216, 227 (1967).

(3a) Holden, A.V. and Marsden, K., Nature 216, 1274 (1967)

(4) Widmark, G., J. Assoc. Off., Anal. Chem. 50, 1069 (1967)

(5) Jensen, S., New Scientist, 32, 612 (1966)

from R. E. Keller per phone
to M. Stanley
2/28/69



STLCOPCB4027207

W. R. Richard - Research Center

March 6, 1969

SUBJECT: AROCLOR WILDLIFE ACCUSATIONS

cc: H. Bergen HBERG
J. Springate JSPRI
W. Schalk WSCHA
D. Olson DOLSO
R. Kelly RKELL
J. Garrett JGARR
P. Hodges PHODG
P. Park PPARK
R. Keller JFQ
E. Tucker JFQ

TO : E. Wheeler - EWHEE

Risebrough in a recent paper "Nature", Vol. 220, Dec. 14, 1968, has attacked chlorinated biphenyls in three ways:

(1) a pollutant - widely spread by air-water; therefore an uncontrollable pollutant.

(2) a toxic substance - with no permissible allowable levels causing extinction of peregrine falcon by induced hepatic enzymes which degrade steroids upsetting Ca metabolism leading to reproductive weakness, presumably through thinner egg shells.

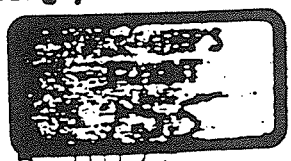
(3) a toxic substance endangering man himself; implying that the peregrine falcon is a leading indicator of things to come.

As outlined in Science, Vol. 163, Pg. 548, Environmental Defense Fund (EDF) is attempting to write new legal precedents in conservation law by hearings and court action. In the Wisconsin case, water quality standards are at issue. "A substance shall be regarded as a pollutant if its use results in public health problems or in acute or chronic (injury) to animal, plant or aquatic life". Wisconsin is one of 7 states which now have federally approved water quality standards. According to Bern Wright, acting chief of the Federal Water Pollution Control Administration's Water Quality Standards Branch, DDT would fit the definition of a pollutant upon a showing that it is harmful to aquatic life.

These people in EDF are saying we must not put stress on any living thing through a change in air or water environment. Eagles, plant life, anything which lives or breathes. This group is pushing hard on the extension of the word harmful. They claim "enzyme inducer" activity is the real threat of DDT and PCB's and are using these arguments to prove that very small amounts of chlorinated hydrocarbons are "harmful".

Monsanto is preparing to challenge certain aspects of this problem but we are not prepared to defend against all of the accusations.

(a) Monsanto is preparing itself to identify trace ppb quantities of chlorinated biphenyls in water samples, in concentrated collected air samples, and in animal tissues. We will know whether we have been falsely identified and accused or not. We will eventually know where any pollution is taking place and the extent of the pollution.



DEPOSITION EXHIBIT Plf's 292 6-11-92

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STLCOPCB4027208

(b) We are not prepared to defend ourselves against the accusations made of enzyme and hormone activity, the isolation of enzymes or metabolic products, the indirect accusation of cancer, or the splitting of genes, when this accusation is made. Whether we can defend this route or not needs further discussion.

(c) Through the Industrial Bio-Test program we are to establish the long term allowable limits of chlorinated biphenyls for certain birds-fish-animals by feeding experiments, pathological examination, and tissue analysis for chlorinated biphenyls. We may be able to answer reproductive ability in some animals.

DDT has been under attack for some years because of its chlorine content, its persistent ability to be identified, and the wildlife problems attributed to it. We will still be under the same attack by the mechanisms listed in (b) even though we might establish safe operating limits for humans and certain animals.

Where does this leave us?

Under identification and control of exposure - we will be able to identify and analyze residues as well or better than anyone in the world. We will probably find residues other than DDT and PCB's. We will probably wind up sharing the blame in the ppm to ppb concentration level.

We can take steps to minimize pollution from our own chlorinated biphenyl plants, we can work with our larger customers to minimize pollution, we can continue to set up disposal and reclaim operations. We can work for minimum exposure in manufacture and disposal of capacitors, transformers and heat transfer systems, and minimize losses for large hydraulic users.

But, we can't easily control hydraulic fluid losses in small plants. It will be still more difficult to control other end uses such as cutting oils, adhesives, plastics and NCR paper. In these applications exposure to consumers is greater and the disposal problem becomes complex. If chlorinated biphenyl is shown to have some long term enzyme or hormone activity in the ppm range, the applications with consumer exposure would cause difficulty.

Risebrough has taken known Aroclor samples and claims to have evidence of enzyme and hormone change. Here there is no question of identification. Either his position is attacked and discounted or we will eventually have to withdraw product from end uses which have exposure problems. Since Risebrough's paper in "Nature", Dec. 1968 has just been published, it is timely, perhaps imperative, that this paper and its implications be discussed with certain customers. This is a rough one because it could mean loss of business on empty and false claims by Risebrough.

Well prepared discussions with Ind. Bio-Test, Monsanto biochemists, the medical and legal departments must take place now. The

T 091773

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position of DDT manufacturers should be determined as a guide. We are being accused of the same things attributed to DDT.

I have written this memo to clarify some of the issues. May I please have comments.

Thanks,

W. R. Richard

ms

Att.

T 091774

B0003393

5 0078

STLCOPCB4027210

~~R. L. Keller~~

DEPOSITION EXHIBIT
PFE 293
6-11-92

Aroclor - Wildlife Meeting 8-25-69

Elmer started the meeting by discussing the recent inquires about the PCB problem. He began with the current WARF study of Lake Michigan and the fact that PCB(s) were found in 9 of 80 water samples so far analyzed. This study is actually concerned with the sources of DDT, Endrin, & Dieldrin found in L. Mich.. Samples (500) of sediment, water, plankton, and fish are being taken from the estuaries around the lake. PCB(s) have been found mostly in city sewer outlets. I believe he has committed us to analyzing some of these samples. The following items were next (memos included):

1. Chlorinated Biphenyl Toxicity - Japanese Kane - Chlor food contamination
   - Suggested an end to our Therminol usage
2. Memo - from Utah State Univ.
3. Press releases and Elmer's attendance at Oregon meeting
4. My memos 8-18-69 & 8-20-69
5. Presence of PCB in dishwasher detergents and samples from Inorg.

Remainder of the meeting dealt with what the problem was, what we should do, and how it should be done.

I think everyone there came to the conclusion that we should increase our efforts in the following areas:

TRAN 058726

STLCOPCB4027211

Biological tests
Control of contamination sources
Alternate products

It was decided that a committee should be appointed to put an overall program on paper.

Wheeler - Chairman
Farran
Richards
Hodges

The proposed program is to be completed by Sept. 15th at which time a meeting will be held to review it.

It was also mentioned that Swanson (Org.) would do biodegradation studies if we will do the analytical work. It was made known that more man power and equipment would be necessary to support this all out effort.

An additional remark was made that the charges for such work should not be against Res. or SEC accounts but from a separate account set up specifically for this project.

It was felt that the necessary funding for the analytical work will be budgeted. Because of this we should probably — outline a proposed budget (manpower and equipment requirements), for eventual approval, of our segment of the project.

TRAN 058727

Monsanto

FROM (NAME & LOCATION): B2SH Cumming Paton

DATE: September 27, 1971

SUBJECT: MCS 1109

REFERENCE:

cc: B2NK W. B. Papageorge
B2SL T. L. Gossage
B3NE W. S. Clark/R. M. Dodd
B2SH D. R. Pogue

TO : T3A
W. R. RICHARD

Following my meeting with Ciba-Geigy on September 24, we need some ammunition very quickly if MCS 1109 is going to be saved.

1. In our many analyses of samples containing PCBs have we ever positively identified GLC peaks belonging to mono- and di-chlorobiphenyl. If they are absent this would be good evidence in favor of MCS 1109 I believe. There is a good chance they may be absent from the flotsam and jetsam that have picked up PCBs in the past few years because of the approximate PCB distribution of Aroclor 1242, 1254, etc.

| | Aroclor 1242 | Aroclor 1254 |
|---|---|---|
| 0 (biphenyl) | 0.05 | 0.05 |
| 1 | 1-2 | 0.05 |
| 2 | 20-25 | <1.0 |
| 3 | 40-50 | <1.0 |
| 4 | 20-25 | 20-25 |
| 5 | 5-10 | 40-50 |
| 6 | <1.0 | 20-25 |
| 7 | - | up to 10 |
| 8 | - | - |

(I think it might be a good thing when our lab identify PCBs quantitatively in a sample if they gave the breakdown as % di-; tri-; tetra- etc.)

2. Ciba-Geigy listed the following information that is needed to give a product clearance re "environmental pollution".

LD-50
BOD and COD 5
Feeding studies including fish (96 hours for fish)
Organ accumulation as a result of feeding.
Chronic studies on oral feeding
Inhalation tests
Dermal testing
Eye effects
Carcinogenic effects

TRAN 044212

DEPOSITION EXHIBIT
PHS 294
6-11-92 989

STLCOPCB4027213

E. Scott Tucker - R&D Laboratories - 1700 So. Second

February 25, 1969

AROCLOR - WILDLIFE

R. E. Keller - So. Second
J. T. Garrett - GO
P. D. Hodges - GO

TO W. R. Richard/D. P. Wheeler
General Offices

On 9-6-68 four samples, two sediment and two water, from Snow Creek were received for Aroclor analysis. The water and sediment were sampled on 8-27-68 at the plant out fall and approximately one block below the plant out fall. The results of the analysis are as follows:

| | Amount Aroclor Found | |
|---|---|---|
| Sample Designation | Sediment | Water |
| Plant Out fall | 0.20±0.02% | 58±2 ppb** |
| Plant Out fall (1 blk) | 1.64±0.04% | - * |

*Sample broken in transit.
**No less than this amount present.

The amount of Aroclor found was calculated as Aroclor 1242. The EC/GC traces of the sediment were unmistakably Aroclor 1242. The water extracts appeared to contain only the lighter chlorinated species along with a number of other electron capture active materials. For this reason a "no less than" value is stated, more quantitative results will have to await the development of more specific separation techniques.

Bill, on the basis of this information, I think that if the PCB accusation turns out to be true and the government people become more active, we should plan on an extensive evaluation of the extent of contamination in this area. For example, samples should be taken further down Snow Creek, down Choccolocco Creek and even from the Coosa River if necessary.

You might check, but I think the lab there has the necessary equipment and could carry out this study.

E. Scott Tucker

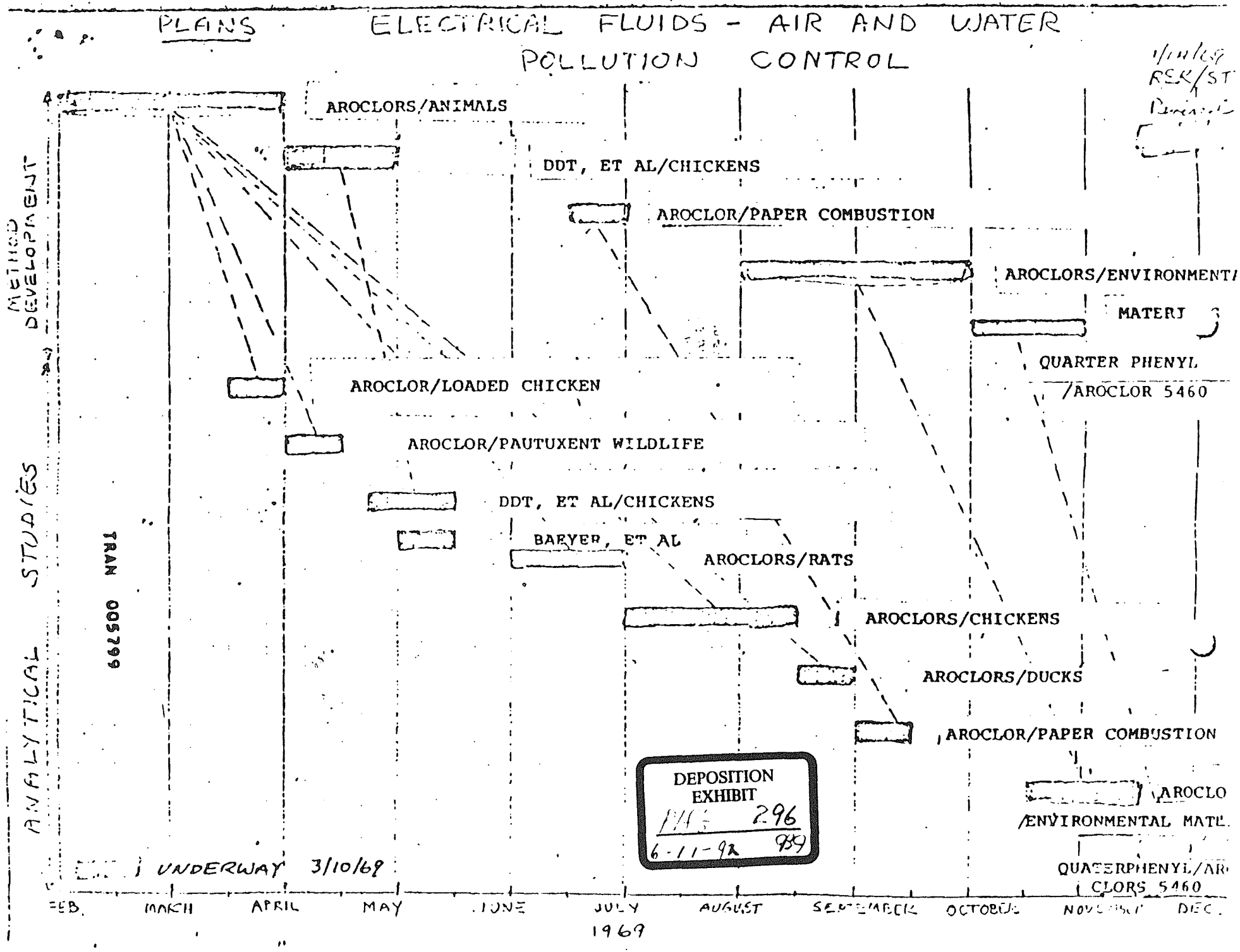
db

TRAN 009858

21

DEPOSITION EXHIBIT
PHS 295
6-11-92

STLCOPCB4027214




STLCOPCB4027215

## ELECTRICAL FLUIDS - AIR AND WATER POLLUTION CONTROL

### FLU-030 PLANS - METHOD DEVELOPMENT

| Priority | Product | Job/Project | Justification | Responsibility | Man Weeks | Target Date |
|---|---|---|---|---|---|---|
| 1 | Aroclor<br>1242<br>1254<br>1260 | Develop sensitive (ppb) GC/EC method for Aroclor products in<br>1. Fat<br>2. Liver<br>3. Kidney<br>4. Muscle<br>5. Urine<br>6. Feces<br>from rats, chickens and ducks. | Provide backup data needed for toxicity studies by Calandra to show biological fate of Aroclors. | Applied Sciences/ Medical Dept. | 8 | 4/1/69 |
| 2 | DDT<br>Toxaphene<br>Chlorinated Napthalenes | Develop sensitive GC/EC method for these components in chickens. | Show biological fate of these products which may be source of chlorinated hydrocarbons found in environmental materials | Applied Sciences/ Medical Dept. | 4 | 5/1/69 |
| 3 | Aroclor<br>1242 | Develop technique for identification and measurement of combustion products simulating the burning of NCR paper. | Demonstrate whether incineration is a source of environmental contamination. | Applied Sciences | 2 | 7/1/69 |
| 4 | Aroclor<br>1242<br>1254<br>1260 | Develop sensitive (ppb) GC/EC, GC/microcoulometer and GC/Mass methods for Aroclor products in environmental<br>1. Water<br>2. Soil<br>3. Wildlife | Determine environmental contamination to protect product position with the customer. Determine Aroclor solubility in water perse. Verify Patuxent/ Risebrough PCB in wildlife extracts. | Applied Sciences | 8 | 10/1/69 |

TRAN 005800

STLCOPCB4027216

| | | | | | | |
|---|---|---|---|---|---|---|
| 3 | Aroclor 5460 | Develop method for quarter-phenyls in terphenyl products. | Provide data possibly needed by regulatory agencies for clearance of product. | Applied Sciences | 4+ | 11/1/69 |
| - | Aroclors | Evaluate Berozas' carbon skeletal analyzer in conjuction with GC | Provide more specific/ accurate method for mixtures of the Aroclor type. | Applied Sciences | 2 | - |

REK/ST
1-15-69

TRAN 005801

STLCOPCB4027217

ELECTRICAL FLUIDS - AIR AND WATER POLLUTION CONTROL

FLU-030 PLANS - ANALYTICAL STUDIES

| Priority | Product | Job/Project | Justification | Responsibility | Man Weeks | Target Date |
|---|---|---|---|---|---|---|
| 1 | Aroclor 1242 1254 1260 5460 | Analysis of Aroclor Loaded Chicken Wheeler 12/9/68 Minutes | Provide backup data needed for toxicity studies by Calandra to show biological fate of Aroclors | Applied Science/? | 1 | 4/1/69 |
| 2 *2a | Aroclor | Analyze wildlife extracts from Pautuxent/Risebrough | Verify PCB reported in wildlife extracts | Applied Sciences | 2 | 4/1/569 |
| 3 | DDT Toxaphene Chlorinated Napthalenes | Analyze fat, liver, kidney and muscle from chickens fed these materials. | Show biological fate of these products which may be source of chlorinated hydrocarbons found in environmental studies. | Applied Sciences/ Medical Dept. | 3 | 5/15/69 |
| 4 | Aroclor 1242 1254 1260 5460 | Analysis of Rat Tissue/Excretia - Calandra 9/9/68 Biotest Protocol | Provide backup data needed for toxicity studies by Calandra to show biological fate of Aroclors | Applied Science/? | 4 | 7/1/69 |
| 5 | " | Analysis of Chicken Tissue/Excretia Calandra 9/9/68 Biotest Protocol | " | " | 6 | 8/15/69 |
| 6 | " | Analysis of Duck Tissue/Excretia Calandra 9/9/68 Biotest Protocol | " | " | 2 | 9/1/69 |
| *2a | Baeyer, et al | Detn composition of competitive products and relate to Monsanto products | To seek financial participation of competitors in long range studies | " | 2 | 4/15/69 |

TRAN 005802

STLCOPCB4027218

| | | | | | | |
|---|---|---|---|---|---|---|
| 7 | Aroclor 1242 | Determine combustion products under conditions simulating the burning of NCR paper | Demonstrate whether incineration is a source of environmental contamination. | Applied Sciences | 2 | 9/15/69 |
| 8 | Aroclor 1242 1248 1254 | Analyze environmental materials from manufacturing sites | Show the extent of environmental contamination | Applied Sciences | 4+ | 11/15/69 |
| 9 | Aroclor 5460 | Analyze plant products for quarterphenyl | Provide data for regulatory agencies for clearance of product | Applied Sciences | 2 | 12/1/69 |

REK/ST
1-15-69

TRAN 005803

STLCOPCB4027219

Monsanto

FROM (NAME & LOCATION) E. S. Tucker - R&D Laboratories - South Second Street

DATE September 25, 1969

cc M. W. Farrar - Res. 1
W. R. Richard - GO
E. P. Wheeler - GO

SUBJECT WISCONSIN ALUMINI RESEARCH FOUNDATION INSTITUTE TRIP REPORT - SEPTEMBER 24, 1969

REFERENCE

TO R. E. Keller - Res. 1

A review and comparison of the PCB analytical methodology developed here and independently in the WARF laboratories was held with F. Coon (director, chemical analysis section), J. Birdsall (project coordinator), and D. Hughes (analyst) in Madison, Wisconsin at their research laboratories. Their procedures, with minor exceptions, were an exact duplication of ours and as such should produce equivalent results.

We, therefore, agreed to exchange a limited number of Lake Michigan samples and extracts for collaborative verification between the two sites. The exchange samples will be restricted to those producing abnormal electron capture chromatograms which could be misinterpreted unless subjected to GC/Mass verification. They do not presently have this capability although they are certainly thinking along these lines. They were also obviously interested in our help in identifying pollutants other than those already identified in these abnormal samples. Whether or not we share information above and beyond the PCB problem is, of course, open and obviously dependent on what other materials, in addition to PCBs, we actually find in these samples.

They requested "pint bottle" amounts of Aroclor 1242, 1254 and 1260. The reason given for the rather large amounts requested was that they have had many requests for standard samples of these materials which they would like to fill in order to maintain these contacts. While they would rather dispense the Aroclors themselves, they agreed that they could and would refer all requests to us if we felt this was better.

2 pt ea sent 9-3-69 LM J. Miller

This laboratory, in my opinion, is well equipped and competently staffed. In addition to this, I found their attitude toward the PCB pollution problem to be acceptably scientific rather than political in nature.

Additionally, I believe that we should consider using WARF facilities to eliminate some of our current manpower and routine sample backlog problems.

The chromatograms I saw of the Lake Michigan samples they had looked at corresponded to the higher chlorinated biphenyls 1254 and 1260. The samples taken in and around the industrial areas (southern one-third of lake) appeared to contain the higher levels.

TRAN 022055

DEPOSITION EXHIBIT P/f's 297 6-11-92

STLCOPCB4027220

In view of this, our current Pensacola problem, and other published information, I would suggest that we step up our efforts at finding immediate replacement formulations for Pydraul AC and Pydraul 625. I also feel that Aroclor 1242 should not be used in replacement formulations. If this were done, I firmly believe that in a short period of time analysts would soon be reporting Aroclor 1242 as a significant water pollutant thus seriously endangering a market which so far has not been questioned.

E. S. Tucker

db



STLCOPCB4027221

## ELECTRICAL FLUIDS - AIR AND WATER POLLUTION CONTROL

### FLU-030 PLANS - METHOD DEVELOPMENT

| Priority | Product | Job/Project | Justification | Responsibility | Man Weeks | Target Date |
|---|---|---|---|---|---|---|
| 1 | Aroclor 1242 1254 1260 | Develop sensitive (ppb) GC/EC method for Aroclor products in 1. Fat 2. Liver 3. Kidney 4. Muscle 5. Urine 6. Feces from rats, chickens and ducks. | Provide backup data needed for toxicity studies by Calandra to show biological fate of Aroclors. | Applied Sciences/ Medical Dept. | 8 | 4/1/69 |
| 2 | DDT Toxaphene Chlorinated Napthalenes | Develop sensitive GC/EC method for these components in chickens. | Show biological fate of these products which may be source of chlorinated hydrocarbons found in environmental materials | Applied Sciences/ Medical Dept. | 4 | 5/1/69 |
| 3 | Aroclor 1242 | Develop technique for identification and measurement of combustion products simulating the burning of NCR paper. | Demonstrate whether incineration is a source of environmental contamination. | Applied Sciences | 2 | 7/1/69 |
| 4 | Aroclor 1242 1254 1260 | Develop sensitive (ppb) GC/EC, GC/microcoulometer and GC/Mass methods for Aroclor products in environmental 1. Water 2. Soil 3. Wildlife | Determine environmental contamination to protect product position with the customer. Determine Aroclor solubility in water perse. Verify Patuxent/ Risebrough PCB in wildlife extracts. | Applied Sciences | 8 | 10/1/69 |

TRAN 007542



STLCOPCB4027222

| | | | | | | |
|---|---|---|---|---|---|---|
| 5 | Aroclor 5460 | Develop method for quarter-phenyls in terphenyl products. | Provide data possibly needed by regulatory agencies for clearance of product. | Applied Sciences | 4+ | 11/1/ |
| - | Aroclors | Evaluate Berozas' carbon skeletal analyzer in conjuction with GC | Provide more specific/ accurate method for mixtures of the Aroclor type. | Applied Sciences | 2 | - |

REK/ST
1-15-69

TRAN 007543

STLCOPCB4027223

## FLU-030 PLANS - ANALYTICAL STUDIES

| Priority | Product | Job/Project | Justification | Responsibility | Man Weeks | Target Date |
|---|---|---|---|---|---|---|
| 1 | Aroclor 1242 1254 1260 5460 | Analysis of Aroclor Loaded Chicken Wheeler 12/9/68 Minutes | Provide backup data needed for toxicity studies by Calandra to show biological fate of Aroclors | Applied Science/? | 1 | 4/1/69 |
| 2 *2a | Aroclor | Analyze wildlife extracts from Pautuxent/Risebrough | Verify PCB reported in wildlife extracts | Applied Sciences | 2 | 4/1/569 |
| 3 | DDT Toxaphene Chlorinated Napthalenes | Analyze fat, liver, kidney and muscle from chickens fed these materials. | Show biological fate of these products which may be source of chlorinated hydrocarbons found in environmental studies. | Applied Sciences/ Medical Dept. | 3 | 5/15/69 |
| 4 | Aroclor 1242 1254 1260 5460 | Analysis of Rat tissue/Excretia - Calandra 9/9/68 Biotest Protocol | Provide backup data needed for toxicity studies by Calandra to show biological fate of Aroclors | Applied Science/? | 4 | 7/1/69 |
| 5 | " | Analysis of Chicken Tissue/Excretia Calandra 9/9/68 Biotest Protocol | " | " | 6 | 8/15/69 |
| 6 | " | Analysis of Duck Tissue/Excretia Calandra 9/9/68 Biotest Protocol | " | " | 2 | 9/1/69 |
| *2a | Baeyer, et al | Detn composition of competitive products and relate to Monsanto products | To seek financial participation of competitors in long range studies | " | 2 | 4/15/69 |

TRAN 007544

STLCOPCB4027224

| | | | | | | |
|---|---|---|---|---|---|---|
| 7 | Aroclor 1242 | Determine combustion products under conditions simulating the burning of NCR paper | Demonstrate whether incineration is a source of environmental contamination. | Applied Sciences | 2 | 9/15/6 |
| 8 | Aroclor 1242 1248 1254 | Analyze environmental materials from manufacturing sites | Show the extent of environmental contamination | Applied Sciences | 4+ | 11/15/6 |
| 9 | Aroclor 5460 | Analyze plant products for quarterphenyl | Provide data for regulatory agencies for clearance of product | Applied Sciences | 2 | 12/1/69 |

REK/ST
1-15-69

TRAN 007545



STLCOPCB4027225

## ELECTRICAL FLUIDS - AIR AND WATER POLLUTION CONTROL

### FLU-030 PLANS - ANALYTICAL STUDIES

| Priority | Product | Job/Project | Justification | Responsibility | Man Weeks | Target Date |
|---|---|---|---|---|---|---|
| 1 | Aroclor 1242 ~~1248~~ 1254 1260 | Analysis of Aroclor Loaded Chicken Wheeler 12/9/68 Minutes | Provide backup data needed for toxicity studies by Calandra to show biological fate of Aroclors | Applied Science/? | 1 | 4/1/69 |
| 2 ? | Aroclor | Analyse wildlife extracts from Pautuxent/Risebrough | Verify PCB reported in wildlife extracts | Applied Sciences | 2 | 4/1/569 4/15/69 |
| 3 | DDT Toxaphene Chlorinated Napthalenes | Analyse fat, liver, kidney and muscle from chickens fed these materials. | Show biological fate of these products which may be source of chlorinated hydrocarbons found in environmental studies. | Applied Sciences/ Medical Dept. | 3 | 5/15/69 |
| 4 | Aroclor 1242 ~~1248~~ 1254 1260 | Analysis of Rat tissue/Excretia - Calandra 9/9/68 Biotest Protocol | Provide backup data needed for toxicity studies by Calandra to show biological fate of Aroclors | Applied Science/? | 4 | 7/1/69 |
| 5 | " | Analysis of Chicken Tissue/Excretia Calandra 9/9/68 Biotest Protocol | " | " | 6 | 8/15/69 |
| 6 | " | Analysis of Duck Tissue/Excretia Calandra 9/9/68 Biotest Protocol | " | " | 2 | 9/1/69 |

TRAN 007562

STLCOPCB4027226

| | | | | | | |
|---|---|---|---|---|---|---|
| 7 | Aroclor 1242 | Determine combustion products under conditions simulating the burning of NCR paper | Demonstrate whether incineration is a source of environmental contamination. | Applied Sciences | 2 | 9/15/69 |
| 8 | Aroclor 1242 1248 1254 | Analyze environmental materials from manufacturing sites | Show the extent of environmental contamination | Applied Sciences | 4+ | 11/15/69 |
| 9 | Aroclor 5460 | Analyze plant products for quarterphenyl | Provide data for regulatory agencies for clearance of product | Applied Sciences | 2 | 12/1/69 |

REK/ST
1-15-69

TRAN 007563

STLCOPCB4027227

Monsanto

FROM (NAME & LOCATION) E. S. TUCKER - R&D LABORATORIES - SOUTH SECOND STREET

DATE October 8, 1969

SUBJECT ANALYSIS OF ESTUARY MUD SAMPLES FROM THE USKMOUTH AREA (NEWPORT,U.K.)

REFERENCE

cc R. E. Keller - S. 2nd
A. F. Regan - Newport
E. P. Wheeler - GO
M. W. Farrar - S. 2nd
W. R. Richard - GO

TO : H. A. Vodden
Ruabon

Results

| PS Out No. N69/ | Sampled At | ppb Aroclor 1242 Found |
|---|---|---|
| 8006 | Peterstone | 470 |
| 8007 | Lighthouse | 2068 |
| 8008 | Goldcliff Pill | 966 |
| 8009 | Cold Harbor Pill | 665 |

The mud samples were prepared and analyzed in the following manner:

Each mud sample was spread evenly in a flat pyrex baking dish and allowed to air dry. After drying the mud was finely ground and divided into two 250-300g samples. The weighed mud was extracted with acetonitrile and the filtered extracts evaporated to dryness. The residue was saponified by refluxing with alcoholic KOH, re-extracted into hexane, extracted with 90% sulfuric acid, and chromatographed on alumina. After concentration the amount of Aroclor present was determined by electron-capture gas chromatography.

The electron capture fingerpoints were identical with that of our Aroclor 1242 standard. There were also indications of the higher chlorinated biphenyls but at much lower levels.

E. S. Tucker

db
Attachment

TRAN 023503

MS-10 REV. 11-68

DEPOSITION EXHIBIT
PIFs 298
6-12-92 984

STLCOPCB4027229

Monsanto

FROM (NAME & LOCATION): E. S. Tucker - R&D Laboratories - South Second Street

DATE: December 3, 1969

SUBJECT: AROCLOR - WILDLIFE, NCR WATER SAMPLES

REFERENCE:

cc: M. W. Farrar - S. 2nd
R. E. Keller - S. 2nd
W. R. Richard - GO
E. P. Wheeler - GO

TO: C. Paton
J. T. Garrett

**CONFIDENTIAL**

| Sample No. | ppm Aroclor 1242 Found |
|---|---|
| 1 | 1063 |
| 2 | 485 |
| 3 | 96.4 |
| 4 | 27.7 |
| 5 | 9.5 |
| 6 | 20.0 |
| 7 | 6.1 |
| 8 | 1.8 |
| 9 | 6.6 |
| 10 | 1.7 |
| 11 | 2.6 |
| 12 | 2.6 |

The electron capture fingerprints of the extracts from each sample do not show any indication of biodegradation.

The decrease in concentration observed is probably the result of dilution and adsorption of the Aroclor in the original waste water as it moves from the NCR plant to the treatment facilities.

However, it is possible that the high level of "fresh" Aroclor present has completely masked any degradation that might have occurred to Aroclor previously discharged.

E. S. Tucker

db

Enclosure

TRAN 022090

DEPOSITION EXHIBIT
PTS 299
6-12-97

PCB - Company Cooks

E. S. Tucker - R&D Laboratories - South Second Street

January 29, 1970

AROCLOR - WILDLIFE
MONTHLY SUMMARY

cc M. W. Farrar - Res. 1
W. B. Papageorge - GO
W. R. Richard - GO
E. P. Wheeler - GO

TO : R. E. Keller
Research 1

The initial biologically populated river water degradation study on Aroclors 1221, 1242, 1248 and 1254 has been completed. An analysis of the results indicates that under the conditions employed the major isomers in Aroclor 1221 are readily degraded (Figures I and II). Aroclors 1242, 1248 and 1254 remained unaffected after an exposure period of five weeks. The samples from the last exposure period (8 weeks) are presently in the process of being analyzed. A report will be issued when the analysis of these samples has been completed.

The semi-continuous activated sludge degradation studies have so far confirmed the river water results in that the major isomers of ARoclor 1221 are readily degraded. Additionally, there are indications that some of the lower chlorinated isomers in Aroclors 1242 and 1248 are beginning to be effected.

A review of the current Bio-Test toxicity results was held with our Medical Department to determine if the amount of residue support analysis could be trimmed still further. It was agreed by all present that the amount of planned analytical work could not be cut any further. Arrangements are being made with Bio-Test to have selected tissues presently in storage, shipped to Monsanto. It is hoped that we will be well into these studies by the end of next month.

Packed column electron capture gas chromatographic fingerprints of competitive PCB products (Electrophenyl, Phenoclor and Clophen) were not startlingly different from those obtained for the equivalent Aroclors. A special study report will be issued for the record.

An informal 2-day review of the Aroclor - Wildlife problem was held with corporate and divisional representatives.

FUTURE PLANS

Analysis of Bio-Test samples.

DEPOSITION EXHIBIT
Papageorge - BMC
8-28-81
26-1D

C001248

DEPOSITION EXHIBIT
PIFS 300
6-12-82

STLCOPCB4027231

Analysis of remaining detergent components.

Investigation of alternate chemical oxidation techniques for isolating and identifying PCBs.

Completion of S.C.O.T./EC/GC study.

Analysis of Gainesville, Florida Aroclor samples.

E. S. Tucker

J. Litschgi

B. J. Westenberger

db

0001249

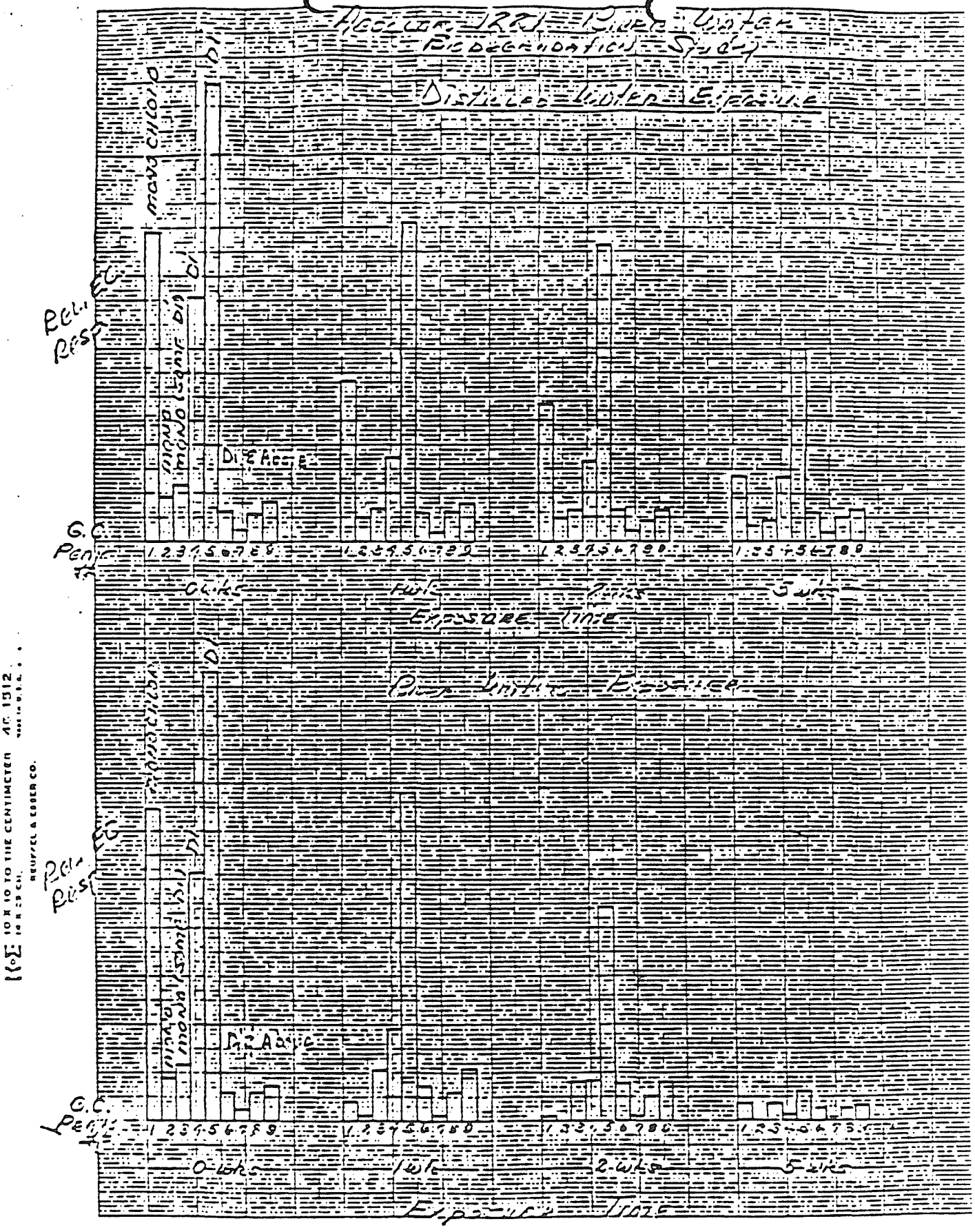



FIGURE I

C001250

STLCOPCB4027233

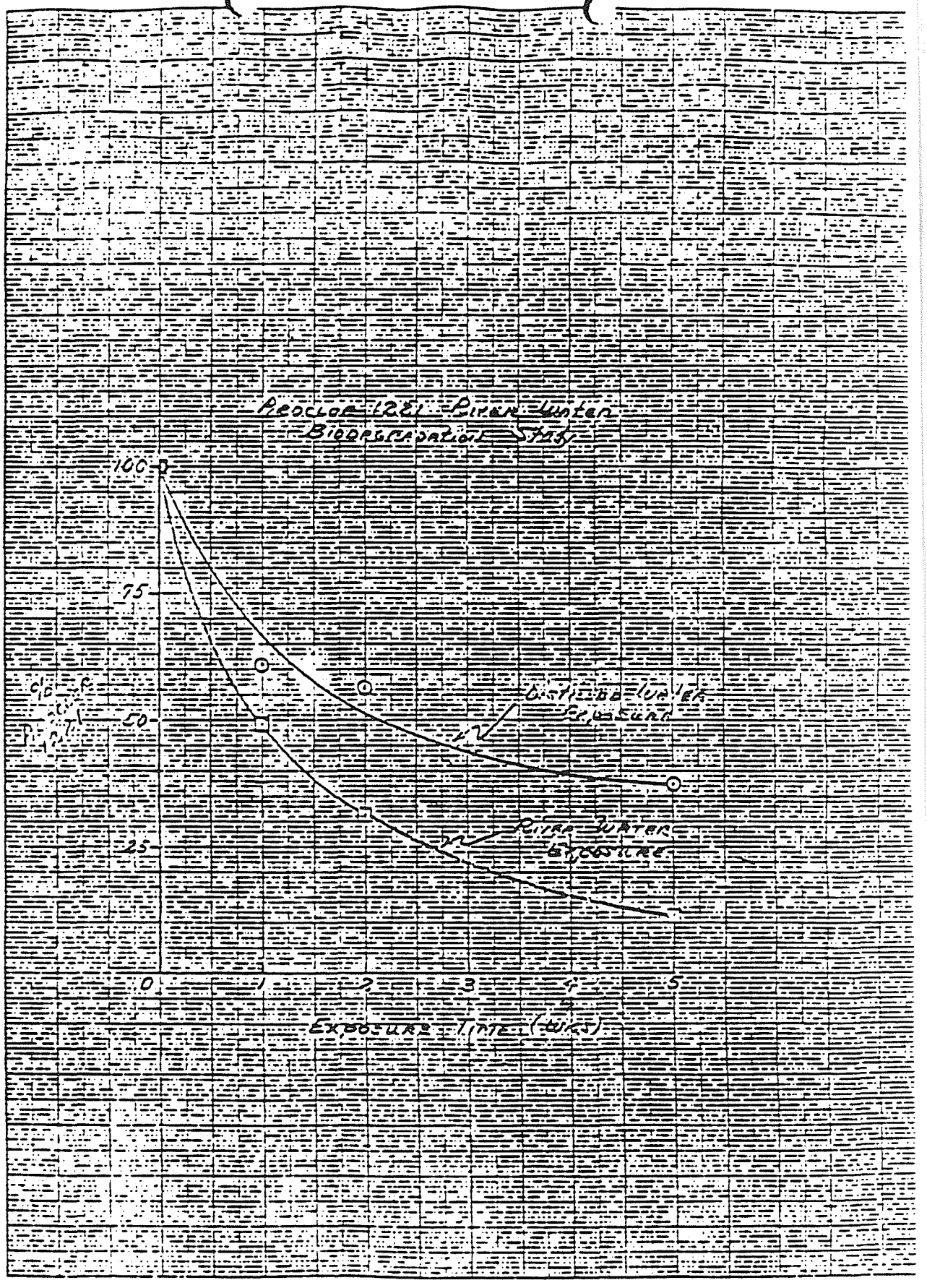
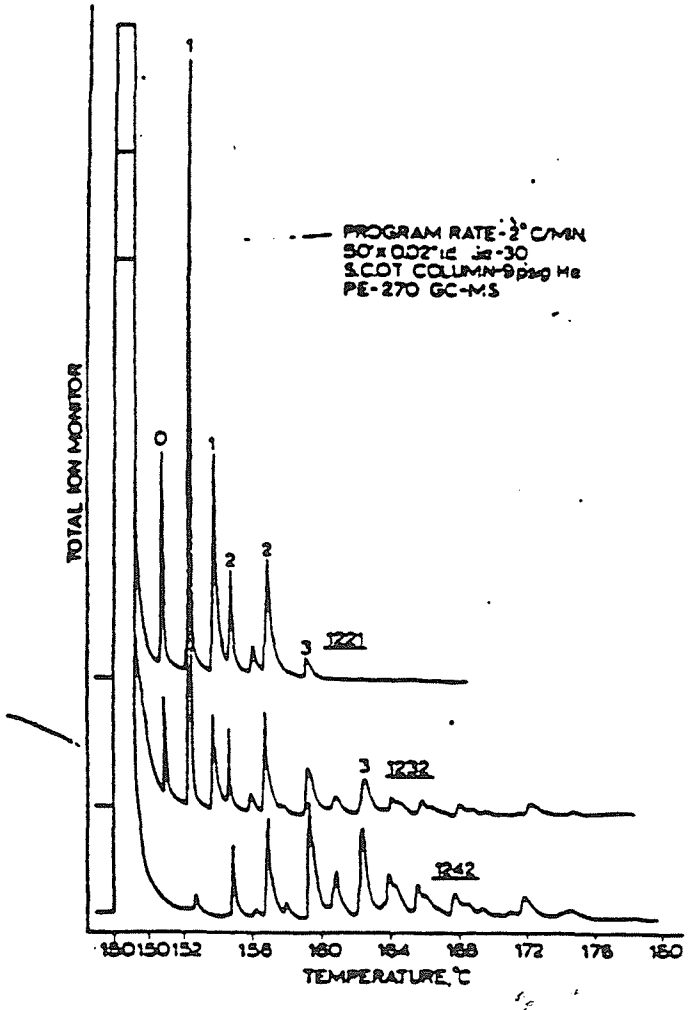



FIGURE 11

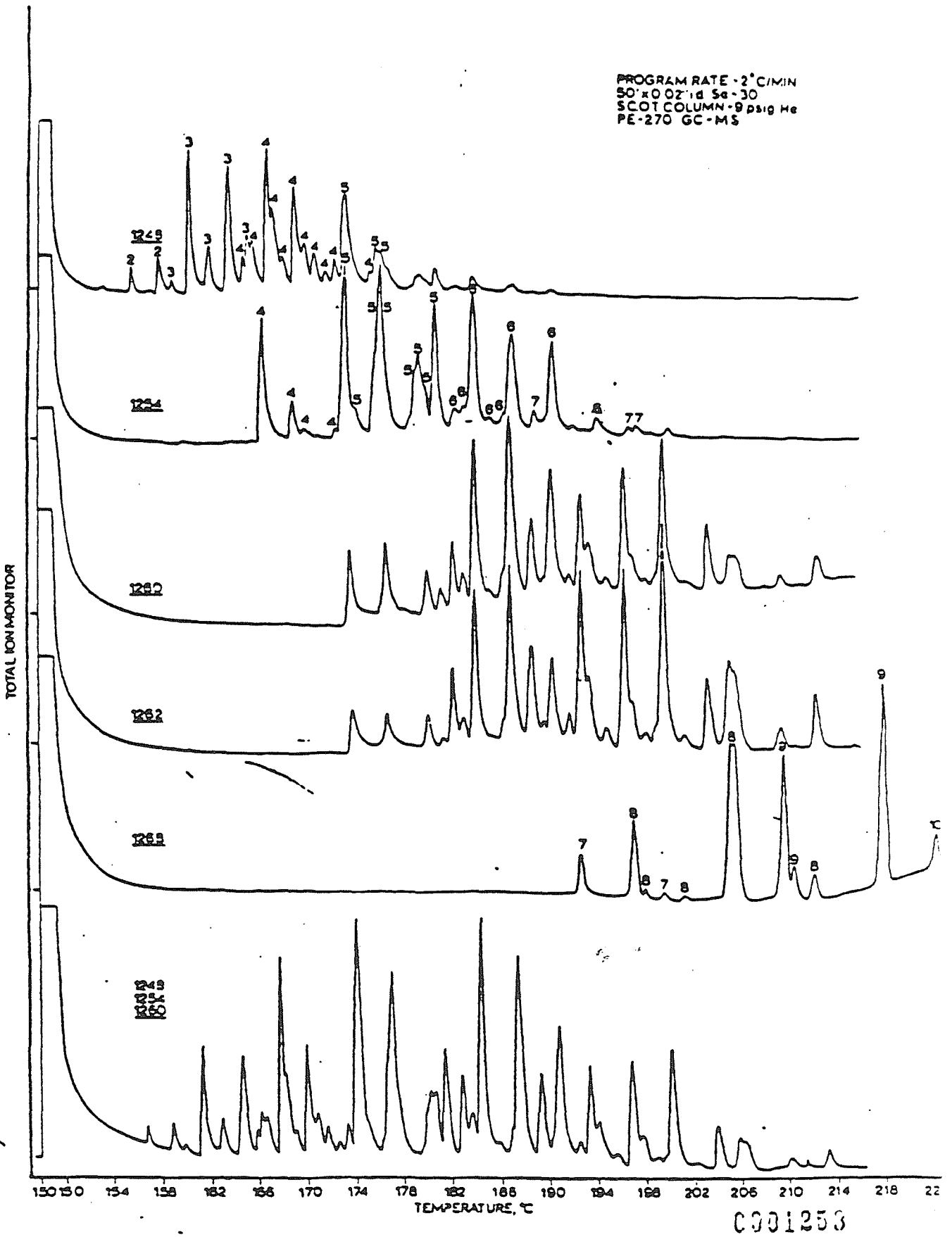
0001251

STLCOPCB4027234

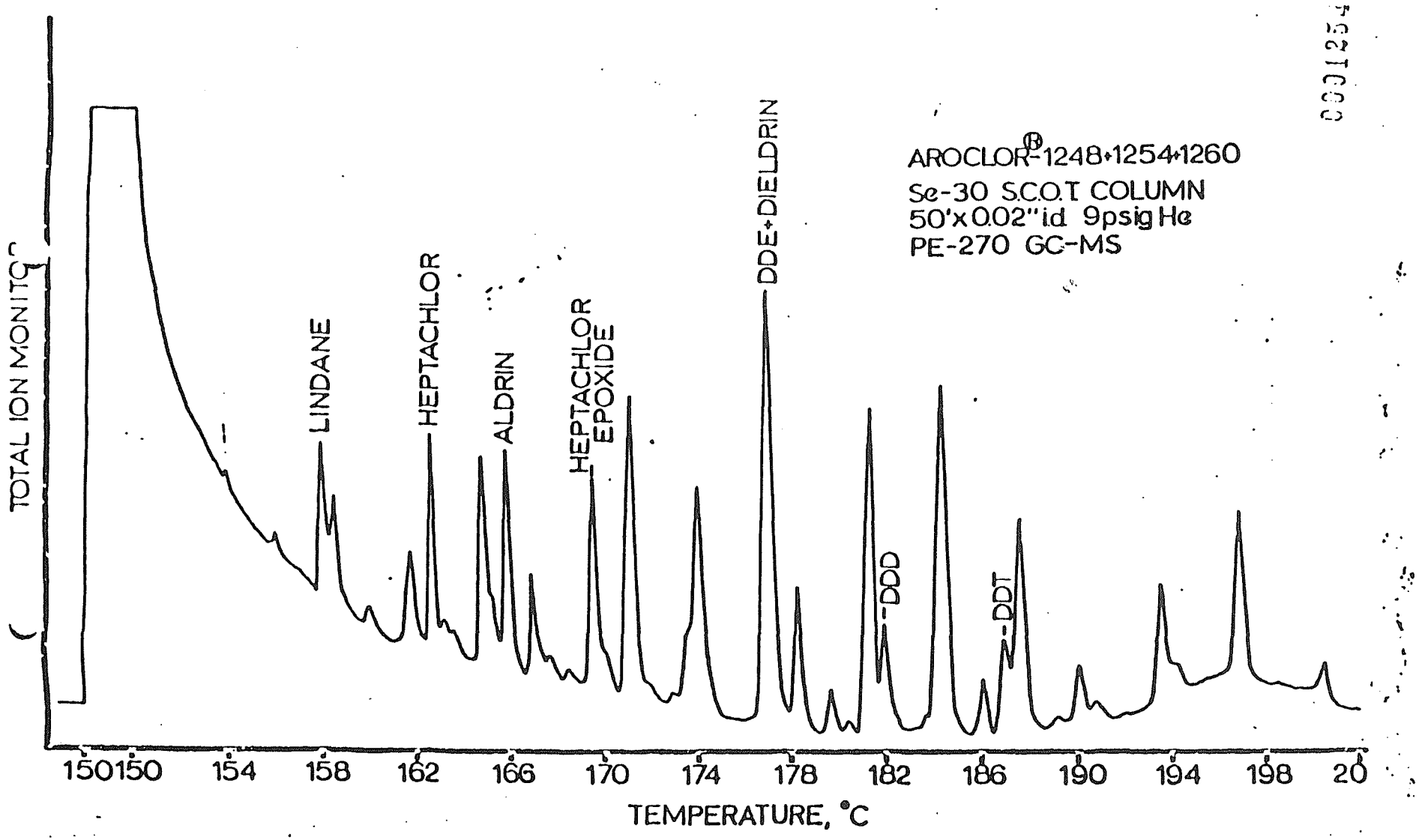
PCB Analytical



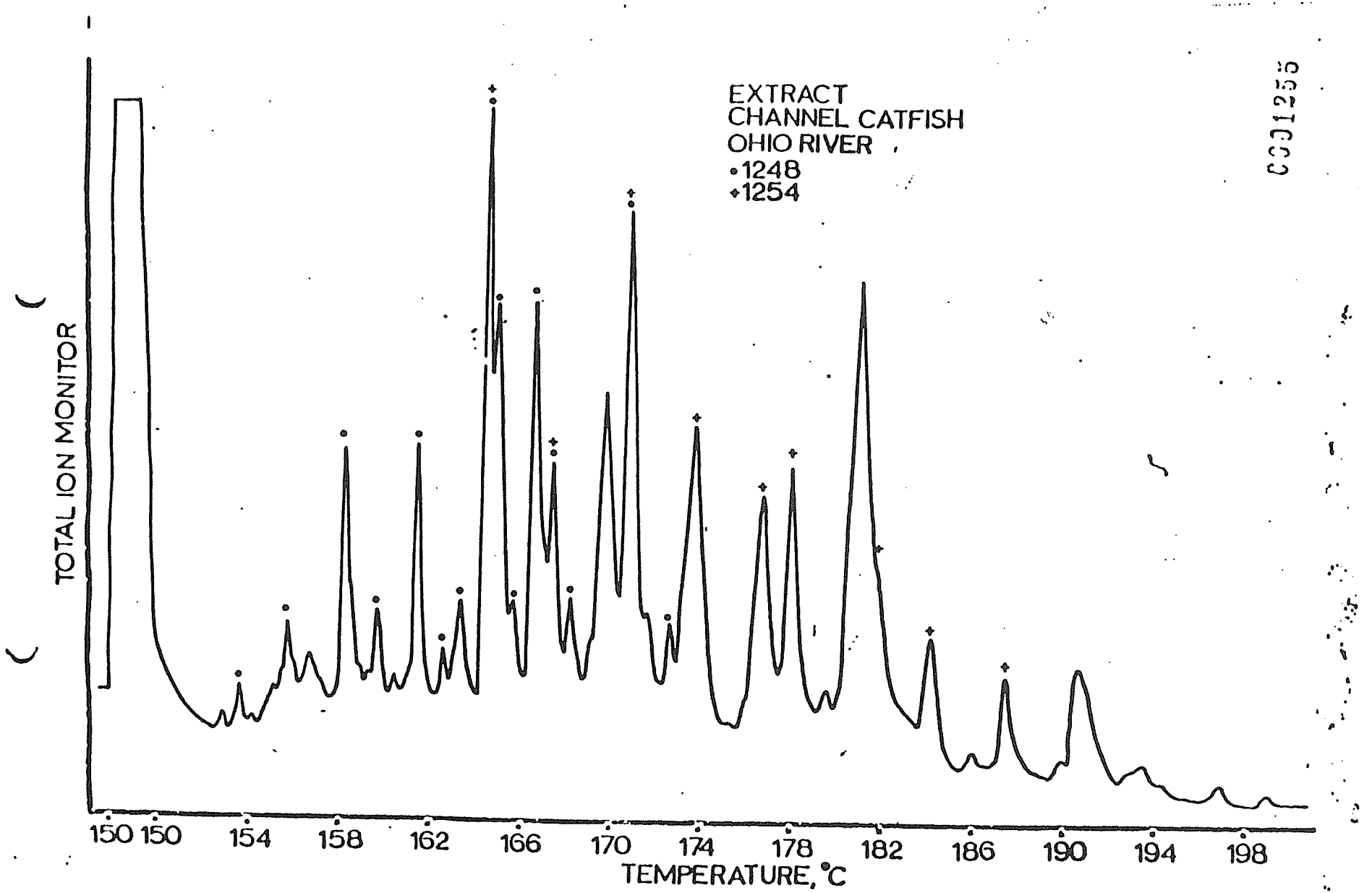

0001252


PROGRAM RATE - 2°C/MIN
50' x 0.02" id SE - 30
SCOT COLUMN - 9 psig He
PE-270 GC-MS
1249
1254
1262
1262
1269
1249
1254
1260
TOTAL ION MONITOR
150 150 154 158 162 166 170 174 178 182 186 190 194 198 202 206 210 214 218 22
TEMPERATURE, °C

C001253




STLCOPCB4027237




STLCOPCB4027238

**Monsanto**

JUL 2 1970

FROM (NAME & LOCATION) W. B. PAPAGEORGE - ST. LOUIS

DATE JULY 17, 1970

SUBJECT

REFERENCE

TO H. S. BERGEN

cc D. S. CAMERON - BRUSSELS
W. S. CLARK
E. M. EMERY - JFQ
M. W. FARRAR - 2ND STREET
R. E. KELLER - 2ND STREET
R. M. KOUNTZ
J. MASON
R. H. MUNCH - 2ND STREET
D. A. OLSON
W. R. RICHARD
J. R. SAVAGE
J. E. SPRINGGATE
E. S. TUCKER - 2ND STREET
H. A. VODDEN - RUABON
E. P. WHEELER

I reviewed our biodegradation studies with R. Munch, R. Kountz, S. Tucker, E. Emery and J. Savage on Thursday, July 16, 1970.

1. Monsanto-USA Studies

River die-away studies indicated Aroclor 1221 disappears, Aroclors 1242, 1248 and 1254 were undegraded.

Semi-continuous activated sludge studies indicate that selective degradation of specific homologs does occur because peaks normally present in Aroclor 1221, 1242 and 1248 disappear.

2. Monsanto-U.K. Studies

Shake flask studies demonstrate that 2,4'-, 2,2,3- and 2,3,4'- isomers degrade readily. 2,2',4,6-, 2,2',4,4', and 4,4' do not degrade.

Ruabon laboratory also demonstrated by peak analysis that MCS-1016 and Aroclor 1130 are more degradable than Aroclor 1242.

3. Environment Samples

Investigators in both Europe and USA find isomers in wildlife that resemble those in Aroclor 1254. Predominant homologs present in Aroclor 1242 are usually not found except close to industrial plants. Since large quantities of Aroclor 1242 are produced and released to the environment, degradation must be occurring.

TRAN 040011

DEPOSITION EXHIBIT
PH's 301
6-12-92

Evidence of degradation has been noted by Monsanto laboratory in studies of foundry plant settling ponds, milk and fish. Peaks which are present in the original Aroclor are modified or missing in the sample extract indicating degradation.

4. MCS-1016

Considerable discussion was held on what is meant by "biodegradable" when describing MCS-1016. Capacitor customers I visited had the erroneous impression that this meant the material could be introduced into the plant sewer system and would be non-detectable a very short distance downstream. For our purposes, the term "biodegradable" means that peaks which persisted through the biodegradation tests with Aroclor 1242 are significantly reduced in this distilled product. Neither Aroclor 1242 or MCS-1016 can be dumped into the environment. Users of either material must exercise control. In some instances this may require activated sludge treatment units. It is generally agreed that the relative improvement in biodegradability is an advantage which the capacitor industry will find interesting. We must establish if the less controllable hydraulic fluids applications can materially benefit from the use of MCS-1016.

5. Current Work

At Ruabon, work continues on HB-40, Aroclors and Pyralene 1500 (a competitive product). Dr. W. C. Evans, University of North Wales, Bangor is investigating some microorganisms which he believes are effective on PCB's.

E. Emery is completing his studies on HB-40 and MIPB. He will immediately start tests to determine relative per cent degradation of Aroclor 1242, Aroclor 1254, MCS-1016 and Aroclor 1230B. These tests take about 3 weeks to complete and his equipment provides for testing four products at a time. Analytical facilities are manned 7 days per week, 12 hours per day. Equipment needs are being evaluated.



STLCOPCB4027240

The backlog of products to be tested continues to increase. The Applied Sciences Group continually reviews the list to re-establish continuing changes in priorities.

In summary, emphasis on this work has been accelerated within the past three months, the work is progressing as planned and our facilities are being utilized to the practical limit.

W B Papageorge

W. B. PAPAGEORGE

ms

TRAN 040013

STLCOPCB4027241

Monsanto

FROM (NAME & LOCATION) R. E. Keller - Technology Department - South Second Street

DATE May 6, 1971

SUBJECT POLYCHLORINATED BIPHENYL STUDIES

REFERENCE

TO :W. R. Richard
General Offices

CC: H. S. Bergen - GO
W. B. Papageorge - GO
E. P. Wheeler - GO
R. H. Munch - Res. 2
T. M. Patrick - Res. 1
C. W. Roos - Res. 1
R. A. Baxter - MCL
E. S. Tucker - Res. 2
V. W. Saeger - Res. 2
W. M. Mees - Res. 2
Jerry Litschgi
Dr. Otis Fancher
Dr. Emmett Kelly

Two meetings have been scheduled on Tuesday, May 11, in Room 160, UU Building at 1700 South Second Street to review progress on biodegradation and residue work on PCB's. Dick Baxter will provide inputs on studies at MCL. Dr. Otis Fancher, Director of Industrial Bio-Test Laboratories, will attend the afternoon meeting. Recipients of this memo are invited to attend.

10:00 A.M. - Biodegradation Studies

1:00 P.M. - PCB Residues in Industrial Bio-Test Tissues

REK

1. Agenda
Oregon got memo [illegible] → R. E. Keller.
- Followers set up except Dr. Kelly of Med. Dept. will arrive with Dr. Fancher at 1:30 for afternoon meeting. Problem for anyone
db
Bio Pharmacy out.
No plan for lunch - plan [illegible]
2. These two meetings scheduled at this time - primarily because of two reasons - [illegible] point in the [illegible] to assess a plan + secondly opportune time with Dick Baxter - [illegible] may go into a little more detail than ordinarily but its [illegible]
3. Got No 13480 -
Describe S. Tuckers group - personnel.
4. Meeting objectives -
Minutes - Vic Saeger

TRAN 063363

DEPOSITION EXHIBIT
Plf's 302

FR-10 REV. 11-69

STLCOPCB4027242

MONSANTO TC

MONSANTO TC

V
A/L0207/841/DE31/1441/1/AH/QZQ

WW R RICHARD ST LOUIS
CCR A BAXTER

BIODEGRADATION WORK HAS CONFIRMED THAT BIPHENYOL-ADAPTEL ORGANISMS AITACK 2.4-DICHLOROBIPHENSL RAPIDLY IN PRESENCE OFBIPHENYL AS TRIGGER SUBSTRATE
4.4 DICHLOROBIPHE NSL APPEARS TO PERSIST.DEGRADATION OF MIXTURE OF 2.2.3-AND 2.3.4-TRICHLOROBIPHENYL ALSO OCCURS BUT LESS RAPIDLY. AI242 IN PRESENCE OF BIPHENYL SHOWS SELECTRIVE DEGRADATION OF ISOMER

S.LONGER TRIAL

NEED
XXX NEEDED TO INDICATE EXTENT TO HEEE WHICH TRICHLOR-AND TETRACHLOR-BIPHENYLS CAN BE DEGRADED

H A VODDEN RUABON
LCC
31 DEC 1537

AH841/DE31/1441

TRAN 060457

DEPOSITION EXHIBIT
PIFs 303
6-12-92

STLCOPCB4027243

FEB 10 1971

**Monsanto**

FROM (NAME & LOCATION): W. B. PAPAGEORGE - ST. LOUIS

DATE: FEBRUARY 8, 1971

SUBJECT: PCB ENVIRONMENTAL PROBLEM
JANUARY STATUS REPORT

cc: E. S. BERGEN
J. MASON

PLAINTIFF'S EXHIBIT 305
8-12-91

TO:
P.J.A. MARSH - BRUSSELS
W. S. CLARK
J. R. DURLAND- TOKYO
E. V. JOHN
R. E. KELLER - 2ND STREET
R. M. KOUNTZ
T. L. GOSSAGE
P. S. PARK
W. R. RICHARD
J. R. SAVAGE
E. P. WHEELER

GENERAL:

PCB's as high as 40 ppm in fatty tissue have been detected in chickens in New York State. FDA and USDA agreed to permit 180,000 pounds to be released for food use if PCB content does not exceed 5 ppm in salvaged edible parts. Source of PCB has not been determined.

The State of Florida informed Monsanto's Pensacola Textile plant it must take emergency measures to prevent PCB's from entering the Escambia River estuary and bay and within 30 days have an approved permanent solution or face a penalty of $5,000 per day. No PCB's are present in the plant effluent. The 2.5 ppb PCB's which were detected are being extracted from sediment contaminated prior to the summer of 1969.

Dr. K. Diane Courtney, National Environmental Health Sciences Institute, Durham, N.C. who conducted terotology studies on 2,4,5-T and EDTA-mercury-cadmium informed us that she found no teratogenic effects on rats with Aroclor 1254 and Aroclor 1260. She plans to repeat her studies using laboratory mice.

Legislation has been proposed in the Massachusetts House of Representatives banning the discharge of PCB's to the environment. A public hearing is scheduled for February 8, 1971.



DEPOSITION EXHIBIT
PK's 304
6-12-92

MARKETING:

Pydraul Reformulations - will be completed by our March 1 deadline. Most of the Pydrauls have already been reformulated, and assuming on February 15 our reformulated Pydrauls 135A, 230A, 312A, 540A, and 150A pass the Factory Mutual Fire Test, our work will be done.

General Motors, Phosgene Issue - seems to be under control. Elmer Wheeler and Raleigh Garcia will visit G.M. Tonawanda the week of February 8 to try and get them to issue a memo which will specify the Pydrauls as non-hazardous. Based on some telephone conversations Raleigh has already had, he thinks there is a fair possibility of accomplishing that goal.

U. S. Steel, Christy Park - converted to Vital 29. Though this competitive fluid is lower in price, Raleigh Garcia is convinced the move was primarily based on environmental considerations. To our knowledge, this is our first major loss of business for ecological reasons.

Pydraul A200-B - is now fully accepted by Johnson Motors and Chevrolet-Massena as a complete replacement for Pydraul A-200.

5-drum dielectric spillage on common carrier cleaned up so future contamination of anything not possible.

Tankcar of fluid from C.E. at Rome, Georgia coming into WGK for incineration.

More heat transfer FR food users switched to Therminol 66. Mrs. Pauls Kitchens was a big one this month - 60 drs. at various locations.

ENGINEERING:

CRA 2615 - Incineration - WGK

Design is nearing completion and field work was started by the plant maintenance force. Foundation work is well along and considerable pipe has been fabricated.

CRA 2617 - MCS-1016 - WGK

The project scope was modified to incorporate facilities for producing all liquid Aroclors at W. G. Krummrich plant. The concept here is that the Anniston facilities would be held in standby position. The revised scope was approved and the revised appropriation request data transmittal was issued.



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Definitive design was continued and field work was started by the plant maintenance forces. Construction was a little slow in the start because outside labor forces would not support the plant's effort. At this time, approximately one-half of the drilled piers for foundations are completed and foundation work has started. This work needs to progress rapidly to permit shipping the fractionation column which is being assembled in St. Louis.

CEA 7817 - Disposal of PCB's on Solids

Quotations were received from selected vendors for various combinations of facilities. Simultaneously, Marketing was surveying Monsanto customers to better define what and how much solids would be returned. Engineering has been temporarily stopped until a forward program is established.

CEA 2451 - N-C Phosphate - JFQ

Research, Manufacturing and Engineering continued to study the process and resolve problem areas. The process is now fixed and Engineering is revising the preliminary scope and getting the capital estimate. An appropriation request data transmittal is scheduled for late this month.

JAPAN:

No new developments reported.

EUROPE:

Customer letter sent to all non-controllable PCB users in the U.K. advising of decision to cease sales on March 1st 1971.

All European D.S.O. Managers have been advised of this decision and background.

We are investigating press statements that restrictions or a complete ban will be put on the use of PCB's in Sweden.

At a meeting with the Marine Safety Department of the Board of Trade in London, we were informed that Aroclor 1242 had been classified under Class 9, Miscellaneous Hazardous Goods, primarily on the grounds that ecological damage could be caused in inshore waters. It was agreed that the hazard from acute toxicological effects was very slight and that classification in any higher category would not be warranted. The question of the suitability of the present Monsanto tote bins was raised. This will be investigated by our Distribution Department.



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MEDICAL:

Reports on the acute vapor inhalation studies with MCS -1016 and Aroclor 1221 were received from Industrial Bio-Test Laboratories.

The data obtained in these brief studies indicate that four hour exposures to a vapor concentration of 0.64 milligrams per liter of air of Aroclor 1221 and 3.7 milligrams per liter of MCS 1016 had no effect on the test animals. The above concentrations represent approximate saturation with the air of the vapor under ambient conditions.

Dr. Keplinger of Industrial Bio-Test was queried by Dr. Garth Fitzhugh of the U. S. Food and Drug Administration as to the current results on the on-going rat reproduction studies. He was reassured that 10 ppm of 1242 show no effect and the continued indication that 100 ppm of 1254 and 1260 show no effect on reproduction.

PUBLIC RELATIONS:

Following inquiries from the Pensacola Plant and Business Week magazine, background information on Monsanto's program to control PCB was furnished. Pensacola had been receiving calls and letters following a TV show "Death of the Gulf" which partially implicated the plant and PCB. Business Week was working on a general pollution control story.

News articles and editorials from Wales have told a positive story of Monsanto's efforts to restrict sales to closed systems and to convince other manufacturers to do the same.

RESEARCH:

Biodegradation Studies - Ruabon

To investigate and define the extent to which biodegradation of polychlorinated biphenyls is likely.

A biphenyl-degrading culture ($C_2$) has been found to degrade many isomers of PCB. A theoretical hypothesis has been formulated, from the known degradation pathway for biphenyl, to predict which chlorinated isomers might be expected to degrade. Experimental work has largely confirmed this hypothesis although a few loose ends still remain.

The extent of degradation of a number of Monsanto products has been studied experimentally. Analytical procedures have been developed to analyse the degradation residues and technique for quantifying these residues is at an advanced stage of development. It has been demonstrated that the



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order of degradability for Monsanto products is as follows:

MCS 1016 > MCS 717 > MCS 762 > Aroclor 1242, MCS 1016 being the most degradable.

Experimental work has also demonstrated that HB40 can be degraded by $C_2$, but as with the PCB products, quantification of the extent of degradation has still to be determined.

We are supporting a project at University College, Bangor, in which it is hoped to select and study cultures specifically for degrading chlorinated biphenyls. Claims to have isolated cultures capable of growing on Aroclor 1242 and 1254 have not so far been substantiated, but a new culture, $C_3$, which appears to degrade biphenyl through a different pathway from $C_2$ has been isolated. This culture is being grown at Ruabon for experimental studies.

Aroclor - Environmental Contamination - Ruabon

1. Prevention of Indiscriminate Pollution by PCB

Technical service to customers has included advice on how to avoid pollution and instruction on disposal of waste material. Analytical service has been provided in many cases to assist customers in defining their pollution problems.

We have established that the incinerator operated by Purle Waste Disposal Ltd., at Rainham is satisfactory for the disposal of liquor PCB wastes. We have had discussion with incinerator manufacturers and operators to find a suitable unit for the incineration of solid wastes, but have not yet completed this.

Advice and assistance has been given to the Newport Plant on monitoring and control of pollution. Improvements in the interception of Aroclor contaminated drain-water and in pollution-control generally, coupled with routine monitoring of the plant effluent, have resulted in a steady decrease in the average level of PCB's., but occasional high levels indicate the need for constant vigilance.

2. Technical Cooperation with National Authorities

During the year we have maintained close relationships with a number of national authorities in the U.K.; these have included the Nature Conservancy, the Ministry of Agriculture Fisheries and Food and the Government Chemist. In the Netherlands, the University of Utrecht



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and the TNO organization have been visited; in Sweden we have liaised with the National Environment Protection Board. Through meetings, discussions and correspondence with numerous societies and organizations interested in various aspects of the environment we have presented Monsanto's technical contribution to the PCB problem and have, in general, received complimentary comment regarding Monsanto's attitude.

The discovery, by analysts at the Ministry of Agriculture's Infestation Control Laboratory of relatively high levels of PCB's (40% chlorine) in small cardboard drums was one of the first reported findings of the lower chlorinated PCB's in the environment. This report was a major factor in influencing MCR/Wiggins Teape to change from Aroclor 1242 to HB40.

Although spasmodic press reports have appeared relating PCB's to the sea-bird incident of late 1969, the NERC report on the incident has not yet been published. The final draft, of which we have been promised a copy, has not yet been agreed amongst all the contribution.

We made technical presentations to the other European manufacturers of PCB products as part of a plan for unified action in Europe.

The paper on "Askarels-Environmental Pollution" was presented at the August CIGRE meeting in Paris and a modified version made available to the Conference on PCB's at Stockholm in September. This paper was well received.

Environmental Studies - Runcorn

During a visit to B.I.C.C. at Helsby we introduced MCS-1016 as a potential replacement product for Aroclor 1242. In a general discussion on pollution problems, with the Technical Manager, it was clear that BICC are anxious to cooperate. They will also look for any technical improvements which MCS-1016 might provide and report on to the results of the trials.

There has been some U.K. press reaction to the publication in "Nature" of the report by A. V. Holden, at Pitlochry, that PCB's are present in sewage sludge which is being dumped in the Clyde and Thames estuaries. This is the report on the work which Holden described to us last May. The "Guardian" report links this with the bird-kill in the Irish Sea in late 1969. We are still awaiting the draft of the NERC report on that incident. The PCB story has now been superseded, at least temporarily, in the U.K. press by the "mercury in tuna fish" scare.

BIR 007956

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Biodegradation - Ruabon

We have still failed to degrade the 2,3,3',4,5-pentachlor biphenyl using the $C_2$ organism, using a technique similar to that used successfully with the 2,2'3,3',4',5'-hexachlor biphenyl. Work is still continuing with this and with 4,4'-dichlor biphenyl.

A new soil culture ($C_3$), claimed by workers at University College, Bangor, to degrade biphenyl by a different mechanism from $C_2$, has been used to degrade Aroclor 1242 and MCS-1016. Preliminary results confirm that degradation occurs, but we do not yet have information on the pattern of degradation.

Work is progressing on the analytical procedures for quantification and identification of residues. We are now able to identify a number of peaks and by a process of elimination suggest probable identification of other components. We hope to give figures for quantifying total residues in degraded Aroclor 1242 and MCS-1016 during the next few weeks. This information will be extremely useful in "selling" MCS-1016 against competitive products.

Environment & Applied Sciences Analytical Chemistry Group

G.E. Samples

The analysis of milk samples, submitted by G. E. Pittsfield, Mass., for PCB's have been completed. The electron capture (EC) chromatograms of the extracts, although appreciably altered, were most similar to our production Aroclor 1254-Aroclor 1260 type materials. The levels found ranged from 24.0 - 29.3 ppb apparent PCB's calculated as Aroclor 1254. A second set of samples was requested to verify these results. They are in hand and will be analyzed as soon as possible.

Bisphenol A - JFQ

Three samples taken from a current lot (QM-852) of Bisphenol A were analyzed for PCB's. The material contained 26 ppb PCB's calculated as Aroclor 1242. Aroclor 1242 was used as the standard, although lighter chlorinated biphenyls were also present. The EC chromatograms were not identical with either Aroclor 1232 or Aroclor 1242.



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Aroclor 1254, Aroclor 1242, MCS-1016 & MCS-1043

Theoretically, the environmental residence time of the above materials should decrease significantly from left to right because of the decreasing levels of refractory PCB isomers in each. To date the SCAS testing of these materials has not shown consistently different bacteriodegradation rates. However, statistical analysis of the last set of SCAS data points did show significant evidence of biodegradability for MCS-1043, some evidence of biodegradation of MCS-1016 and Aroclor 1242, and no evidence of biodegradation of Aroclor 1254. This coupled with the MCL $C_2$ data which also indicates that MCS-1016 is more degradable than Aroclor 1242 leads us to believe that if the SCAS test continues our optimizing degradation differences will be measurable. In February, after the completion of testing of current compounds, the units being fed the four PCB test materials will be monitored again. It is hoped that the longer acclimation period in conjunction with a switch to a longer feeding cycle (48 hours versus 24 hours) will produce measurable degradation rates this time. These tests will require 4 to 5 weeks to complete.

In the meantime, J. Mainprise of MCL has been contacted and has agreed to calculate relative degradation rates for the materials they studied by comparison of the EC traces before and after exposure to the $C_2$ culture.

Aroclor Pollution Study - Anniston

Aroclor was removed from the plant effluent by filtration of the neutralization pit solids on which Aroclor was adsorbed and by adsorption on the filtration media. Aroclor-concentrations (100-8,000 ppb) in the plant effluent were consistently reduced to 10 ppb upon passing through a four (4) inch diameter tube (packed to a bed depth of 5.5 inch with activated carbon) at flow rates up to 350 cc/min. In the sand filtration study conducted under similar operating conditions, the Aroclor levels in the effluent were not consistently reduced to the target level ($\leq$ 10 ppb). The present of 200-1000 ppm of solid particles (size range 1.5 - 250 $\mu$ ) tended to blind the filter, thus setting a maximum limit on the pressure and flow at which the unit had to be operated.



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Conclusion

1. Aroclor is removed from the plant effluent by the filtration of the solids on which Aroclor was adsorbed.

2. Aroclor is adsorbed on activated carbon within a very short adsorption zone.

3. The effectiveness of sand in removing Aroclor is not sufficient at the experimental bed depth.

4. Solids removal is a primary concern in any final clean-up method.

Future Work

1. A pilot adsorption study has been conducted in a series of 3 inch diameter columns. These results are being compiled for an evaluation and recommendation. A report will be issued by 1/28/71.

2. A pilot clarification (flocculation) unit is scheduled to be put in operation by 1/29/71. This unit will also be used to carry out semi-continuous adsorption studies with other finely divided solids.

Dielectrics

Capacitor Fluids

Research reported on a series of dielectric fluids with dielectric constants from 3.5 to greater than 20 which could be replacements for Aroclor 1242. We are seeking an optimum combination of the following variables: dielectric constant, biodegradability, fire resistance, suitable physical properties and manufacturing cost. We prepared electrical grade 2,2'-dichlorodiphenyl ether, overcoming problems of high power factor with new clean-up procedures.

Three new diphenyl sulfones were prepared for evaluation: p-cyclohexyl-p'-methyl diphenyl sulfone, p-ethyldiphenyl sulfone, and 4-methyldiphenyl sulfone. We are seeking suitable solvents to blend with these sulfones.



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Pydraul Reformulations

We issued a Tentative Process for Pydraul 312A, an oil, Santicizer 711, CDP, Aroclor 5432 blend. We are seeing increasing competition from Vital 29 and Dasco FR-300 for the Pydraul 312A-type market. Vital 29 contains 56% hydrocarbon oil, 39% Reofos type phosphate ester, 5% fatty acid solubilizer plus stabilizers. Fire resistance is close to Pydraul 312 and the fatty acid has a positive effect on fire resistance.

Pydraul 540B has accumulated 700 hours on the oil gear pump with no signs of wear.

We are adjusting the spray fire resistance of Pydraul 135A and Pydraul 230A to meet FM requirements.

Pydraul 150A was formulated with improved fire resistance over Pydraul 150.

MANUFACTURING:

Anniston Plant

Current Level

Aroclor losses during January averaged 1.8 lbs/day (307 ppb). This was up from December. Lbs/day up - ppb down due to 300 gpm water flow from start-up of Hiram.

| | | |
|---|---|---|
| 28 day average | 138 ppb | .8#/day |
| Amount from Hiram | | (.4) |
| Increase due to work on sewer to stop contamination of current water flow | 169 | 1# |
| | 307 | 1.8# |

Aroclor Removal From Waste Water

Issued Progress Report R&D 646-514, Report No. 1, sent to all Aroclor producing locations.

Projects & Evaluations

No. 1 EC1 pit 90% complete - 2 more weeks work to do.

Revised scope of carbon collection & disposal. Will be published by end of February.

Solids removal Pilot Plant construction is complete. PR&D work is underway.

Had no spills of Aroclor in January.

BIR 007960

W. G. Krummrich Plant

PCB Levels In Sewer

A. Aroclor losses from the Aroclor Department averaged about 0.6 lbs/day with a high of about 1.1 lbs/day.

Losses from the treatment plant for the period 12/17 - 1/15 averaged 22 lbs/day (114 ppb). Range for 6 samples was 2 to 53 lbs/day (9-278 ppb).

Data gathering on Aroclor in the various plant sewers is in progress. Samples are being composited twice weekly for analysis.

Projects to Reduce Sewered PCB's from Aroclor Department

Design package for Estimate 2422 to pave and trench loading and storage areas was completed and issued in January.

Programs to Eliminate Sewered PCB's From Using Departments

Therminol system in CHA (Dept. 222) was changed to Therminol 66 during the month.

Completion of design package for 13 additional units is scheduled for 2/15/71. Changeout of all units is targeted for completion by 6/1/71.

Several alternates including use of a eutectic salt are being evaluated for the Dept. 245 ($P_2S_5$) Therminol unit. Evaluation is scheduled to be completed by 4/1/71.

Decision on chemical compatibility of the new Pydraul in Dept. 251 will be made by 3/1/71.

In order to reduce the risk of PCB's being sewered from washing Aroclor trailers at Rogers Terminal, all Rogers trailers containing PCB's will be washed at MCK's Rip Track.

Reach 10 ppb in the Treatment Plant Effluent

At this time, it appears probable that PCB losses to the sewer can be controlled at less than 2 lbs/day (equivalent to 10 ppb at the treatment plant) by 9/1/71. This will be accomplished by:

1. Continuing current control of losses from Dept. 246.

2. Changeout of all Therminol units to non-PCB fluids.



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3. Installation of pads and catch basins at the loading and storage areas in the Aroclor Department.

4. Installation of a catch basin at the Rip Track.

Losses at the treatment plant are expected to remain above 10 ppb by 9/1/71 due to past sewer contamination. Projects to clean sewers could run several hundred thousand dollars. A preliminary estimate to clean the sewer from the Chlorine Department to the plant fence was $70,000 and another $70,000 would be required to clean from the fence to the treatment plant.

Both main plant sewers appear contaminated with PCB's (30W and 24W). Our plan is to continue monitoring the main sewers to determine extent of losses and loss dissipation rate. By 6/1/71, when essentially all Therminol units have been changed out, we should be able to extrapolate to when the PCB level should reach 10 ppb at the treatment plant without sewer cleaning. Based on this information, we should decide promptly on whether or not sewer cleaning is justified.

PCB Levels in the Atmosphere

Estimate 2675 for $6,000 expense was approved to replace the nitrogenpurge with a nitrogen blanket at the mix tanks. Project package is scheduled for completion by 3/15/71.

PCB Rework Project (Est. 2095)

Project package was issued in January.

W. B. Papageorge

W. B. PAPAGEORGE



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Monsanto

File Aroclor 1016

Reference Number MP 10227

Security Class A

Type Review Report

TITLE: Biodegradability of Aroclors 1242 and 1016

AUTHORS: R.A. Lidgett H.A. Vodden P.E. Gilbert

JOB NO: 660 10 122 13 REPORT NO: 2 DATE. 13.9.71

ABSTRACT

The degradation of Aroclors 1242 and 1016 has been studied using two biphenyl degrading organisms $C_2$ and $C_3$ for periods up to 100 days. Results indicate good biodegradability for all components of Aroclor 1016.

Issuing Authority R.A. Lidgett

TBA

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63

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SUMMARY OF RESULTS

N.J. !

1. In the presence of biphenyl, Aroclors 1242 and 1016 are degraded by both C2 and C3 organisms. After 100 days Aroclor 1016 residues are less than 5% the original concentration with either organism. Aroclor 1242 is degraded to a lesser extent, there being approximately 5 - 10% remaining with the C2 organism and 15 - 20% with the C3 organism after 100 days.

2. The longest half life for any component of Aroclor 1016 is approximately 25 - 30 days.

3. For pure compounds, there is evidence to suggest that C2 and C3 have different rates of degradation and each is specific to certain basic structures; in complex mixtures this does not appear to be the case since C2 and C3 degrade the components of Aroclor 1016 at approximately the same rate and to approximately the same amount.

4. Analytical developments show that the electron capture detector response to individual components can be predicted with an accuracy sufficient for most environmental analytical problems using packed columns and that with capillary columns quantitative measurements can be made upon degraded P.C.Bs at the 0.01 ppm level

[redacted]

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INTRODUCTION

A previous review report (No. MP 10103) discussed results obtained for the biodegradation of pure chlorinated biphenyls using a biphenyl degrading organism C2 (the Sudan organism) obtained from Professor Charles Evans of the University College of North Wales, Bangor. The effect of C2 on commercial and development samples (1242, 1016, 717, 762 etc.) was also discussed but, because of analytical difficulties encountered in the identification of specific components in the mixtures, only very limited interpretation could be made of the results.

Since the issue of that report the identification of components has been greatly improved and, in the work reported here, Aroclors 1242 and 1016 have been analysed after submission to biodegradation in the presence of biphenyl by two cultures C2 and C3 for one hundred days. Improvements have been made in the quantitative analysis and it has been possible to study the change in individual isomers at levels below 0.05 parts per million.

1. DETAILS

1.1 Organisms used for degradation of Aroclors 1242 and 1242B

Two organisms both received from Professor Charles Evans at University College of North Wales, Bangor have been used for biodegradation.

The first organism C2 is that originally received and used for the initial work on the degradation of pure compounds. This was isolated in a soil sample from the Sudan and is reported as being a nocardium of the order actinomycetes.

The second organism C3 was isolated from a canal mud sample in the screening work carried out by Professor Evans student, C. Harrison. This is reported as being of the pseudomonas family.

The mode of attack by these organisms on biphenyl has been studied by Professor Evans' group and a simplified scheme is given later in the Appendix.

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1.2. Preparation and maintenance of stock cultures of biphenyl degrading organisms

The procedure used is identical to that reported earlier (Review Report No. MP 10103).

1.3. Procedure for studying P.C.B. biodegradation

A number of changes have been made in the procedure used to study biodegradation. These have been made to increase the number of samples examined at any time and to improve the qualitative and quantitative accuracy of the analytical method.

(i) Addition of Orbital Incubator

To increase capacity, an orbital incubator was purchased (Gallenkamp) with a capacity of 15 x 1000 ml, 28 x 500 ml. or 36 x 250 ml. flasks.

Past experiments have been designed so that the complete contents of any shake flask have been extracted and analysed. A more attractive method would be the use of aliquots whereby the fate of a sample in any flask could be studied over a period. This method was rejected earlier because (a) the flask shaker was too small to handle the larger volumes needed for aliquot work and (b) it was strongly suspected that the sample would not be homogeneously dispersed in the medium but may be partly adsorbed on the flask wall.

In view of the increased size of flask, which could be accommodated in the incubator, it was felt that the use of aliquots should be evaluated - if successful the variation in results caused by the use of separate flasks would be eliminated and a more varied biodegradation programme could be designed.

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The evaluation was carried out as follows :

Six 1 litre flasks were dosed with Aroclor 1242 in the normal way. Each flask contained 700 mls of water with an Aroclor 1242 concentration in the flask equivalent to 5 ppm. If the aliquot method was suitable each 100 ml aliquot of water should contain 5 ppm of 1242.

The flasks were shaken in the incubator (1.25 r.p.m., 25°C) for eight days after which a 100 ml aliquot was taken from each flask. Of the remaining 600 mls of water in each flask, 500 ml was discarded and the remaining 100 ml recovered for analysis. These first and final aliquots were extracted with pentane; in the case of the final 100 ml aliquot the pentane was first used to remove any Aroclor 1242 adhering to the flask. The pentane extracts were analysed using packed column gas chromatography. Results are given below, the figures in brackets are the theoretical figures which should be obtained if the aliquot system is reliable.

| | 1242 content (ppm) 1st Extract | | | 1242 content (ppm) Final E | | |
|---|---|---|---|---|---|---|
| 1 | 1.4 | (5.0) | ×6 = ۸.۴ | 24.4 | (5.0) | |
| 2 | 1.7 | " | ۱۰.۲ | 22.6 | " | |
| 3 | 0.8 | " | ۴.۸ | 24.5 | " | |
| 4 | 0.7 | " | ۴.۲ | 35.7 | " | |
| 5 | 1.3 | " | ۷.۸ | 25.5 | " | |
| 6 | 1.0 | " | ۶.۰ | 29.8 | " | |

It can be seen from these results that the aliquot system is not suitable for aqueous samples containing Aroclors and that adsorption to the container wall is so large that this factor is likely to be important in the interpretation of any data from similar experiments.

Degradation data from these experiments may be a "wall effect".

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Thus the problem still remained of how to obtain the maximum information using a limited number of samples. In previous work each sample under study had three flasks prepared as follows :

1. Sample + Culture + Nutrients + Biphenyl
2. " " " no Biphenyl
3. " " " " " + $HgCl_2$

In every case to date, the sample with mercuric chloride has given results in agreement with a normal standard not submitted to biodegradation and therefore this series of flasks has been excluded from the present work. This has increased the relative capacity of the incubator by 30%.

1.4. Improvements in the Analytical Procedure

The details of the analytical method used are given in the Appendix.

1.4.1. Identification of Components

The development of a suitable analytical method has been a major problem in the study of the biodegradation of complex commercial and development samples of P.C.B's. Packed column gas chromatography using the electron capture detector will give a measure of whether a complex sample is undergoing biodegradation but the resolution attainable is not sufficient to determine the fate of individual components. Without this knowledge and because the response of the E.C. detector to individual P.C.Bs varies with structure, quantitative results must be viewed with caution.

It is therefore necessary to use high resolution capillary columns to obtain detailed information but a further complication is introduced in that there are insufficient pure P.C.Bs available to identify all the peaks in a chromatogram (up to 50 in 1242) using the normal procedure of "spiking".

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In the early stages of this work it was therefore decided to concentrate upon the use of capillary columns coated with the non polar phase Apiezon L. While these have a poorer resolving power than many polar columns of equivalent length they have the major advantage in eluting components with respect to boiling point and this property can often be predicted from a knowledge of structure and the behaviour of similar compounds.

Emperical rules were drawn up to predict the probable retention times of unavailable P.C.Bs, the rules being based upon steric factors, molecular weight etc. Such rules aided in the identification of certain components but were not sufficiently detailed or established to cover all components.

This approach was discussed with Welti, Sissons and Telling at Unilever who were working along similar lines. A thorough investigation by this group using confirmatory techniques of N.M.R., mass spectrometry and the spiking procedure resulted in complete (within experimental error) identification of all components in 1242, 1254 and 1260.

This work is awaiting publication but we have had access to the data for the previous six months and, combined with our own, this has been used for the identification of all components named in the results section.

1.4.2. Quantitative Accuracy

While improvements have been made in identification of components, the quantitative accuracy of the gas chromatographic method has still to be improved. Use of capillary columns mainly overcomes the difficulty of merged peaks but it has a disadvantage in that it requires more concentrated samples than packed column gas chromatography.

To overcome this, the concentration proceudre described in the Appendix was developed. This involves concentration of the pentane extract to a low volume, followed by addition of a small known amount of decane. This allows complete removal of the pentane giving a concentration factor of up to 200X without significant loss of P.C.B. components and enables accurate measurements to be made on degraded components at levels as low as 0.05 ppm.

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### 1.4.3. Calibration

Work by Emery et al (M.C. Physical Chemistry Method 70-20. N.N. 1348322) has shown that the F.I.D. response factors of chlorinated biphenyls depend upon chlorine content and that within any group of compounds of the same chlorine number, structural differences have a minimal effect upon response factor. Thus, in any high resolution chromatogram, if the chlorine numbers of the peaks are known, response factor correction can be applied and a true figure for either the composition of the Aroclor or its concentration in a sample can be calculated.

Unfortunately, because of sensitivity limitations, the F.I. detector cannot be used for determinations involving very low levels of P.C.Bs and the electron capture detector must be employed.

There has been much speculation on the quantitative accuracy of this detector for P.C.B. analysis since it is known from other applications that its response is very dependent upon structure; work with pure components within Monsanto and elsewhere (eg. see Zitko et al Bull Environ Contam and Toxicol 1971 6 2 pp 160-163) has indicated that with P.C.Bs there can be large differences in response for compounds of the same chlorine number but different structure

e.g. $\frac{\text{Response 2 4' dichloro biphenyl}}{\text{" 44' " "}} \approx \frac{12}{1}$

It was therefore decided that for accurate quantitative analysis using the E.C. detector the most reliable approach would be to determine the ratio for each component of the E.C.D. to F.I.D. response, use this to convert the E.C.D. chromatogram to an "equivalent" F.I.D. chromatogram and apply the F.I.D. correction factors calculated by Emery et al.

The ratio of $\frac{\text{E.C.D.}}{\text{F.I.D.}}$ response is relatively easily obtained by running the same standard sample on a capillary column using a flame ionisation detector, then repeating the experiment (after dilution) with an electron capture detector (See Figs 9 & 10). Peak areas are calculated then ratioed. The ratios obtained for each component can then be used for all subsequent samples provided that the column used has suitably high resolution.

Not correct figure
Fig 9 [illegible] not a gas chromatogram
Should be Fig 10 & 11.

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In carrying out this experiment and also in previous work it was noted that for all the concern about the unpredictable response of the E.C.D. the chromatograms obtained with the two detectors were remarkably similar and calculation of the $\frac{E.C.D.}{F.I.D.}$ ratios for all peaks shows that large differences between the two detector responses are only observed for a small number of components.

It is suspected that the similarity of E.C.D. response factors of compounds of the same chlorine number but different structure is related to the fact that almost all the components of Aroclor 1242 (and higher Aroclors) contain at least one chlorine in the ortho position. Such compounds will have non coplanar benzene rings, there will be much reduced conjugation and this general property may have such a large effect upon E.C.D. response as to make other structural differences of much lower importance for components of the same chlorine number. This would explain the anomalous E.C.D. response factor of the unhindered 44' dichloro biphenyl.

Initial measurements with pure 33' dichloro biphenyl show that this compound also has a low E.C. response almost equivalent to that of the 44' dichloro biphenyl.

As discussed, if high resolution capillary columns are used the $\frac{E.C.D.}{F.I.D.}$ ratios coupled with the F.I.D. will give an accurate quantitative figure for P.C.B. content at low ppm and ppb levels.

If the $\frac{E.C.D.}{F.I.D.}$ ratios for each set of components with the same chlorine number are averaged (neglecting components of very low concentration) an unexpected result is obtained viz.

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To convert dichlorobiphenyl E.C.D. areas to F.I.D. areas using $\frac{E.C.D.}{F.I.D.}$ ratio divide by

" " tri " " " " " " " " " "

" " tetra " " " " " " " " " "

To normalise F.I.D.(using M.C.data) areas of dichloro diphenyls multiply by

" " " " " tri " " " "

" " " " " tetra " " " "

Overall result

To normalise E.C.D. areas dichlorophineyls multiply by $\frac{0.9}{0.7}$ i.e.

" " " " tri " " " " $\frac{1.1}{1.1}$ i.e.

" " " " tetra " " " " $\frac{1.3}{1.2}$ "

Thus, within the requirements of most pollution/environmental work the total area of components of a chromatogram of P.C.Bs can be quantitatively related directly to a chromatogram of a standard P.C.B. mixture (preferably not to a pure P.C.B. since this may have a response removed from the average).

Since the start of the environmental P.C.B. problem most of the published work has been carried out with packed columns and E.C. detection. In many cases authors have advised caution in the interpretation of results because of the expected difference in response factors of the individual PCBs with the E.C. detector. It would now seem that in the case of Areclor 1242 there is less need to apply so much caution to the quantitative figures.

This, however, still leaves a major query as to whether packed column work for environmental samples is reliable since interfering non P.C.B. components with high E.C. responses present within the P.C.B. envelope cannot be detected.

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## II. RESULTS

The results obtained for Aroclor 1242 and 1016 together with interpretation and discussion are given in Sections 11.3 and 11.4.

### II.1. Sources of Error in Interpretation

In interpreting the results it must be emphasised that there are still unknown factors which may cause significant errors. Two such sources of error are considered below :

#### A. Identification of components

As discussed earlier components have been identified using one or a combination of the techniques of spiking, N.M.R. and mass spectrometry. All such techniques are open to error in a complex sample of this type, even, in some cases, the authenticity of the original component used for spiking may not be guaranteed.

B. The graphs for degradation of components are prepared from results obtained for separate samples of Aroclors taken at 32 and 100 days. Thus while the samples have been prepared under as identical conditions as possible, once biodegradation is initiated each sample is an isolated system and different rates and/or processes may occur. Every attempt has been made to ensure reproducible conditions but it has been found in a number of previous runs that occasionally a 50 day sample shows better degradation than a 100 day sample.

An attempt to reduce the chances of this situation occurring will be made in future by the introduction of the "viable cell count". This is discussed below.

### II.2 Viable Cell Count

In the discussion on degradation of 1242 and 1016 the results have been interpreted without considering in detail the effect of time on the growth of the organisms in samples in a closed system.

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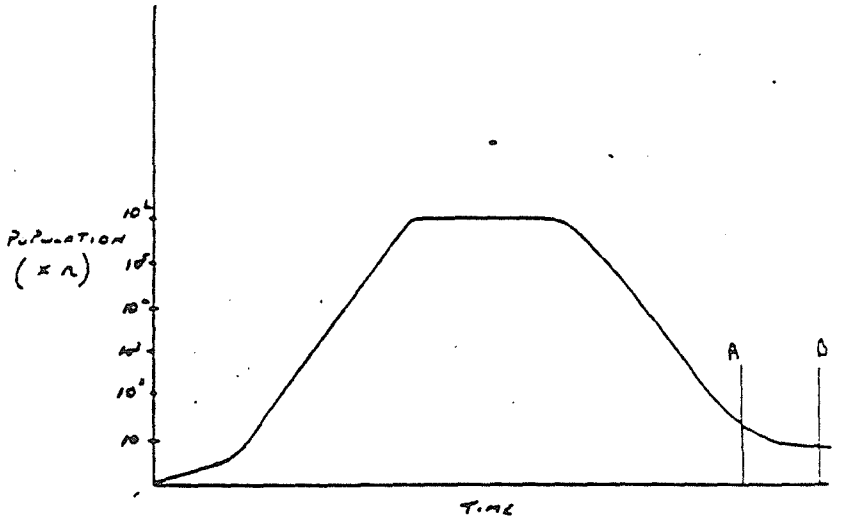
It is known that under optimum conditions a culture of micro organisms will have a growth curve for viable cells similar to that drawn below :




In the experiments described, the viable cell population has not been counted when samples have been taken, as it has been assumed that where the rate of degradation is considerable, i.e. during the first 50 days, this was unnecessary. However, when the residual material becomes very low i.e. after 100 days, the viable cell population may be in the region AB on the graph. Here the reduction in cell count is several orders of magnitude below the maximum count but the concentration of P.C.B. is reduced by less than two orders of magnitude. Thus the relative efficiency of degradation in the closed system will reduce considerably with time.

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This is not the case in most normal environments where the P.C.B. could be exposed to a number of organisms at different times, each working at high efficiency.

11.3 Biodegradation of Aroclor 1016

The percentage degradation for Aroclor 1016 by C2 and C3 after 52 and 100 days is given in Table I and schematic diagrams of the rates of degradation of the individual components are given in Fig. 7.

The rate graphs are meant for illustration only since insufficient points for each component have been obtained to allow accurate decay curves to be obtained. If the graphs are used as drawn, to obtain an approximate half life for each component (i.e. time to 50% degradation) then it can be estimated that the slowest degrading component has a half life of 33 days with C3 and 28 days with C2. If however, degradation is assumed to be exponential (which is inferred if component half life is used as a measure of efficiency of degradation) the figures are lower still probably in the region of 10 - 20 days. In practise even these numbers are on the high side since, as has been discussed earlier, efficiency of biodegradation will fall off with time in a closed system.

The graphs show that degradation appears to be smooth and relatively rapid with both organisms. Small amounts of degradation products can be seen in the early part of the chromatogram but the retention times of these confirm that they are not chlorinated biphenyls.

It has been noted earlier that the mechanisms of biodegradation for C2 and C3 with biphenyl differ (see Appendix Fig. 9) and it would therefore be expected that the high resolution gas chromatograms of Aroclor 1016 degraded by the two organisms would show major differences. In practice while there are differences these are relatively small (in comparing chromatograms it should be borne in mind that although a 1 ppm. standard is used the original concentration was 5 ppm.)

This suggests that when the organisms after adaption with biphenyl are exposed to a wide range of components they are capable of further adaptability to suit the available food source, that degradation is uniform and extensive after 52 days shows that adaptation is not difficult. This is supported for C2 by the evidence in the literature that the Actinomycetes family are known to adapt to a wide range of feed substrates.

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II.4 Biodegradation of Aroclor 1242

The percentage degradation for Aroclor 1242 by $C_2$ and C3 after 52 and 100 days is given in Table I and the apparent rates of degradation of the components are given in Fig. 8.

The comments made earlier concerning half lives of the components of Aroclor 1016 also apply in this case.

The graphs and the chromatograms from which they are derived show that biodegradation appears to be more complicated and while degradation is considerable after 100 days the rates of degradation for many components do not follow smooth curves.

Reasons can be given for the differences between the biodegradation of 1242 and Aroclor 1016 but these must be treated with caution : -

Two main differences are apparent (a) certain components in 1242 appear to decrease in the first 52 days then increase in the second period. (b) the rates of degradation for $C_2$ and C3 for individual components differ far more than with Aroclor 1016.

Both differences may be caused by the effect of the higher chlorinated components which are present in greater concentration in 1242 than in 1016.

In the first case, these may be degrading to give lower molecular weight components which elute with the same retention times as the lower chlorinated biphenyls or, in fact, the higher chlorinated biphenyls may lose chlorine and gain hydrogen by an unknown process to give the lower chlorinated compounds.

In the second case, initial degradation of the higher chlorinated materials may give rise to products which have different toxicities for $C_2$ and C3 and, in a medium of increased toxicity, these organisms will utilise the available food by different mechanisms than they would in a non toxic medium.

Again, in future work, the effect of toxicity may be detected using the "viable cell count" procedure discussed in Section II.2.

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11.5 Relationship between structure and ease of biodegradation

The present results on 1242 and 1016 for C2 and C3 provide an additional means of studying the effect of structure upon biodegradability and it should be possible to obtain a considerable amount of information from relatively few experiments. Because of the complex nature of the degradation process and the apparent ease of adaptation of the organisms it is felt that the present data is insufficient, and that further experiments should be completed before detailed correlation between structure and biodegradation are considered or attempted.

## III. FUTURE WORK

### III.1. Quantitative Accuracy

It has been suggested earlier (Section 1.4 III) that the response of the electron capture detector is largely conditioned by the non coplanarity of the benzene rings in the chlorinted diphenyl molecule.

Samples of unhindered chlorinated niphenyls have been supplied by Unilever and these will be examined to decide whether this postulate is true.

### III.2 Sample Programme

a) A repeat series of Aroclors 1242 and 1016 is underway using $C_2$ and $C_3$ and degradation will be followed until complete disappearance (50 day ready Sept. 6th).

b) A mixed culture of $C_2$ and $C_3$ is being used to attempt biodegradation of Aroclors 1242 and 1016 to determine whether the organisms can co-exist and still work equally effectively.

c) A river water die away test on 1242 and 1016 has been initiated for a long term study.

d) A variety of development samples including chlorinated terphenyls has been subjected to biodegradation by C2 and C3. The results on these will be reported separately when complete.

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III.3 Viable Cell Count

The viable cell count method is being carried out to obtain expertise in this technique. It will then be applied to each sample submitted for analysis.

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Appendix


| | | | |
|---|---|---|---|
| 1. | Table I | % Biodegradation with $C_2$ and $C_3$ after 51 and 100 days | |
| 2. | Table II | Half lives of components of Aroclor 1016 | |
| 3. | Fig.1. | Chromatogram of 1 ppm of Aroclor 1242 | (0 days) |
| 4. | Fig.2. | " 5 ppm " " " | 51 days ($C_2$) |
| 5. | Fig.3. | " " " " " | 100 days ($C_2$) |
| 6. | Fig.2a. | " " " " " | 51 days ($C_3$) |
| 7. | Fig.3a. | " " " " " | 100 days ($C_3$) |
| 8. | Fig.4. | " 1 ppm " " 1016 | 0 days |
| 9. | Fig.5. | " 5 ppm " " " | 51 days ($C_2$) |
| 10. | Fig.6. | " " " " " | 100 days ($C_2$) |
| 11. | Fig.5a. | " " " " " | 51 days ($C_3$) |
| 12. | Fig.6a. | " " " " " | 100 days ($C_3$) |
| 13. | Fig.7. | Rates of degradation for components of Aroclor 1016 ($C_2$ and $C_3$) | |
| 14. | Fig.8. | " " " " " " " | 1242 ($C_2$ and $C_3$) |
| 15. | | Method for G.C. Analysis of degraded samples | |
| 16. | Fig.9. | Metabolic Pathways for degradation of biphenyl with $C_2$ & $C_3$ | |
| 17. | Fig.10. | Chromatogram of Aroclor 1242 F.I. detector. | |
| 18. | Fig.11. | " " " " E.C. detector. | |
| 19. | E.C.D. and F.I.D. Relative Response Factors. | | |


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TABLE I  % Biodegradation after 52 and 100 days

| Time | 1242 | | 1016 | |
|---|---|---|---|---|
| | $C_2$ | $C_3$ | $C_2$ | $C_3$ |
| 52 days* | 88 | 76 | 96 | 91 |
| 100 days * | ~95 | ~ 85 | > 98 | > 96 |

* Analyses carried out using packed column method

* 100 day figures estimated by comparison of capillary column chromatograms for 52 and 100 days.

With added biphenyl

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TABLE II Half Lives of Components of Aroclor 1016 (days)

| Component | $C_2$ | $C_3$ |
|---|---|---|
| 2. 2'5' Tri-Di Chloro Biphenyl | 26 | ~26 |
| 2. 2'4' " " | 26 | 33 |
| 2 2'3' and/or 3 2'6' Di/tri " " | 26 | 33 |
| 4, 2'6' " " | 27 | 32 |
| 44' Dichloro " | 25 | 27 |
| 2,2'4'6' and/or 25,2'6' Tetra Chloro Biphenyl | 30 | 30 |
| 2 3'5' and/or 3,3'6' Trichloro Biphenyl | 26 | 26 |
| 4 2'5' " " | 26 | 26 |
| 3 2'4' " " | ~26 | ~26 |
| 2 3'4' " " | 26 | 26 |
| 2, 2'3'5' and/or 25 2'5' Tri/Tetra Chloro Biphenyl | 26 | 26 |
| 2, 2'4'5' and/or 24,2'5' " " " " | 26 | 26 |
| 3, 2'3'6' and/or 3, 2'4'6' " " " " | 26 | 26 |
| 4,2'3'6' " " " | 26 | 26 |
| 23, 2'4' " " " | 28 | 24 |
| 2,2'3'4' and/or 23, 2'3' " " " | 28 | 24 |
| Unknown | 26 | 24 |

* Peak off scale at attenuation used, half life estimated.

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## Gas Chromatographic Analysis of Biodegraded Samples

Pentane extracts were obtained in a manner identical to that previously reported.

### Method

A suitable aliquot (50 or 25 mls) of the pentane extract was evaporated to low volume (2 - 3 ml) in a Dreschel bottle on a water bath at 60°C using a nitrogen purge.

n Decane (0.1 ml) was placed into a tared specimen tube (13 mm x 45 mm) which was then reweighed. The concentrated pentane extract was pipetted into the decane followed by a small amount of pentane used to rinse the Dreschel bottle. The n pentane was then removed at room temperature again using nitrogen and the complete removal of pentane confirmed by reweighing the specimen tube.

Standards of Aroclor 1242 and 1016 were prepared as above by evaporation of 25 mls of a 1 ppm solution.

The rate of loss of any component was determined by measurement of the peak height of that component.

The concentration procedure was checked for reliability by running standards in duplicate - results were repeatable within ± 5%.

### Gas Chromatographic Conditions

Column - 200' s/s capillary 30 thou bore (ex Phase sep.) coated with Apiezon L using the method of Malec E.J. ( ).

Temperature 210°C

Detector $Ni^{63}$ (H&P) at 325°C

Carrier Argon/Methane 95/5 Input Pressure 5 p.s.i.

Internal Standard - None

Chromatogram Duration 90 minutes.

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TABLE 3. E.C.D. AND F.I.D. Relative Response Factors

| Pk No | Cl No | E.C.D. | F.I.D. | E.C.D./F.I.D. |
|---|---|---|---|---|
| 5 | 2 | 0.02 | 0.01 | 2.0 |
| 6 | 2 | 0.06 | 0.09 | 0.7 |
| 7 | 3 | - | - | -- |
| 8 | 2 | 0.38 | 0.83 | 0.5 |
| 9 | 3 | 0.61 | 1.08 | 0.56 |
| 10 | 3 | 0.37 | 0.47 | 0.79 |
| 11 | 3 | 0.40 | 0.47 | 0.85 |
| 12 | 3 | 0.42 | 0.25 | 1.68 |
| 13 | 2 | 0.03 | 0.21 | 0.14 |
| 14 | 4 | 0.05 | 0.05 | 1.0 |
| 15 | 3 | 0.18 | 0.17 | 1.1 |
| 16 | 3 | 0.19 | 0.14 | 1.35 |
| 17 | 3 | 1.0 | 1.0 | 1.0 |
| 18 | 3 | 2.10 | 1.71 | 1.23 |
| 20 | 3 | 0.54 | 0.40 | 1.35 |
| 21 | 4 | 0.23 | 0.29 | 0.79 |
| 22 | 4 | 0.31 | 0.32 | 1.0 |
| 24 | 4 | 0.35 | 0.27 | 1.30 |
| 25/26 | | - | - | - |
| 27 | 4 | 0.13 | 0.07 | 1.9 |
| 28 | 4 | 0.29 | 0.20 | 1.45 |
| 29 | | 0.25 | 0.12 | 2.08 |
| 30 | 3 | - | - | - |
| 31 | 3/4 | 0.16 | 0.32 | 0.50 |
| 0 | | - | - | - |
| 32 | 4 | 0.08 | - | - |
| 35 | 4 | 0.35 | 0.32 | 1.10 |
| 36 | 4 | 0.17 | 0.14 | 1.21 |
| 37 | 4 | 0.08 | 0.08 | 1.00 |
| 38 | 4 | 0.02 | 0.03 | 0.66 |

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FIG. 9 Metabolic pathways for the biodegradation of biphenyl with $C_2$ and $C_3$

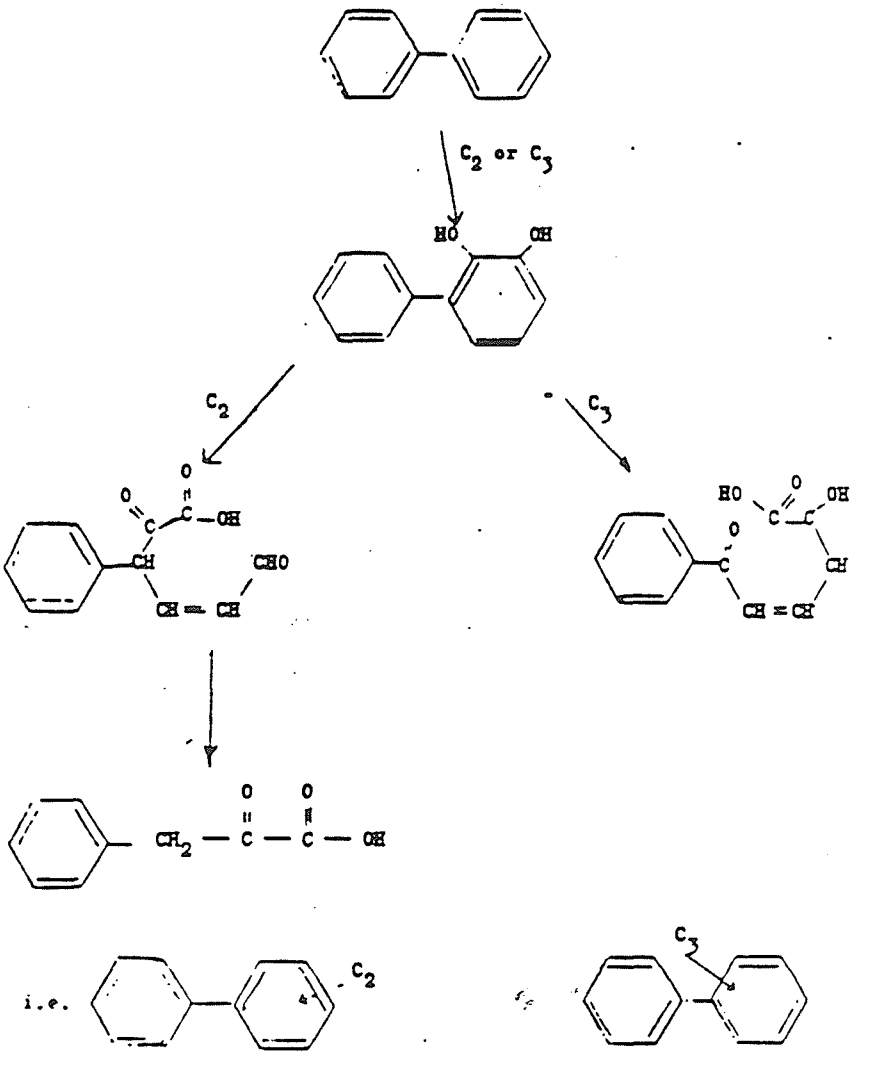


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fig 1

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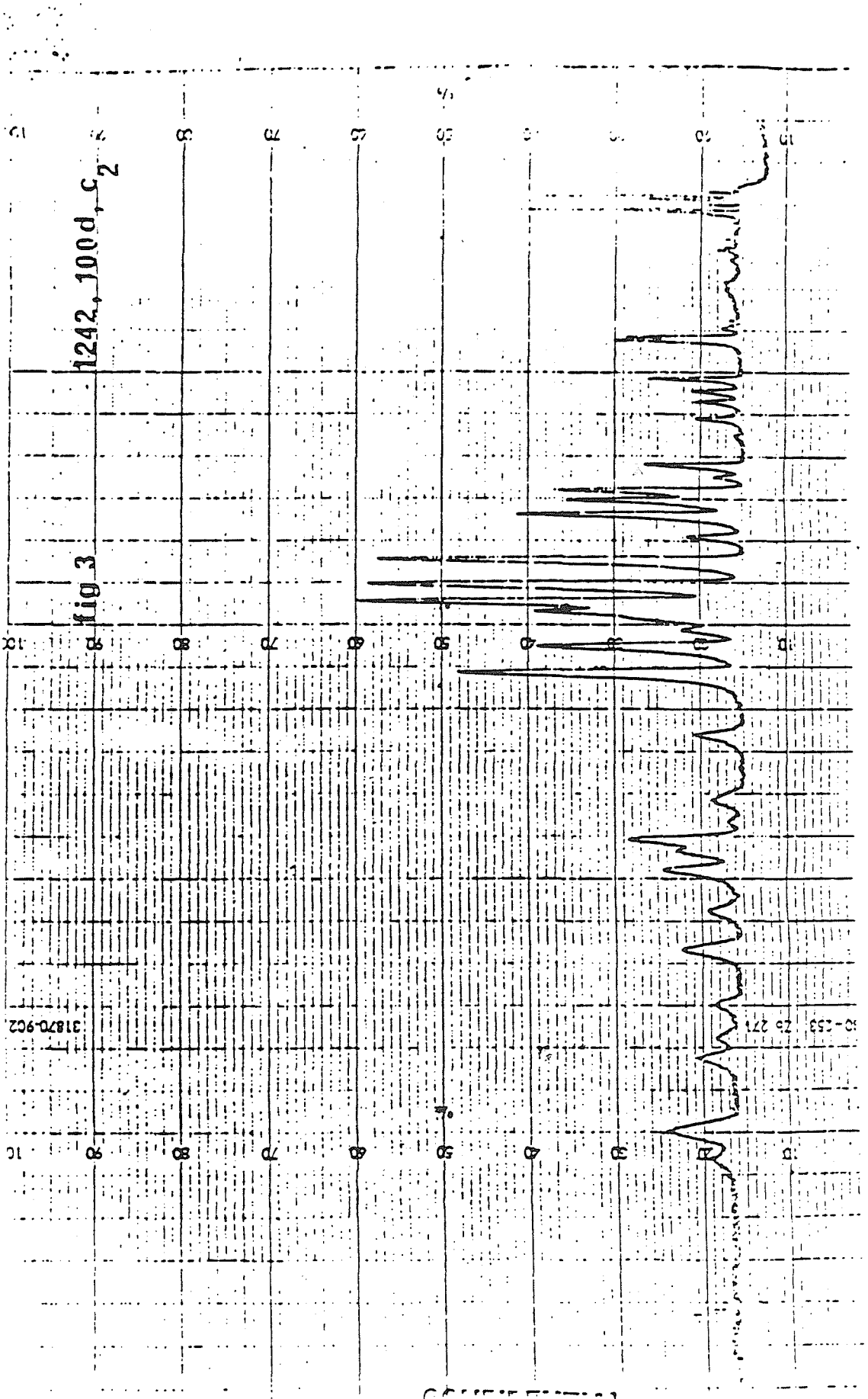


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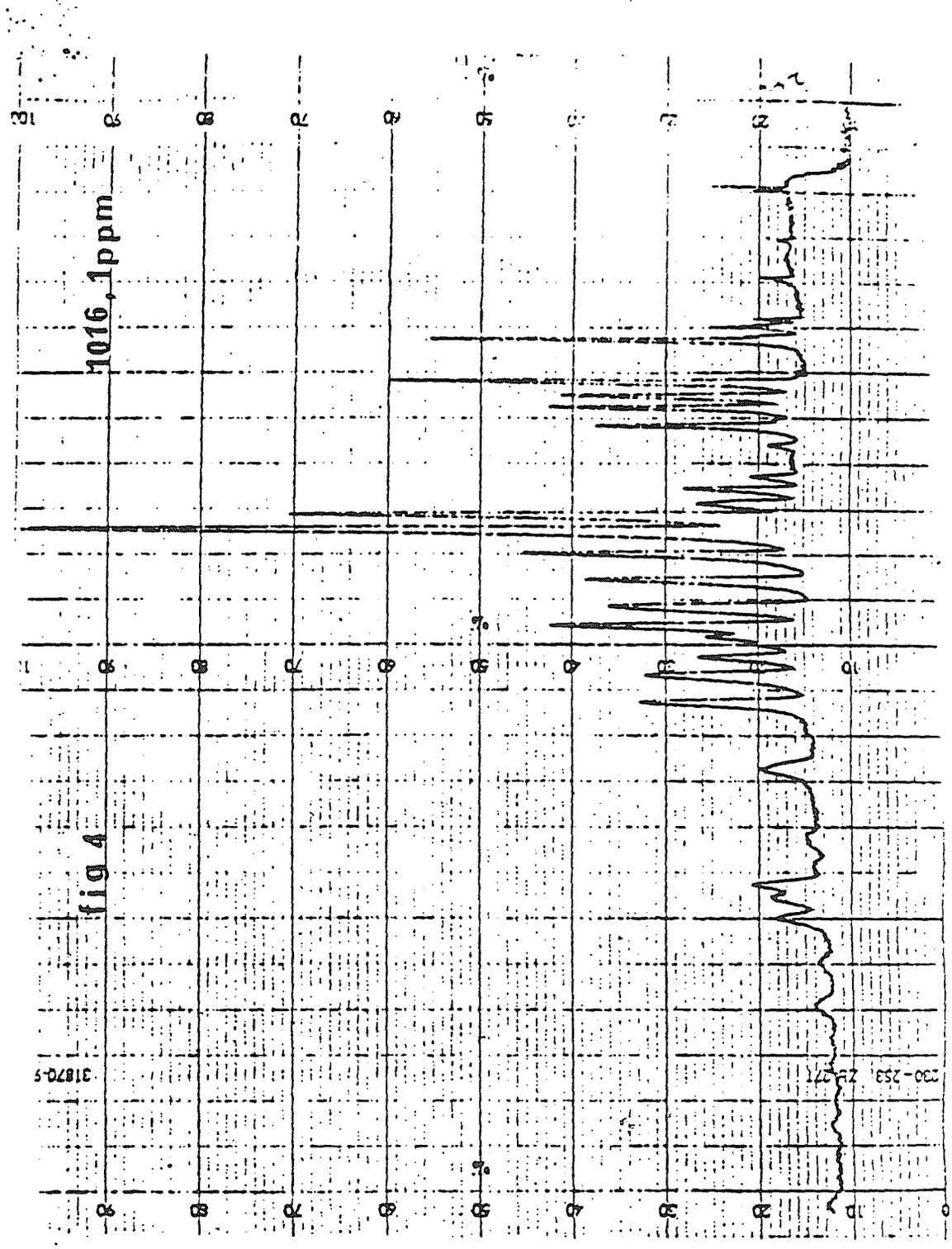


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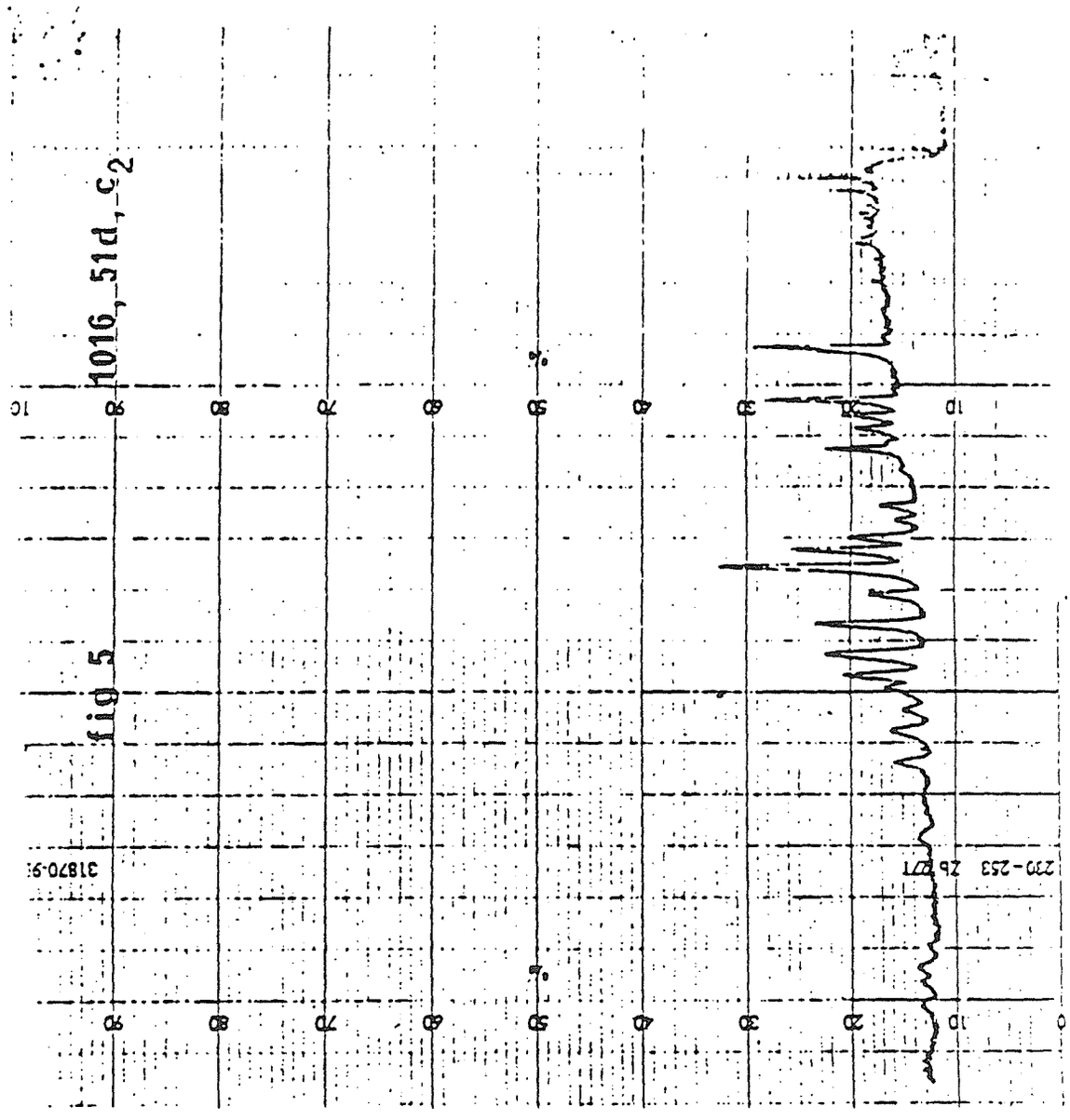


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fig 6

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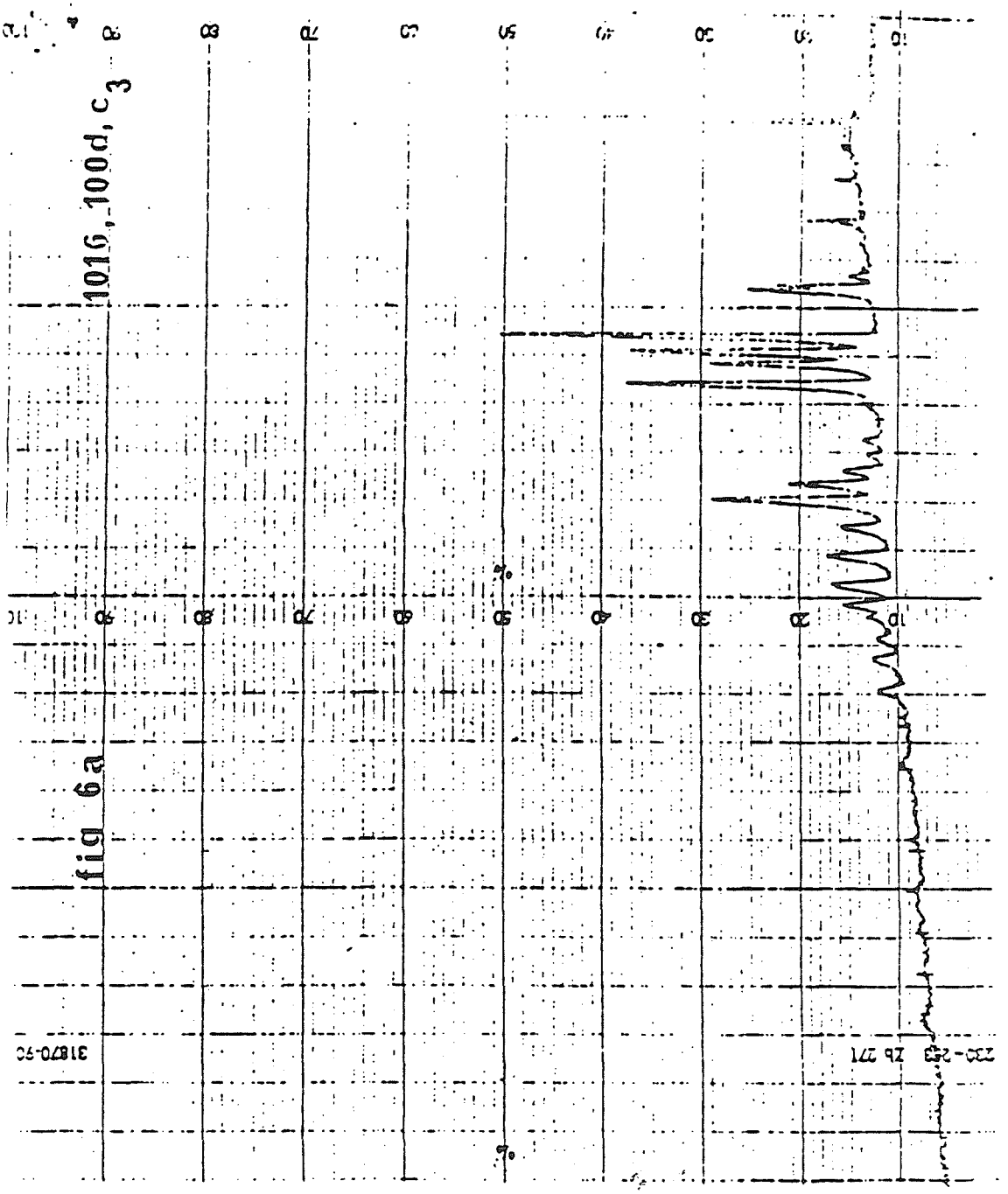


fig 6a

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fig 11

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with added $\phi_2$




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THE PCB-POLLUTION PROBLEM
January 21 and 22, 1970
St. Louis Meeting With General Electric Co.

GENERAL ELECTRIC REPRESENTATIVES:

Mr. Edward L. Raab, GE Pittsfield, Mass., representing all GE locations

Mr. H. Gerade, GE toxicological consultant

Dr. K. Murphy, GE Schenectady, New York, Environmental Pollution Control

MONSANTO REPRESENTATIVES:

H. S. Bergen, D. A. Olson, E. P. Wheeler, Dr. W. R. Richard, Dr. R. H. Munch, Dr. R. Keller, Dr. S. Tucker, W. B. Papageorge, J. G. Bryant, P. G. Benignus

A. Presentation and Discussion of Published Articles About Chlorinated Aromatic Hydrocarbon Insecticides, (DDT etc.) and PCBs

Mr. Wheeler presented to Mr. Raab a booklet containing most of the pertinent publications, to date, and indicated that additional articles will appear shortly.

He mentioned that manufacturers of DDT and chlorinated aromatic hydrocarbon insecticides will tend to emphasize the finding and interference of PCBs as the Government hearings limiting or banning use of the insecticides are held.

This lead GE to seek understanding of the scope, reproducibility, reliability and validity of the analytical procedures used by various investigators who reported finding PCB in concentrations as low as parts per billion.

B. The Analytical Procedures

Drs. Keller and Tucker presented details of Monsanto's GLC - Mass Spectrometric analytical capability and apparatus, as portrayed in Table 1. The sophistication of our analytical capability was emphasized to assure that our approach is the ultimate and is not surpassed. On this basis our views of the validity of results given in various publications are indicated in the attachments to Table 1.

General Electric were impressed and completely satisfied with the scope of our analytical capability and work.

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DEPOSITION
EXHIBIT
PHS 306
6-12-92

C. Biodegradeability of PCBs

Mr. Wheeler related that while Aroclor 1254 and 1260 are being found, especially in aquatic environment, the lower chlorinated biphenyls are not being observed. It is anticipated that the lower chlorinated members may be largely biodegradeable.

Drs. Richard, Keller and Tucker discussed biodegradeability studies by Monsanto at Ruabon and elsewhere.

These studies appear preliminary and not conclusive. Much more needs to be done in this area to allow GE to draw conclusions.

Since the literature indicates that trichlorobenzene and tri-tetrachlorobenzene are not susceptible to biodegradation, GE are intrigued with the absence of reports about finding these materials in the environment. The obvious implication is that since these materials are used with the higher chlorinated PCBs found -- the source of the latter is not from dielectric fluids. However, it was reasoned that chlorobenzenes may not remain due to their relatively higher vapor pressure or may not be found to date because they have not yet been zeroed in to the analytical spectrum.

D. Status of Aroclor Studies At Industrial Bio-Test. Table 2.

In essence results reported by Mr. Wheeler on chronic animal toxicity tests and animal reproducibility studies underway are not as favorable as we had hoped or anticipated. Particularly alarming is evidence of effect on hatchability and production of thin egg shells regards white leghorn chickens. The studies involved Aroclor 1242, 1254 and 1260. Some of the studies will be repeated to arrive at better conclusions.

E. The Location of Askarel Transformers. Table 3.

Messrs. Raab and Benignus formulated Table 3 to portray the use and locations of askarel transformers throughout industry and our commercial and residential areas.

On discussing these askarel transformer applications Mr. Raab was most impelling and forceful about the non-replaceability of transformer askarel fluid and the critical or essential use and need for askarel transformers, which have safety from fire as their outstanding virtue. Without dwelling on details and instead carried to the ultimate, the concensus is that without availability of askarel transformers large cities like New York would be shut down with no power. Certain industries that rely mainly on askarel transformers would go down with no power. Without Aroclor capacitors most of the lights across our country would go out and motors in air conditioners and many industrial applications would not run.

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Frankly, no one could think of a suitable replacement for transformer askarel fluid. Fortunately, Aroclor 1242 with promise about biodegradeability is used in most all of the Aroclor type capacitors. Moreover, possible favorable isomer rearrangements are foreseen in the case of Aroclor 1242.

Need to control, accumulate and properly dispose of scrap askarels is unquestionable in light of the PCB pollution problem, and essential toward maintaining the use of askarel dielectrics. At GE alone apparatus in which askarel fluid is used represents 100 million dollars annually. About 60% is in the capacitor area and 40% represents transformers.

GE requested and we were pleased to give Dr. Murphy their Environmental Control man, a list of all GE and other locations receiving Pyranol shipments in 1969. This amounts to about 16 million pounds of askarel fluids with economic worth of near 2.5 million dollars. This listing included 244 different locations of which 115 were GE plants and service shops scattered throughout our country.

Of course in addition to dielectric use in hermetically sealed capacitors and transformers major amounts of PCBs are used as Plasticizers, Industrial Hydraulic Fluids and Heat-Transfer media.

F. Environmental Sources of PCBs From Dielectric Applications

1. Spills
2. Disposal of waste
3. Ultimate disposal of product -- for failed apparatus
4. Ventilation of operation for employe protection
5. Waste from containers
6. Field on service failures
7. Repair and return apparatus "service shops"

G. Considerations of Degradation Disposals

1. PCBs up to and including 3 chlorine atoms appear biodegradeable in preliminary laboratory work.
2. Thus far there is no evidence that higher chlorinated biphenyls will biodegrade.

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3. According to the literature TCB and TTCB biodegrade at a very slow rate.

4. Chemical, catalytic breakdown would probably require high temperatures.

5. Incineration will require 800°C. and 5 second sojourn time. HCl scrubbing would be required.

H. Estimated Annual Amounts of Contaminated and Scrap PCBs From The Electrical Industry

1. From The Transformer Industry:

a) In plant and field spills are small and controllable with adsorbents, which should be incinerated.

b) Near 2 million pounds a year of transformer askarels are sold to service and repair shops. These people do not manufacture new transformers, although on occasion they may fill new transformers sent into the field without fluid. As these service shops are devoted primarily to repairing faulty transformers, we can assume that as much as 1.0 million pounds annually of "scrap" is generated. Most of this has been dumped or disposed of in streams.

c) We estimate that probably 150,000 pounds of this is arced beyond reworking and needs to be incinerated. The remainder may be reworkable by distillation.

2. From The Capacitor Industry:

a) Collectable waste from normal capacitor impregnation operations amounts to about 850,000 pounds annually. Most of this should be reworkable via simple take-over distillation.

b) Scrap, badly contaminated with polypropylene, epoxides, solvents, oil, grease and "junk" is generated at not over 50,000 pounds a year. This material should be incinerated, along with the 150,000 pounds of scrap from transformers.

c) Power capacitors are designed to last over 30 years. Modern motor runs may last 10 years and the small lighting ballast usually last not over 10 years.

Eventually and cumulatively there is a large potential of field-failed capacitors. Fortunately Aroclor 1242 has been used almost exclusively since about 1950.

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The failed units are disposed of in industrial dumps. In case of ballasts the Aroclor capacitor along with the transformer imbedded in asphalt or encapsulated in epoxy resin, all encased in a metal box are discarded as a unit. This is emphasized to indicate that incineration of such apparatus is not applicable.

3. From Containers:

Most askarel moves in bulk, tank cars or tank wagons, which do not present a problem.

It is questionable that drum shipments may present a problem. The drums can be used for returning scrap.

I. Monsanto's Program To Handle Scrap

1. GE asked what is Monsanto's plan about reclamation of scrap PCB from GE plants, service shops, utilities, industrial users, commercial users, etc.?

2. What is our view about a "Buy-Back" arrangement?

3. What arrangements will Monsanto make for incineration? Disposal in suitable land-fills? Reclamation by distillation at Monsanto?

4. To date Monsanto's posture is:

a) We have taken "good quality" scrap from GE's capacitor plant at Hudson Falls and had it reworked by simple filtration at Findett. This Findett arrangement is not practical nor economical. We paid GE 1 ¢/lb. for this "quite good" Aroclor, plus 1.9 ¢/lb. freight. Findett charged 2.1 ¢ to filter this material, making our cost 5 ¢/lb.

b) 130,000 pounds of somewhat lower quality scrap from Westinghouse capacitor plant has accumulated at Findett for lack of distillation equipment.

c) Scrap from GE's Ft. Edward plant appears to need reprocessing by distillation.

d) About ½ million pounds of "bad scrap" from GE Hudson Falls, has been disposed to a land-fill in New Jersey. Monsanto paid half of the freight cost.

e) A car load of scrap transformer askarel from Westinghouse, South Boston, Virginia is being sent to W. G. Krummrich for reprocessing.

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f) We advised Westinghouse, Sharon to incinerate 12,000 gallons of oil contaminated askarel.

g) We talked with Sangamo, Picken, S. C. and strongly urged them to discontinue present disposal.

5. The above "take-back" arrangements have been made on an individual and experimental basis. We have no fixed "buy-back" arrangement, regards answering GE's question.

6. We have no established process for reclaiming either capacitor or transformer scrap.

7. We have no incinerator for disposal of totally unreclaimable material.

8. To date and for the foreseeable future our only effective disposal is to a land-fill. While this is not desireable, it is better than indiscriminate dumping.

J. Transformer Askarel Blends Discussed With GE. Table 4.

Table 4 lists the transformer askarel blends reviewed with GE.

1. GE Rome has discontinued use of Blend A due to combustibility of arc formed gas. For this same reason, as dictated by their legal people they will not use Blend B.

2. Today both GE, Pittsfield, Mass. and Rome, Ga. use Blend C (Pyranol A13B3B), which they mix themselves.

3. Blend D optimizes the Aroclor concentration in conformance with GE's requirements for non-combustibility of the arc formed gas and with the pour point requirements. The price is in direct conformance with our previous quotation for Aroclor 1242, when GE was using this at a concentration of 80% by weight.

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Accordingly Blend D meets GE's requirements, although containing 71.6% of the higher chlorinated Aroclor it does not conform with Monsanto's concern with the PCB pollution problem.

4. For all practical purposes Blend E (Inerteen 70-30) is identical properties with Blend D, which latter GE, Rome, Ga. proposes to use. In the interest of standardization, we seek to have GE, at Pittsfield and Rome, Ga. use Blend E, which is used by Westinghouse for low temperature applications and is used by many other askarel transformer manufacturers for general application.

5. Although Westinghouse USA uses Blend F (100% Aroclor 1242) for all applications, except for low-temperature application, GE does not accept this use of 100% Aroclor 1242 in transformers.

GE will not accept Aroclor 1242 or any other blend with less than a 1 to 1 ratio of chlorine to Hydrogen atoms. A ratio significantly lower than 1 to 1 tends to yield combustible arc-formed gasses. GE feels strongly that this does not conform with the original definition of an askarel. Accordingly their legal people stress that this presents potential liability in case of an accident for which various precedents have already been set in court actions.

Mr. Raab points out that this precedent for liability regards combustibility is already set, in contrast with the PCB pollution situation which thus far is void of legal actions. He reiterated that the pollution problem thus far is a source of technical publications and warnings and emphasized that in the case of electrical applications which involve only hermetically sealed apparatus, adequate control should be possible.

K. Facts As Agreed By Those In Attendance

1. $Cl_5$ - $C_{6-8}$ Biphenyls -- found in aquatic eco system in populated and industrial areas.

2. DDT + Cl hydrocarbon insecticides have effect on fish-eating birds (reproduction) leading to possible extinction of some species.

3. PCBs (some) are definitely suspected along with polychlorinated hydrocarbon insecticides.

4. Research data being developed seems to confirm incrimination of PCBs subject to rechecking.

5. Public, political and government pressures stopping some uses of chlorinated hydrocarbon insecticides (PCHI).

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6. PCBs have been brought into hearings proposing restrictions or elimination of DDT, PCHI.

7. As manufacturer of PCHI's fight for their life, attention will be directed to PCBs.

8. To our knowledge PCBs are not being found in terrestrial birds or animals, but DDT and PCHI's are found.

9. PCBs are not being found in the eco system without the presence of DDT or PCHI's.

10. PCHI's residue are found without PCBs.

11. We have no evidence that anyone has found lower PCBs except that Dutch researchers found PCBs in roaches.

12. There is no evidence of natural sources of PCBs nor any source other than industrially produced and used.

13. PCBs in a single dose have a low order of acute toxicity and are no significant problem to rats, dogs, chickens, fish and humans.

14. Based on six months of chronic studies in rats and dogs some PCBs are "moderately" toxic and more so than DDT, but less toxic than some of the other chlorinated hydrocarbon insecticides.

15. Some PCBs affect rat reproduction, (10 ppm. apparent no effect levels).

16. Some PCBs affect leghorn chicken reproduction (100 ppm.). PCBs have no affect level for chickens, estimated 10 ppm.

17. Some environmentalists are claiming that the PCBs pose a threat to humans, are so "philosophizing" in publications.

18. One published page indicates that PCBs are as bad (or worse) than DDT in microsomal enzyme effect. What is significant? Since many chemical compounds react similarly!

19. Human experience in production of PCBs and their use has been favorable. Less than 20 instances of illness known to Monsanto Company since 1940-45. Proper precautions were not always followed.

20. GE Company's use of PCBs as dielectric fluids, has been extremely favorable without illness throughout 40 years.

21. Monsanto Company is convinced that analytical techniques and data from some laboratories investigating PCBs are reliable and that conclusions being formulated are valid.

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22. Monsanto Company has not run duplicate analysis on samples reported in the literature.

L. What GE Desires

1. GE seeks that Monsanto take no precipitous reaction to the PCB problem that would result in withdrawing supply of Aroclor 1254 or 1260 to GE.

2. The consensus is that no suitable replacement for transformer askarel fluid is foreseen.

3. In event of development of a suitable fire-resistant fluid replacement for askarel, Mr. Raab emphasized that a minimum of 2 years testing work would be required before commercial use could be adopted.

4. In reply to Monsanto's legal question whether with continued use of Aroclor 1254 and 1260 GE would assume sole and complete liability -- Mr. Raab answered, No! To substantiate his reply, Mr. Raab cited case examples involving GE where damages were sought and collected, even though GE was only the third party. He further stated that any arrangement seeking to delegate and confine liability to GE relative to the PCB problem would be worthless.

5. GE seeks to know the magnitude and time of Aroclor price increase that would result if Monsanto discontinues sale of Aroclor for non-electrical uses, or if pollution control expenses warrant a price increase (as anticipated).

6. GE seeks to send a letter to their plants and service shops and major users of transformer askarel, such as the utilities giving notice of the PCB problem and guidance about the most suitable controls.

7. In the case of GE service shops scattered throughout our country it is unrealistic that these people would assume any expense to return scrap to Monsanto.

   The minimum we can expect is that Monsanto pay the freight charges, and reimburse for a suitable container to avoid additional and excessive contamination.

   This emphasizes need for Monsanto to have an effective reclaiming process. Otherwise, the best we can expect from service shops and many other users is to continue to dump in a land-fill and stop discarding into the sewers.

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8. GE realizes that Monsanto is caught between the explosiveness of arc-formed gasses problem and on the other hand the PCB pollution problem. Realizing possible unfavorable outcome of the arc-formed gas problem, GE desires to withhold the PCB problem from involvement at NEMA, ASTM, IEEE, EEI and ASIA at this time, (making it an industry-wide problem) pending better in-depth understanding of the PCB problem.

9. GE strongly seeks to continue manufacture of askarel type transformers, because in many applications this apparatus cannot be replaced with mineral oil nor open dry, nor seal gas dry type units.

   Mineral oil burns. Open dry accumulates dust, lint, moisture and can then fail with explosion and burning. Sealed gas dry types are very expensive, space consuming and very difficult to maintain sealed.

   Transformer design and application is governed by National Electric Code, by local building codes, Fire Underwriters, NEMA, IEEE, ASTM, IEC, ASA, Insurance companies and others. Because of the diversity of organizations involved a directive to discontinue askarel transformer manufacture would assume highly complex proportions.

P. G. Benignus
January 26, 1970

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PCB

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Reference Number MP10103

Security Class B

Type Review Report

TITLE: BIODEGRADABILITY OF POLYCHLORINATED BIPHENYLS

AUTHOR: J.H. Mainprize

JOB NO: 660.10.122.47 REPORT NO: 1 DATE: 2nd December, 1970

ABSTRACT

The degradation of single P.C.B's and complex mixtures was studied using a biphenyl degrading organism - $C_2$. From the results a theory was developed relating degradability with P.C.B. structure.

The relative degradabilities of Aroclor 1242 and potential alternative products was established.

Issuing Authority J.H. Mainprize

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## INTRODUCTION

A review report[1] issued earlier this year gave details of the development and application of laboratory techniques for the study of P.C.B. biodegradation. Using these techniques, little evidence was obtained of the microbial breakdown of P.C.B's when present as sole carbon sources. It was shown, however, that if unchlorinated biphenyl was included in the shake flask substrates then degradation of certain single isomers could be triggered or accelerated. A similar effect was observed in the case of Aroclor 1242 where a reduction in concentration occurred of P.C.B's associated with two predominant peaks in the gas chromatogram. The mechanism by which biphenyl influences P.C.B. breakdown is not known but it has been suggested that it may be analogous to the phenomenon of co-oxidation which has been shown by Raymond[2] to occur in the degradation of hydrocarbons.

Whilst there is no evidence to support the suggestion that P.C.B. degradation proceeds by a similar mechanism in the natural environment, it was felt that the inclusion of biphenyl in culture media would form the basis of a sorting test and thus assist in correlating P.C.B. structure with potential degradability.

For all the earlier work[1] substrates were inoculated with a suspension of activated sludge taken from the Ruabon biological treatment plant. Before use in the P.C.B. studies the inocula were normally preadapted to biphenyl degradation.

In February of this year a letter was received from Prof. W.C. Evans, Head of the Department of Biochemistry and Soil Science at Bangor University, N. Wales, in which he stated that they had isolated, in pure culture, a biphenyl degrading organism, $C_2$, from a soil source. In view of the then considerable publicity being given to environmental pollution by P.C.B's, Prof. Evans was keen to examine the action of the biphenyl organism on chlorinated derivatives, and asked whether we could assist by providing suitable compounds. A meeting was arranged with Prof. Evans at which we were given a sample of the culture for our own trials. A stock of active inoculum was grown on biphenyl in the laboratory and used exclusively for the work described in this report.

## SUMMARY OF RESULTS

1. The biodegradation of single P.C.B. isomers and complex mixtures has been studied using a standardised shake flask procedure.

2. Results for single compounds and simple binary mixtures have indicated a correlation between biodegradability and P.C.B. structure. Theory based

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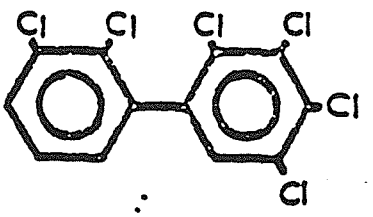
on this correlation suggested that the most highly chlorinated degradable P.C.B. isomer would have the following structure -




This isomer was synthesised and shown to be degradable only when present in the substrate with lower chlorinated isomers, and with biphenyl also present.

3. Long term work with Aroclor 1242 and related products has shown a high degree of degradation when biphenyl is included in the substrate. For the M.C. products the indicated order of degradability is -

MCS 1016 > MCS 717 > MCS 762 > Aroclor 1242

4. Little degradation of Aroclor 1242 was achieved in river water die away tests over a period of 180 days. Breakdown was again improved by inclusion of biphenyl initially in the substrate.

5. H.B.40 is degraded by $C_2$ but again the extent of degradation is increased by initial addition of biphenyl.

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## DETAILS

### 1. Experimental Procedure

#### 1.1 Preparation and maintenance of stock culture of biphenyl-degrading organism

A stock of active organisms was prepared and maintained using a procedure recommended by Prof. Evans. Basically this consisted of initially inoculating a prepared growth media (see Table 1) with the culture provided. The total mixture was aerated continuously in the dark at a temperature of 25°C using scrubbed and filtered air. When inocula were subsequently required for experimental substrates the air flow to the stock culture was stopped and after a brief time to allow excess biphenyl to settle, the required volume of supernatant was decanted into a separate vessel. Some difficulty was experienced in the initial work in avoiding carry-over of suspended biphenyl. Experience showed, however, that this could be overcome by careful manipulation of the stock culture during transfer.

When only relatively small volumes of the stock culture were required for experimental work (i.e. one or two litres) the bottle was replenished by addition of an equal volume of a solution containing the inorganic constituents of the growth medium. Where larger volumes were used, the practice was adopted of starting a fresh culture using new media which was inoculated with organisms reserved from the previous stock.

TABLE 1. MEDIA FOR CULTURE OF BIPHENYL-DEGRADING ORGANISM

| Constituent | Wt. per litre gm. |
|---|---|
| $(NH_4)_2SO_4$ | 1.0 |
| $KH_2PO_4$ | 1.0 |
| $MgSO_4.7H_2O$ | 0.5 |
| $FeSO_4$ | 0.05 |
| Biphenyl | 1.0 |

The media was prepared in 15 litre quantities. On the advice of Prof. Evans, no trace elements were added as he considered that these were adequately supplied by impurities in the major chemical constituents. The pH of the media as prepared was about 5.8 and was brought to 7 by addition of NaOH solution before introducing the inoculum.

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### 1.2 Basic procedure for studying P.C.B. degradation

The basic procedure was essentially as described in the earlier report[1] and is shown in diagrammatic form in Fig. 1. The degradation of each compound of interest was studied alone and in the presence of added biphenyl and control flasks were included in which biological action was prevented by the addition of mercuric chloride (100 ppm. $Hg^{++}$). For the longer term work where several three flask sets of the same P.C.B's were set up, further additions of biphenyl were made to those substrates initially containing that material.

Residual P.C.B's were recovered, as before, by extraction into n-heptane, the extracts being submitted to Physical Methods Group for analysis. Details of analytical procedures are given in the Appendix.

## 2. Results and Conclusions

### 2.1 Degradation of pure P.C.B. isomers and simple binary mixtures

The overall objectives of the biodegradation studies has been to obtain evidence of the potential breakdown of commercially produced Aroclors and also to assist in the assessment of possible alternative fluids. It was felt, however, that fundamental knowledge of the influence of P.C.B. structure on biodegradability was also required. Since, at the time, it was not possible because of analytical problems, to resolve P.C.B. chromatograms into individual identifiable peaks, it was decided that this type of fundamental information was best obtained by the study of single, or simple mixtures of, known compounds. Samples representing a range of possible P.C.B. structures were therefore assembled from U.S. and U.K. sources. Later, when results indicated that certain structural requirements were necessary for biodegradation to proceed, more highly chlorinated key compounds were synthesised in the Ruabon Research Laboratory. Detailed results for each isomer or mixture of isomers are given in the following sub-sections.

#### 2.1.1 Degradation of 2,4'-dichlor biphenyl

Initial Concentration - 5 ppm  Flasks shaken 7 days

| Substrate | % Recovered |
|---|---|
| P.C.B. + culture | 30 |
| P.C.B. + B.F. culture | Nil |
| P.C.B. + B.F. + $Hg^{++}$ culture | 82 |

Conclusion  Degradable

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2.1.2 Degradation of 4,4'-dichlor biphenyl

Initial Concentration - 5 ppm  Flasks shaken 12 days

| Substrate | % Recovered |
|---|---|
| P.C.B. + culture | 70 |
| P.C.B. + B.P. + culture | 66 |
| P.C.B. + B.P. + $Hg^{++}$ + culture | 72 |

Conclusion Non-degradable

2.1.3 Degradation of mixture of 2,3- and 3,4-dichlor biphenyls

Initial Concentration - 5 ppm  Shaken 3 days

| Substrate | % Recovered | |
|---|---|---|
| | 2,3- | 3,4- |
| P.C.B. + culture | 33 | 20 |
| P.C.B. + B.P. + culture | 36 | Nil |
| P.C.B. + B.P. + $Hg^{++}$ + culture | 82 | 82 |

Conclusion Both degradable 3,4 more readily than 2,3

2.1.4 Degradation of mixture of 2,3,2' and 2,3',4' trichlor biphenyls

Initial Concentration - 2 ppm  Shaken 7 days

| Substrate | % Recovered | |
|---|---|---|
| | 2,3,2' | 2,3',4' |
| P.C.B. + culture | 50 | 6 |
| P.C.B. + B.P. + culture | 5 | Nil |
| P.C.B. + B.P. + $Hg^{++}$ + culture | 90 | 95 |

Conclusion Both degradable 2,3',4' more readily than 2,3,2'

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2.1.5 Degradation of 3,3',4-trichlor biphenyl

Initial Concentration - 5 ppm  Shaken 12 days

| Substrate | % Recovered |
|---|---|
| P.C.B. + culture | 24 |
| P.C.B. + B.P. + culture | 30 |
| P.C.B. + B.P. + $Hg^{++}$ + culture | 98 |

Conclusion Degradable

2.1.6. Degradation of 2,4,6-trichlor biphenyl

Initial Concentration - 5 ppm  Shaken 12 days

| Substrate | % Recovered |
|---|---|
| P.C.B. + culture | 100 |
| P.C.B. + B.P. + culture | 100 |
| P.C.B. + B.P. + $Hg^{++}$ + culture | 100 |

Conclusion Non-degradable

2.1.7. Degradation of 2,2',4,4'-tetrachlor biphenyl

Initial Concentration - 5 ppm  Shaken 9 days

| Substrate | % Recovered |
|---|---|
| P.C.B. + culture | 100 |
| P.C.B. + B.P. + culture | 100 |
| P.C.B. + B.P. + $Hg^{++}$ + culture | 100 |

Conclusion Non-degradable

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2.1.8. Degradation of 2,2',4,6-tetrachlor biphenyl

Initial Concentration - 5 ppm  Shaken 9 days

| Substrate | % Recovered |
|---|---|
| P.C.B. + culture | 94 |
| P.C.B. + B.P. + culture | 92 |
| P.C.B. + B.P. + $Hg^{++}$ + culture | 94 |

Conclusion Non-degradable

2.1.9. Summary of results obtained and suggested correlation between biodegradability and structure

The results given in Sections 2.1.1. - 2.1.8. are summarised in Fig. 2.

Microbiological and analytical work carried out by Prof. Evans at Bangor University[3] has shown that metabolism of biphenyl follows the pathway shown in Fig. 3.

The reasonable assumption that breakdown of degradable P.C.B's follows the same initial steps indicates ways in which microbial attack could be prevented or hindered by chlorine substitution at specific sites in the biphenyl molecule. With the supporting evidence of the results summarised in Table 1 it was theorised that the following basic structural requirements must be fulfilled if, at least, primary degradation of P.C.B's was to occur -

(a) Chlorine substitution at neither or only one of the two para (4 or 4') positions. Supported by failure to degrade 4,4' dichlor biphenyl and 2,2',4,4' tetra chlor biphenyl. Substitution at both 4-positions presumably prevents hydroxylation at the adjacent 2,3 positions and would also interfere with the ring opening step III.

(b) At least two of the four adjacent 2,3 positions must be unsubstituted.

(c) No chlorine substitution at the 2 and 6 positions in the same ring. Supported by failure to degrade 2,4,6-trichlor biphenyl. It is suggested that degradation is prevented by the steric effects of chlorine at the 2 or 6 positions which blocks hydroxylation at the 5' and 6' positions on the unchlorinated ring.

In the absence of other possible factors which could limit biodegradability,

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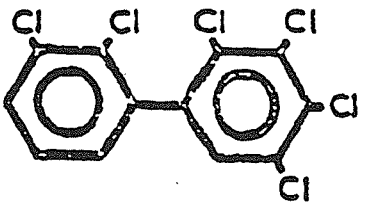
e.g. toxicity, insolubility, the above theorising suggested that the most highly chlorinated biphenyl which could still be subjected to at least primary degradation would have the following structure –




Also the structure of all other degradable P.C.B's can be arrived at by progressively reducing the chlorine content of the above isomer.

To obtain further support for the proposed correlation between structure and degradability, two isomers were synthesised in the Ruabon Research Laboratory, (i) 2,3,3',4,5–penta chlor biphenyl, and (ii) 2,2',3,3',4',5'–hexa chlor biphenyl, i.e. as above.

These were tested for degradability under the standard shake flask conditions. Results after 14 days shaking showed no degradation to have occurred. In the case of the penta substituted isomer, a second set of shake flask substrates were analysed after 33 days agitation, again with negative results.

In discussion of the above results, it was suggested that failure to degrade these key compounds might be associated with their very low solubility and high melting points which prevented effective contact between P.C.B. and organism active centres. Also the gentle agitation given by the oscillating motion of the flask shaker may not have been sufficient to ensure satisfactory dispersion of the evaporated P.C.B. deposit in the culture flask. To overcome these possible limitations, a further run was carried out using the hexa chlor biphenyl mixed in the ratio of 1:1 by weight with the isomeric mixture of 2,3',4' and 2,3,2'–trichlor biphenyls (see Section 2.1.4.). Substrates were prepared in the usual way with a total P.C.B. content of 5 ppm, i.e. 2.5 ppm of hexa substituted isomer but flasks were agitated in a vibratory bench shaker. After 15 days agitation the flask contents were extracted into n–heptane and the abstracts submitted for analysis. Results showed that a 50% loss of the hexa chlor biphenyl was achieved when biphenyl was also included in the substrate; no degradation was observed when biphenyl was excluded. The significant degradation of this previously refractory P.C.B. could have been attributable to either of the changed experimental conditions,



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i.e. improved agitation or solubilisation by mixing with other lower chlorinated isomers. Unfortunately when the same technique was applied to the study of the degradation of the penta compound, completely negative results were again obtained.

To confirm the observed degradation of the hexa compound, the earlier run was repeated but with the effect of agitation also being compared. Results of this run, which are given in Table 2, confirm that significant degradation can be achieved in the presence of the lower chlorinated isomers with agitation apparently not, under the experimental conditions, being a controlling factor. In the light of these results, the failure to degrade the penta chlor isomer cannot be explained and the run is again being repeated. Also the earlier theorising on the influence of P.C.B. structure on degradation cannot be considered proven until it has been demonstrated that degradation of other hitherto intractable isomers, e.g. 4,4'-dichlor- cannot also be induced by mixing with lower melting point derivatives.

TABLE 2. DEGRADATION OF 2',3',4',5',2,6-HEXA CHLOR BIPHENYL IN THE PRESENCE OF MIXTURE OF 2,3,2' and 2,3',4'-TRICHLOR BIPHENYLS

Concentrations 2',3',4',5',2,6, hexa chlor biphenyl 2.5 ppm
Mixed trichlor biphenyls 2.5 ppm

| | | Substrate | % Recovered |
|---|---|---|---|
| Shaker 1 | 1. | Hexa chlor biphenyl + $C_2$ | 100 |
| | 2. | Hexa chlor biphenyl + $C_2$ + mixt. of 2,3',4' and 2,3,2' | 100 |
| | 3. | As 2 but with 50 ppm B.P. added initially and after 3 and 11 days | 52 |
| | 4. | As 2 but with 50 ppm B.P. and 100 ppm $Hg^{++}$ added initially | 100 |
| Shaker 2 | 1. | Hexa chlor biphenyl + $C_2$ | 100 |
| | 2. | Hexa chlor biphenyl + $C_2$ + mixt. of 2,3',4' and 2,3,2' trichlor biphenyls | 92 |
| | 3. | As 2 but with 50 ppm B.P. added initially and after 3 and 11 days | 36 |
| | 4. | As 2 but with 50 ppm B.P. and 100 ppm $Hg^{++}$ added initially | 100 |

Shaker 1 High frequency bench vibratory instrument
Shaker 2 Oscillating shaker, as used for all other work.

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## 3. Degradation of complex P.C.B. mixtures (Aroclors and related products)

The degradation of a range of P.C.B. mixtures was studied using the standardised technique with culture $C_2$ as the active inoculum. Also by setting up several three-flask sets which were removed after increasing time intervals, it was possible to obtain some measure of the rate of degradation and to produce base chromatograms representing residual non-degradable P.C.B's. Results are given in Figs. 4 - 21. For convenience of visual comparison, chromatograms were traced on to a single page by hand. Whilst the general forms of the chromatograms are faithfull copies of the originals, excessive levels of recorded 'noise' were deliberately 'cleaned up' for ease of comparison. Brief notes on the significance of the results are given in the following sections under the relevant headings. No attempt has been made to quantify the degree of degradation although it is hoped that this will be possible in the near future with the continuing improvement which is being achieved in higher resolution chromatography.

### 3.1. Degradation of Aroclor 1242 - Figs. 4 and 5

Degradation of 1242 compared after agitation for 37 and 83 days respectively. Results demonstrate accelerating effect which biphenyl (B.P.) has on the rate of P.C.B. degradation.

### 3.2. Degradation of Aroclor 1242 - Figs. 6, 7 and 8

Three-flask systems set up in quadruplicate. Extracted after 13, 38 and 73 days respectively. Biphenyl additions made to relevant flasks after increasing time intervals. Because of instrument problems, the chromatograms of the 38 day samples were recorded at half the attenuation used for the 13 and 73 day extracts. The results indicate that no further degradation occurred after 73 days.

### 3.3. Degradation of Pyralene 1500 - Figs. 9 and 10

Pyralene 1500 is a competitive blend of polychlorinated biphenyls manufactured by Prodelec of France. In this run, provision was made to demonstrate the need for the continuing presence of biphenyl if maximum breakdown of P.C.B's is to be achieved within reasonable contact times. The results unfortunately are anomalous in that greater degradation occurred in the flask containing no added biphenyl than in that to which biphenyl was initially added (Fig. 10, Traces 2 and 3). This anomaly could be explained by accidental interchange of samples at some stage between substrate preparation and extraction but there is no evidence to show that this occurred. However, comparison of chromatograms 2 or 3 in Fig. 9 with chromatogram 4 indicate that degradation was enhanced in the latter by the second addition of biphenyl made after 34 days.



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### 3.4. Degradation of Aroclor 1242 - Effect of Agitation - Fig. 10

When visiting Prof. Evans' laboratory at Bangor University, it was noted that, for degradation work culture flasks were agitated in a high frequency vibratory shaker. It was suggested that this type of agitation might cause better dispersion of liquid P.C.B's and Aroclors and hence more effective contact with the organisms. As reported earlier in Section 3.4 it was shown that for simple mixtures the degree of agitation had no effect on the rate or extent of degradation. This was confirmed for Aroclor 1242 by the results shown in Fig. 11.

### 3.5. Degradation of MCS-717, MCS 762 and MCS 1016 (1242 B) - Figs. 12 to 14, 15 to 17 and 18 to 20.

This run was set up simultaneously with that for 1242 described in Section all flasks being treated similarly with respect to agitation time and frequency of biphenyl addition. Visual comparison of the chromatograms therefore gives some indication of the relative biodegradability of each P.C.B. mixture. For convenience of comparison, chromatograms representing the highest degree of degradation for each product are shown in Fig. 20.

Assuming that peak areas can be related quantitatively to the concentration of residual P.C.B's, comparison of the chromatograms shown in Fig. 21 indicate the approximate order of degradability to be MCS 1016 > MCS 717 > MCS 762 > 1242. It is hoped that with the continuing progress in higher resolution chromatography and identification of P.C.B's associated with individual peaks, more detailed information will shortly be generated on the nature of non-degradable residues.

One unexplained aspect of this work is the appearance of an additional peak in the chromatograms of non-degraded residues for MCS 762, MCS 717 and MCS 1016. This peak presumably represents the accumulation of some non-degradable breakdown product of PCB's originally present.

## 4. Other biodegradation work

### 4.1. Degradation of HB 40

This material has been shown to be a suitable alternative to Aroclor 1242 for use in the manufacture of no-carbon duplicating paper. Degradation studies were undertaken using the same basic procedure as for P.C.B's and also using the culture $C_2$. Unfortunately at a level of 5 ppm the concentrations of extracts were too low for satisfactory measurement using a flame-ionisation detector. The chromatograms shown in Fig. 22 were obtained by preparing a 10-fold concentrate of the extracts before analysis.

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4.2. River water die away tests

To determine whether micro-organisms populating normal surface waters were capable of long term acclimatisation to P.C.B. degradation, a run was carried out in which the basic procedure was used but flasks were inoculated with fresh samples of water taken from the River Dee. Results in Fig. 23 show that after 180 days contact measurable losses occurred only in that substrate to which biphenyl was initially added.

## 5. Discussion of Results

Work to date has shown that considerable degradation of P.C.B's can be achieved using specific organisms under laboratory conditions. Although these conditions are probably more favourable than those which exist in the natural environment, when results are considered in conjunction with the failure to detect significant levels of lower chlorinated residues in marine life, they lend strong support to the statement that degradation is probably occurring.

The apparent correlation between P.C.B. structure and biodegradability, although not yet proven, should assist in the formation of suitable products although the difficulty of excluding non-degradable isomers may be insurmountable, at least on a commercial basis.

It is now considered that priority should be given to improved analytical evaluation of the nature of non-degraded residues. Considerable progress is being made in improving chromatogram resolution and in identifying individual peaks. Typical recent chromatograms of degraded and non-degraded 1242 are shown in Fig. 24.

## Further work

Prof. Evans has recently isolated, from a soil source, another biphenyl degrading organism with a different metabolic pathway. A stock of this culture is being grown at Ruabon and used to repeat some of the work described in this report.

## References

1. Review Report - Biodegradability of polychlorinated biphenyls. Acc. No. AP32412.
2. Process Biochemistry, September 1969, pp. 71-74.
3. Private communication, Lunt D. and Evans W.C., 1970.

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JHM/JB

J.H. MAINPRIZE

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## APPENDIX

### Gas Chromatographic Analyses

Analyses of extracts of single components, simple mixtures and complex mixtures represented by Figures 4 to 23 were made using a Hewlett Packard 402 chromatograph equipped with a $Ni^{63}$ high temperature electron capture detector. The column consisted of 4' glass tubing packed with 4% Silicone Oil OV101 on 60 - 80 mesh Gas Chrom Q.

Operating conditions were -

Flow 40 ml./min. through column plus 60 ml./min. purge flow in detector. Both of Argon/Methane 95/5.

| | |
|---|---|
| Oven temperature | 170°C |
| Flash Heater | 250°C |
| Detector | 250°C |
| Pulser | 150 m. sec. |

For the more highly resolved chromatographs shown in Fig. 24 and 25, the column consisted of 150' S.S. Capillary coated with Apiezon L.

Operating conditions with E. C. detector -

| | | |
|---|---|---|
| Temperature | 200°C isothermal | |
| Flow | 5 psi through column<br>2 psi purge | 95% Argon/5% Methane |

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FIG 1 BASIC PROCEDURE FOR THE STUDY OF P.C.B. BIODEGRADATION

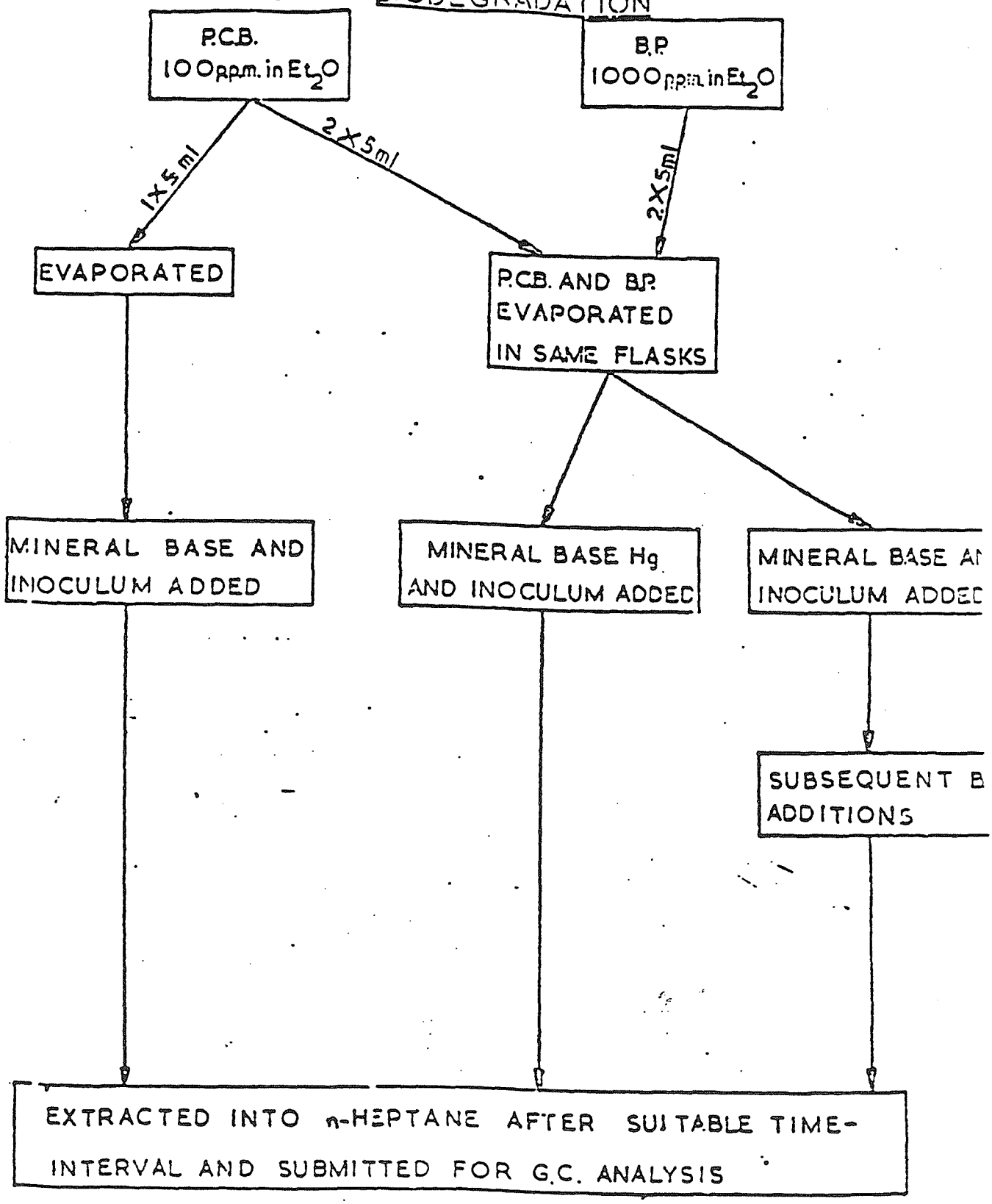


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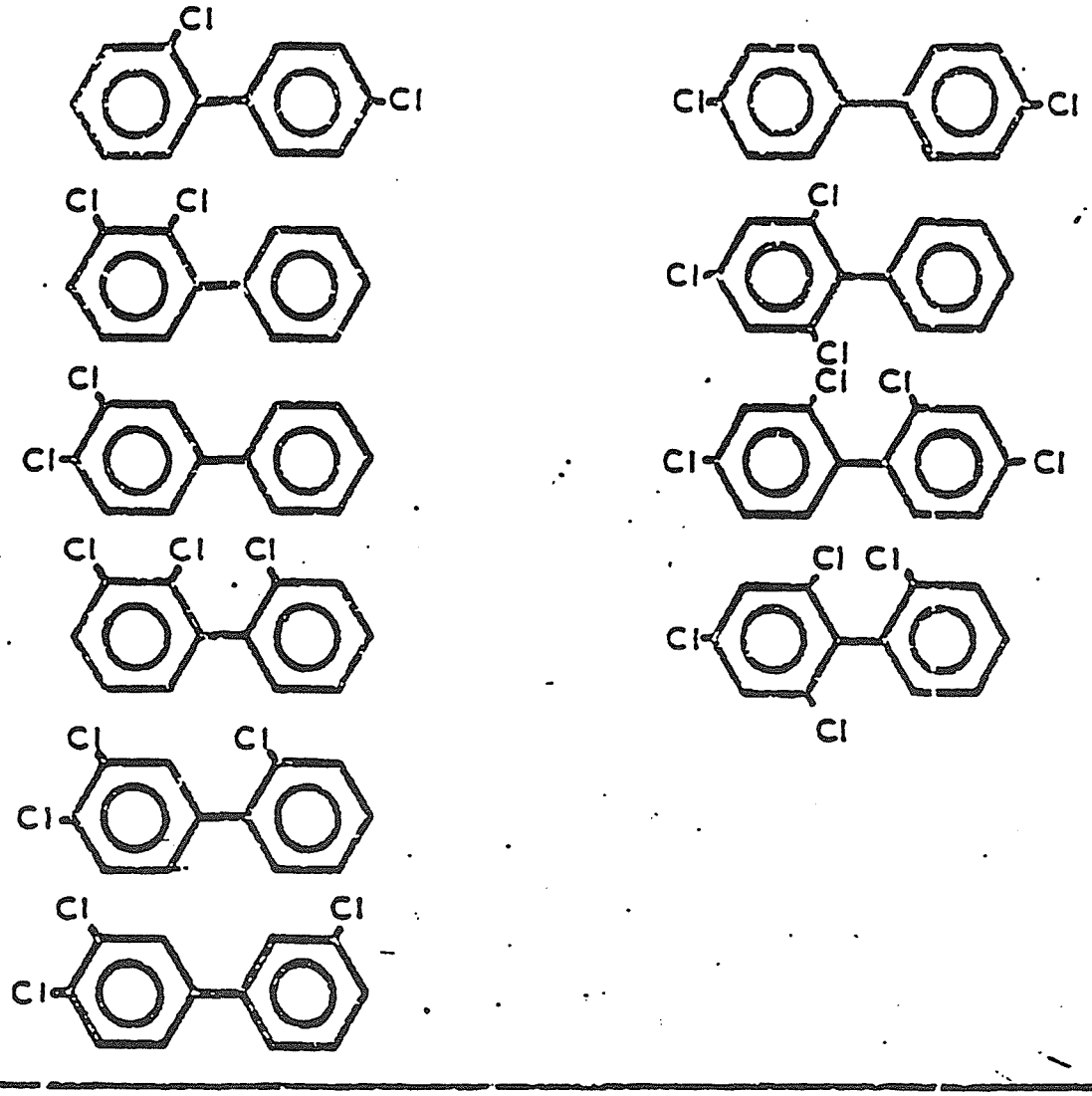
FIG 2 DEGRADABILITY OF SINGLE P( 3. ISOMERS



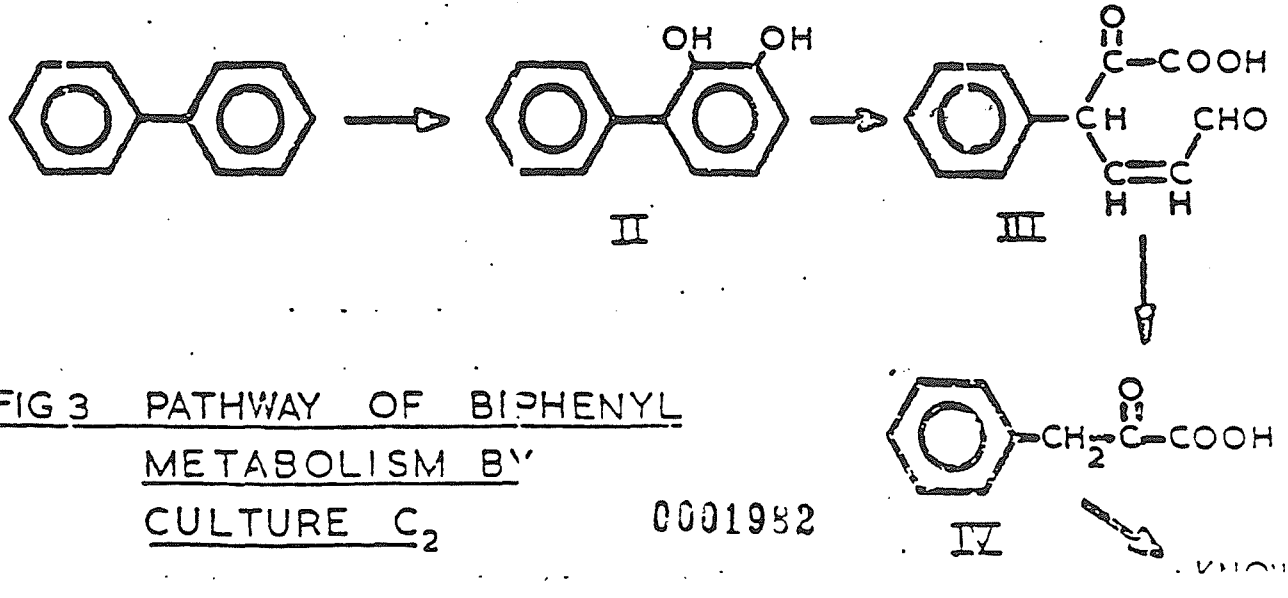




FIG 3 PATHWAY OF BIPHENYL METABOLISM BY CULTURE $C_2$

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FIG4 DEGRADATION OF AROCLOR 1242 BY CULTURE $C_2$

1. CONTROL ie AS 2 BUT WITH 50 P.P.M. B.P AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. AROCLOR 1242 + $C_2$ SHAKEN 37 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P ADDED INITIALLY AND AFTER 9 DAYS

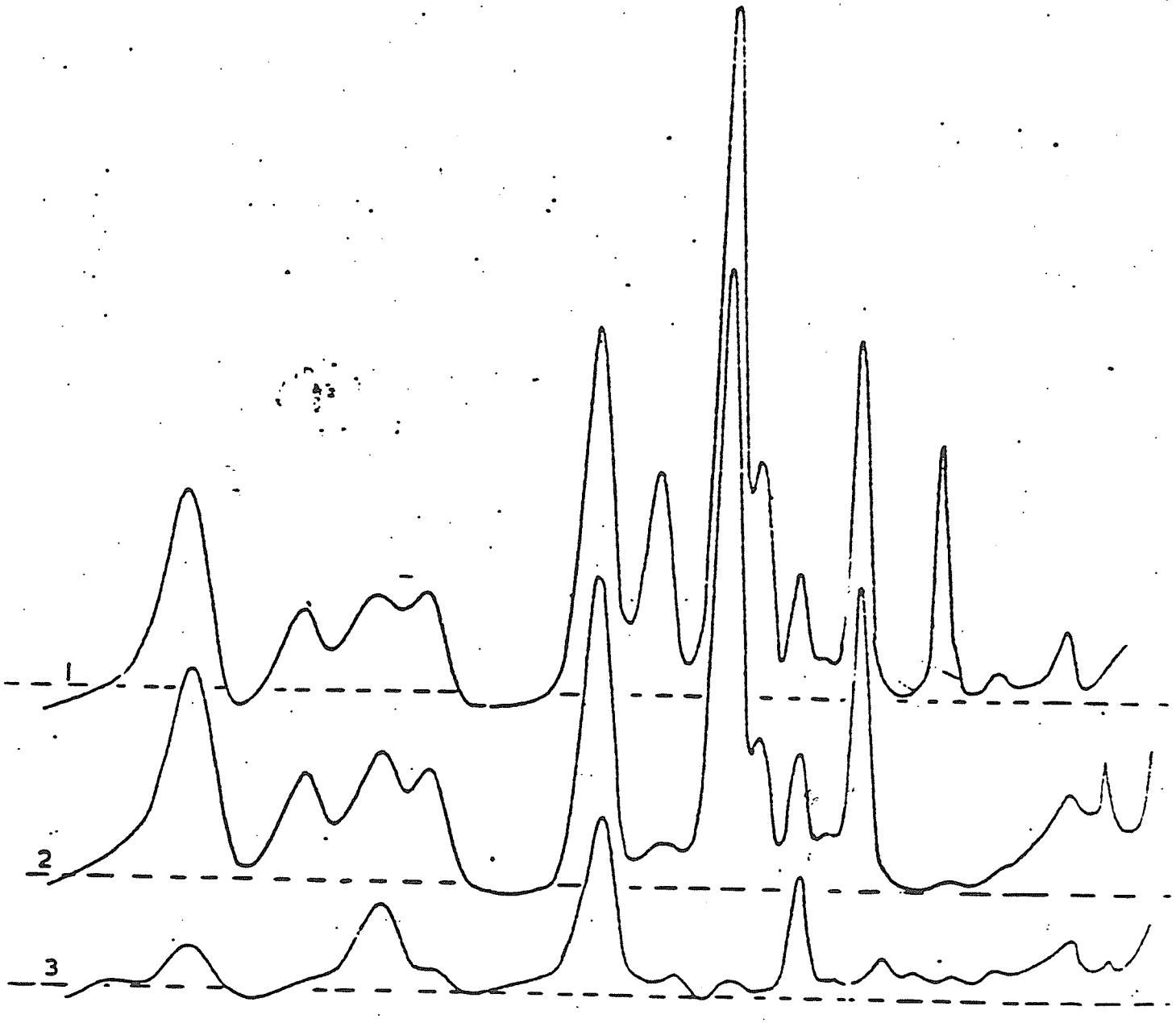


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FIG 5 DEGRADATION OF AROCLOR 1242 BY CULTURE $C_2$

1. CONTROL ie AS 2 BUT WITH 50 P.P.M. B.P AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. AROCLOR 1242 + $C_2$ SHAKEN 83 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P ADDED INITIALLY AND AFTER 9, 36, & 49 DAYS

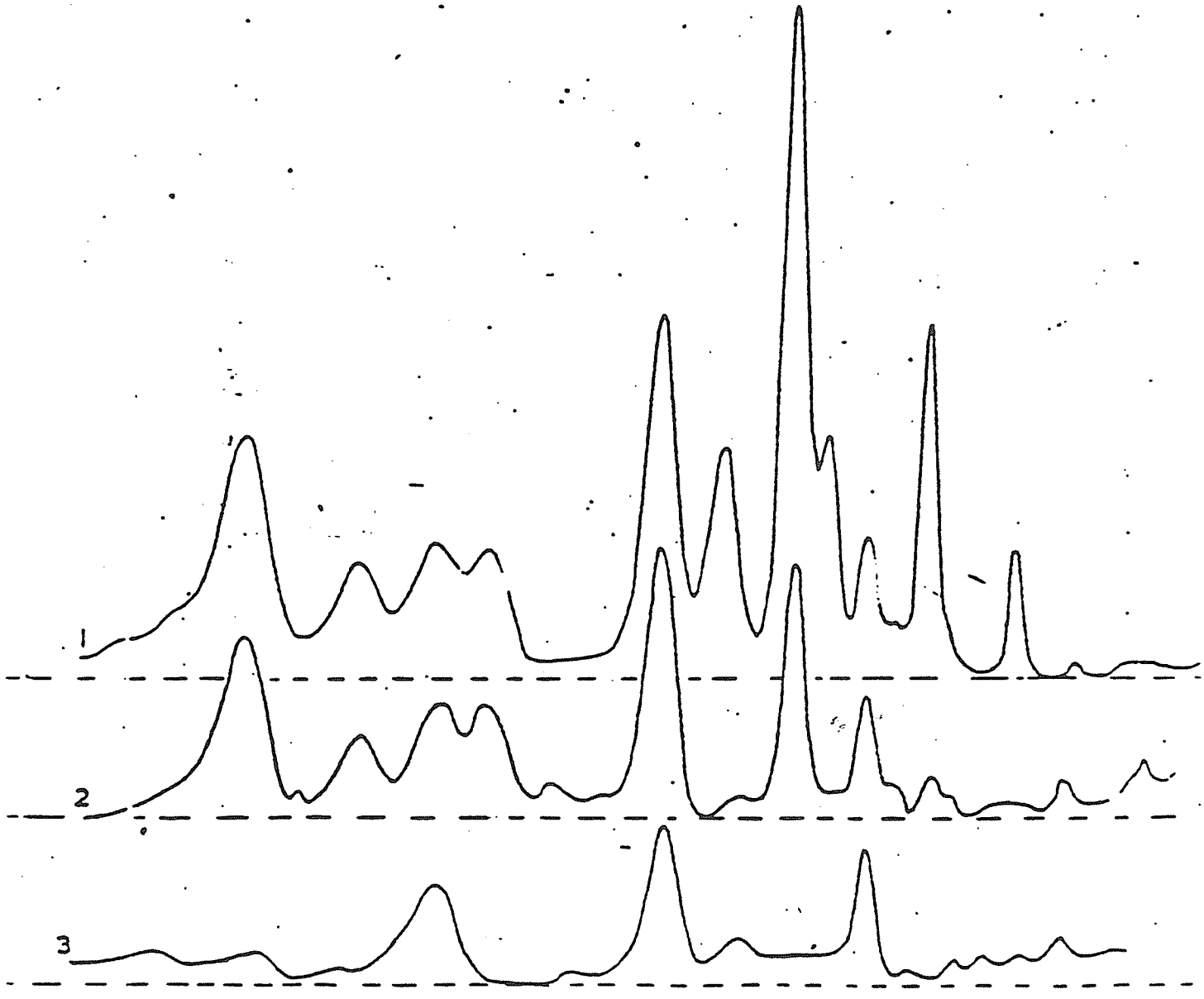


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FIG 6 DEGRADATION OF AROCLOR 1242 BY CULTURE $C_2$

1. CONTROL I.E. AS 2 BUT WITH 50 P.P.M. B.P AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. AROCLOR 1242 + $C_2$ SHAKEN 13 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P ADDED – INITIALLY AND AFTER 3 DAYS

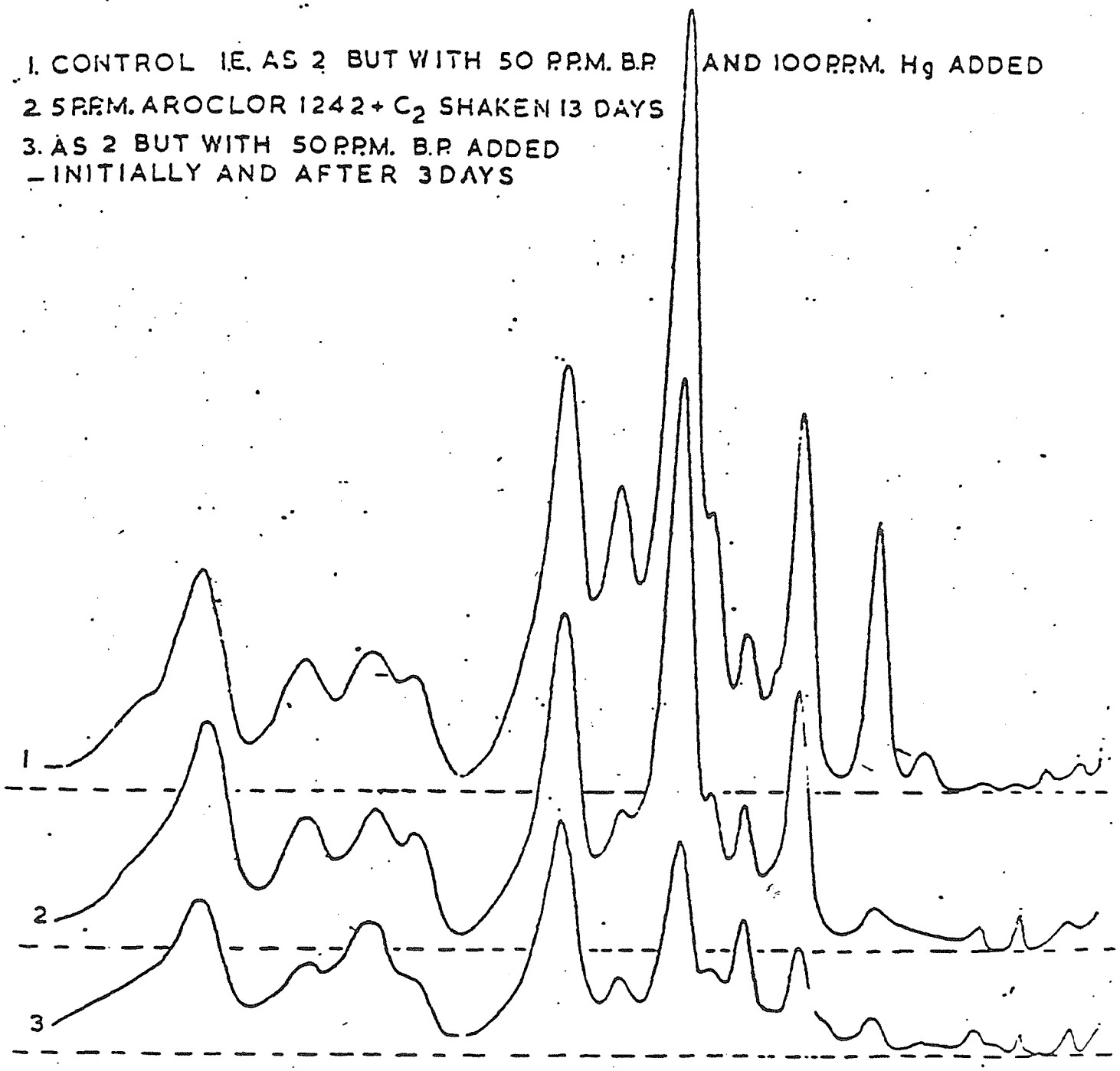


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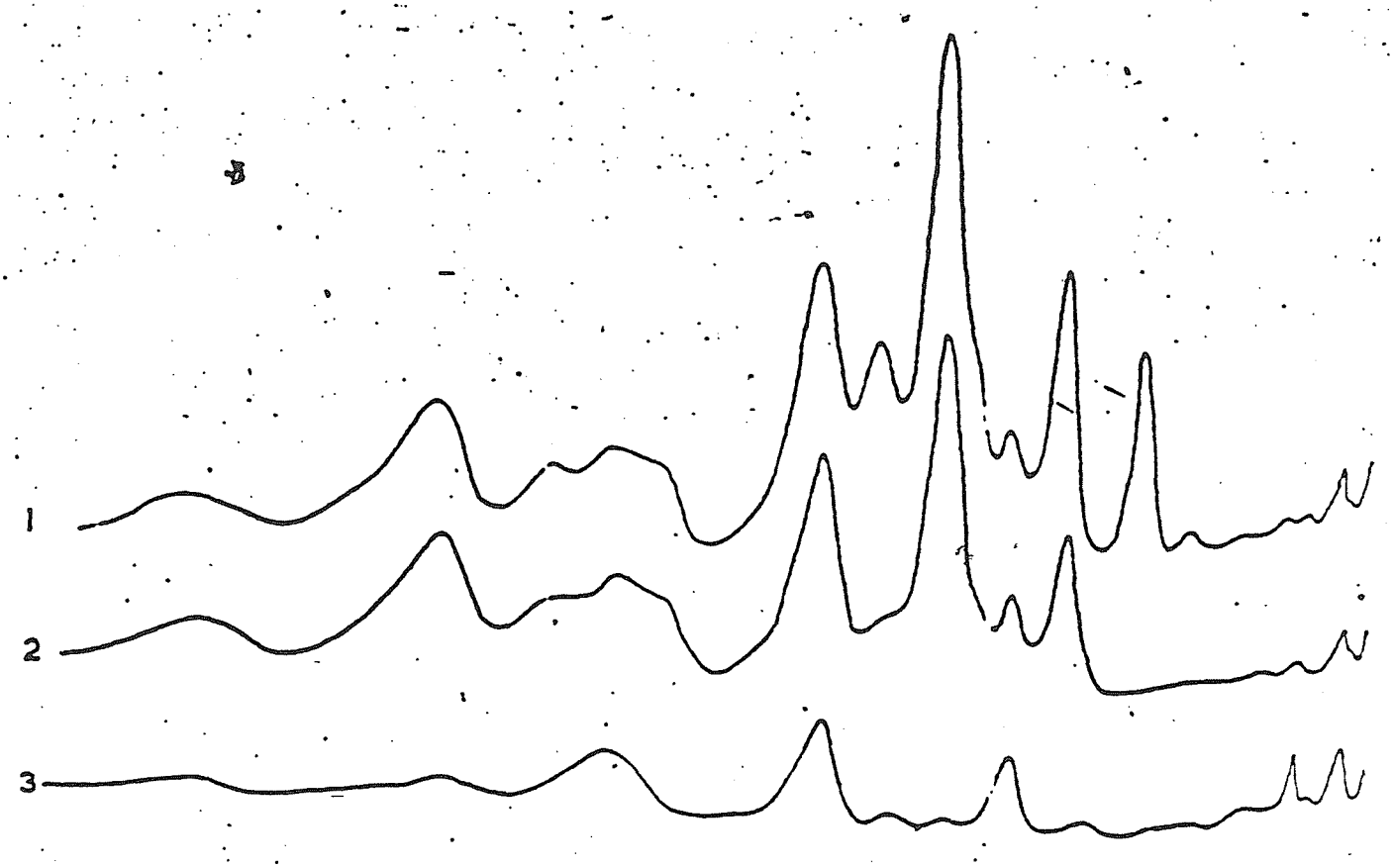
FIG 7 DEGRADATION OF AROCLOR 1242 BY CULTURE $C_2$

1. CONTROL ie. AS 2 BUT WITH 50 P.P.M. BP AND 100 P.P.M. $Hg$ ADDED
2. 5 P.P.M. AROCLOR 1242 $C_2$ SHAKEN 38 DAYS
3. AS 2 BUT WITH 50 P.P.M. BP ADDED INITIALLY AND AFTER 3, 8 AND 18 DAYS

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1. CONTROL ie. AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M. Hg ADDED

2. 5 P.P.M. AROCLOR 1242 + $C_2$ SHAKEN 73 DAYS

3. AS 2 BUT WITH 50 P.P.M. B.P. ADDED INITIALLY AND AFTER 3, 18, 28, 39, 46 AND 67 DAYS

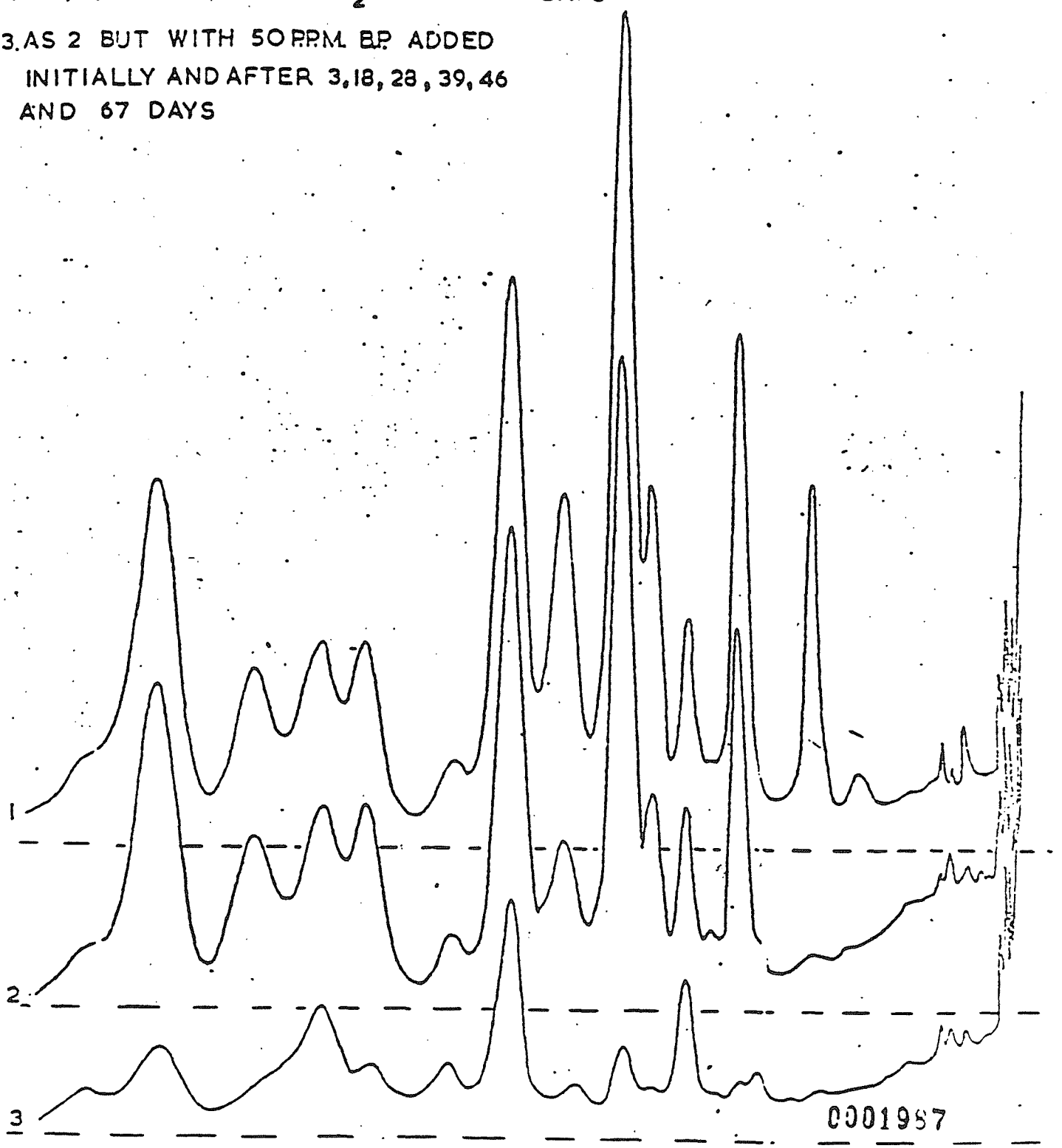


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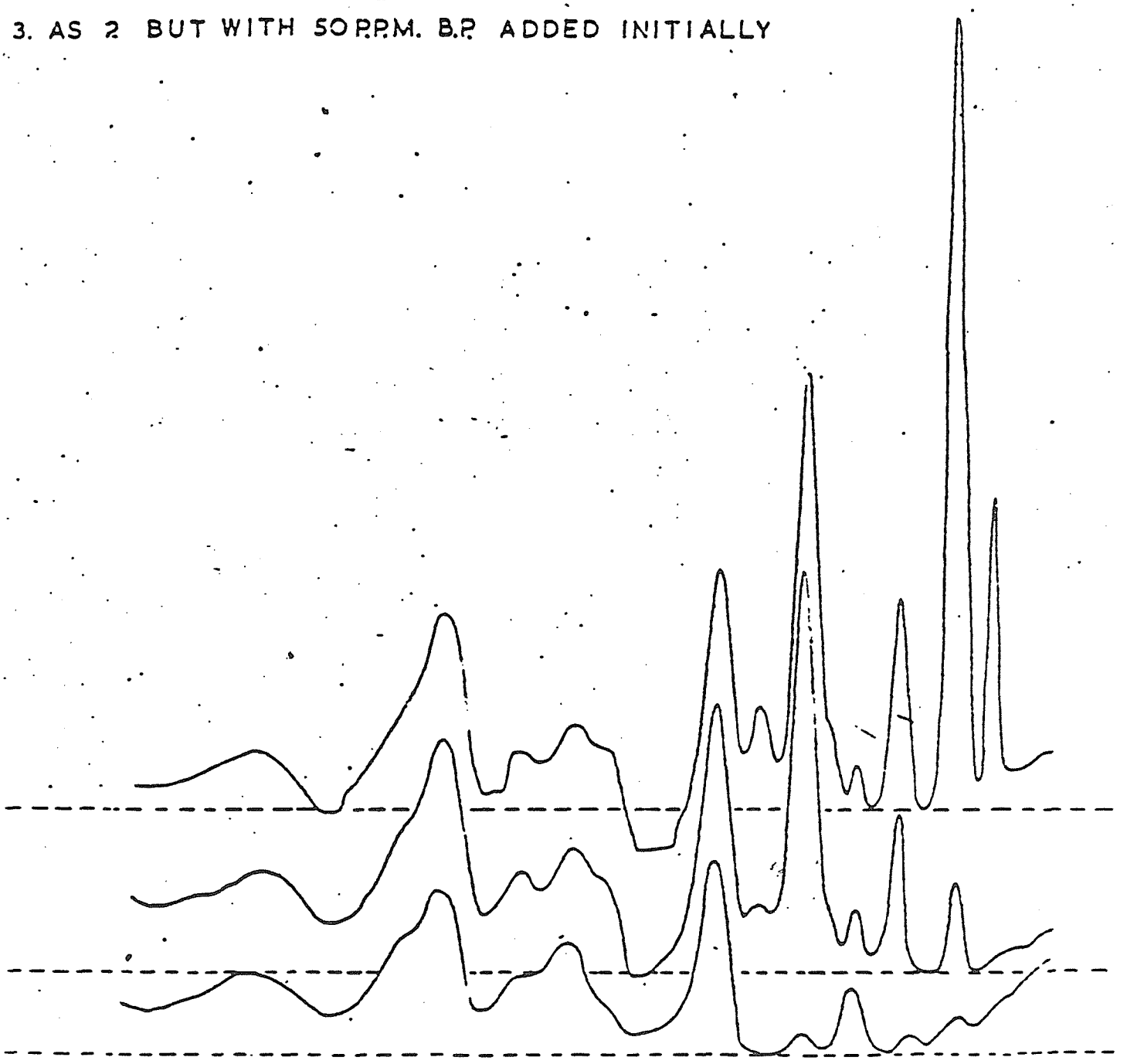
FIG.9 DEGRADATION OF PYRALENE BY CULTURE $C_2$

1. CONTROL Ie AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. PYRALENE + $C_2$ SHAKEN 18 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P. ADDED INITIALLY



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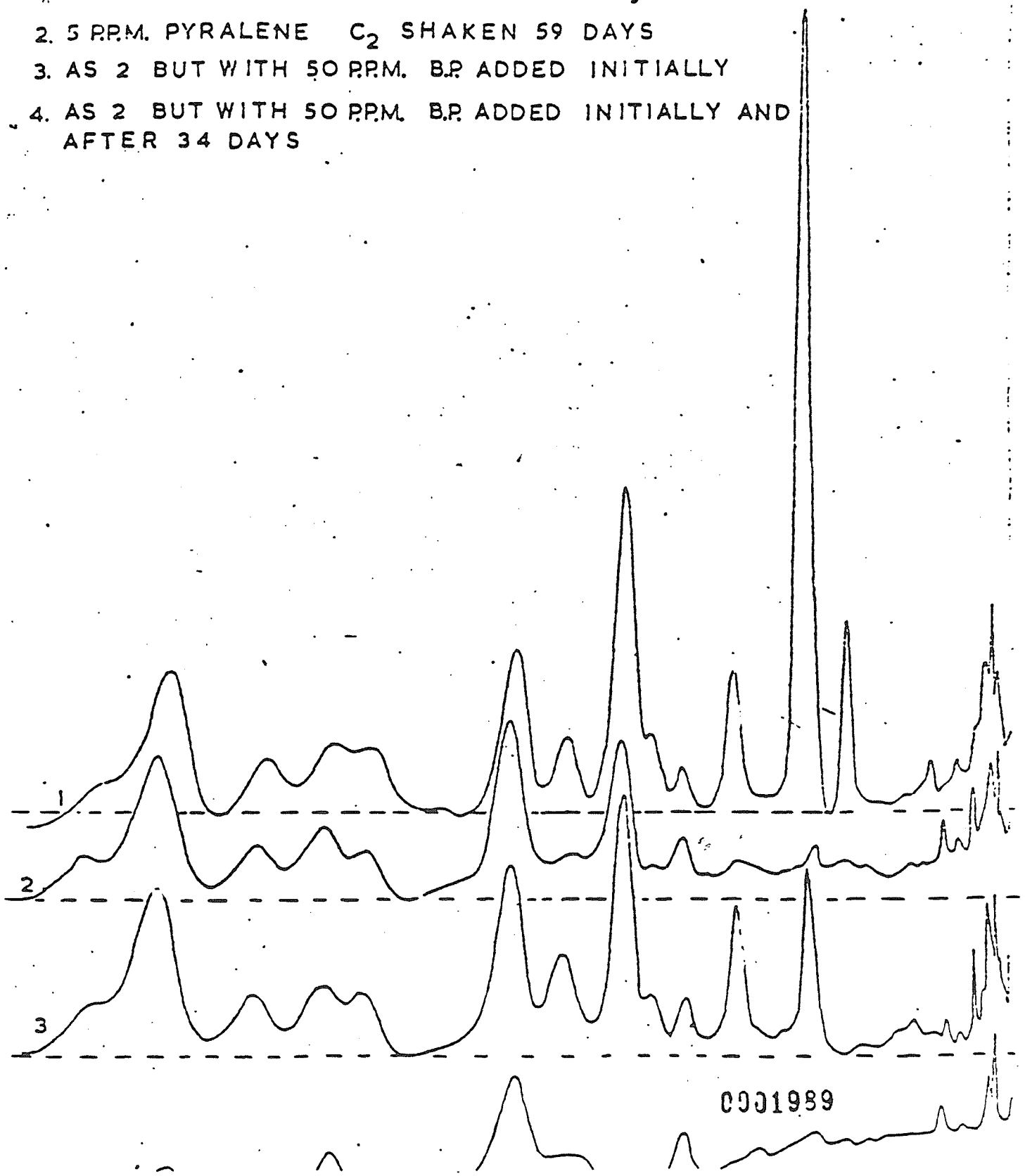
# FIG 1 DEGRADATION OF PYRALENE BY CULTURE $C_2$

1. CONTROL ie AS 3 BUT WITH 100 P.P.M. Hg ADDED
2. 5 P.P.M. PYRALENE $C_2$ SHAKEN 59 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P ADDED INITIALLY
4. AS 2 BUT WITH 50 P.P.M. B.P ADDED INITIALLY AND AFTER 34 DAYS

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FIGII DEGRADATION OF AROCLOR 1242 BY $C_2$ (EFFECT OF AGITATION)

1. CONTROL ie AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. AROCLOR 1242 + $C_2$ SHAKEN 8 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P. ADDED INITIALLY AND AFTER 2 DAYS
4. AS 3 BUT AGITATED ON VIBRATORY BENCH SHAKER

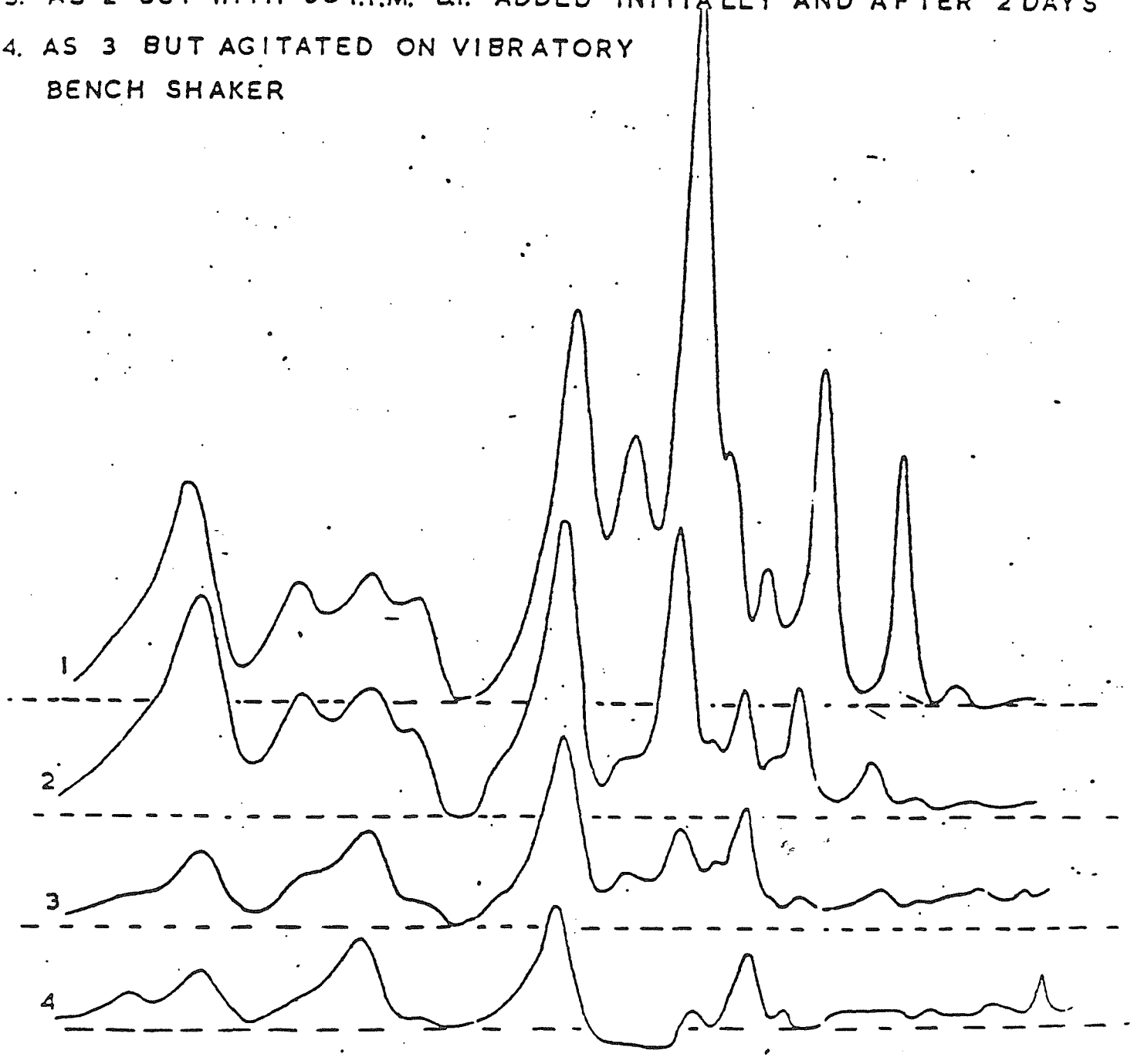


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FIG 12 DEGRADATION OF MCS 717 BY CULTURE $C_2$

1 CONTROL ie AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M $Hg$ ADDED

2 5 P.P.M. MCS 717 + $C_2$ SHAKEN 13 DAYS

3 AS 2 BUT WITH 50 P.P.M. B.P. ADDED INITIALLY AND AFTER 3 DAYS

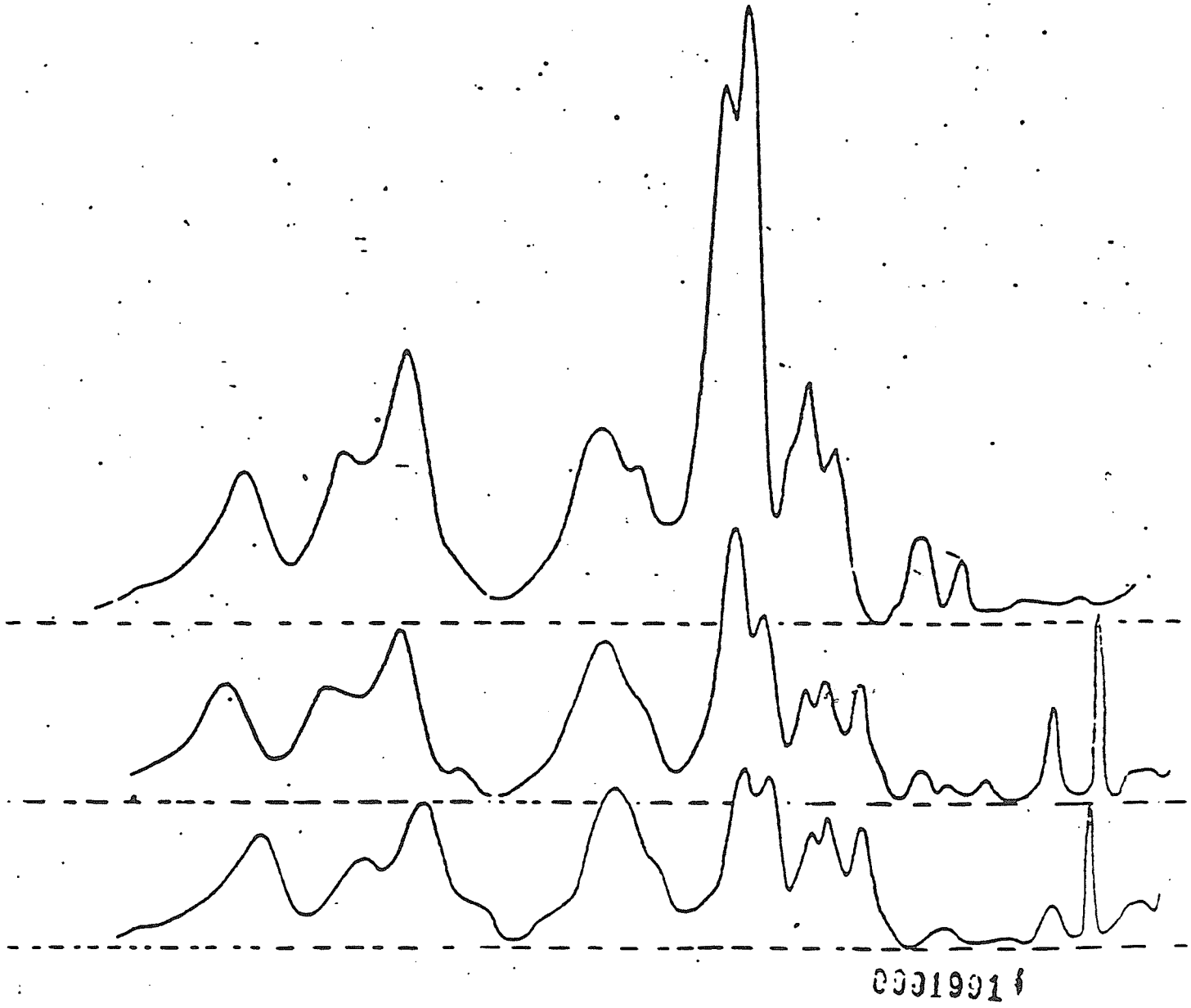

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FIG 13. DEGRADATION OF MCS 717 BY CULTURE $C_2$

1. CONTROL i.e. AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. MCS 717 + $C_2$ SHAKEN 38 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P. ADDED INITIALLY AND AFTER 3, 18, AND 28 DAYS

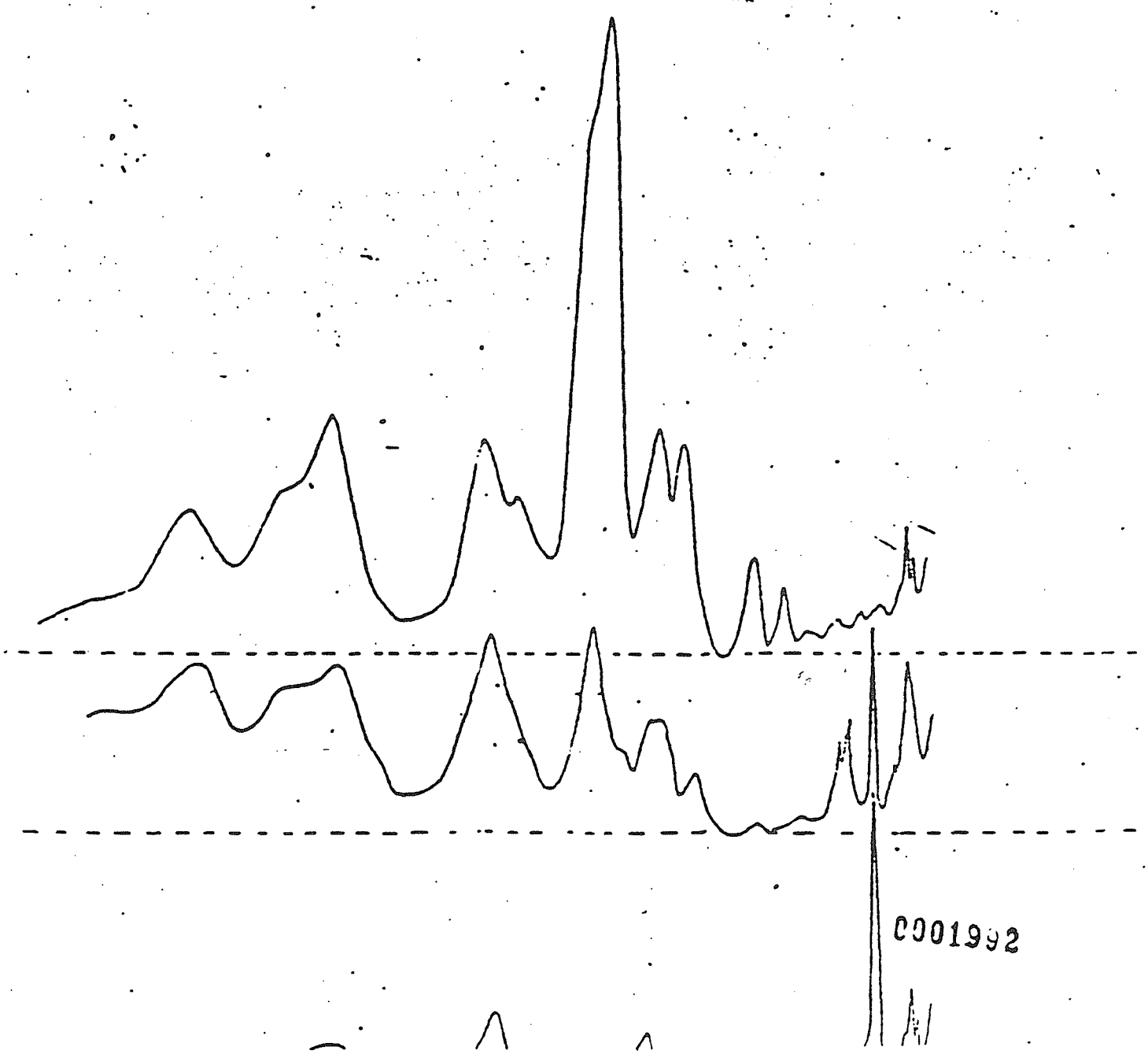

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FIG 2 DEGRADATION OF MCS 717 BY CULTURE $C_2$

1. CONTROL ie. AS 2 BUT WITH 50 P.P.M. BP AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. MCS 717 + $C_2$ SHAKEN FOR 73 DAYS
3. AS 2 BUT WITH 50 P.P.M. BP ADDED INITIALLY AND AFTER 3, 18, 28, 39, 46 AND 67 DAYS.

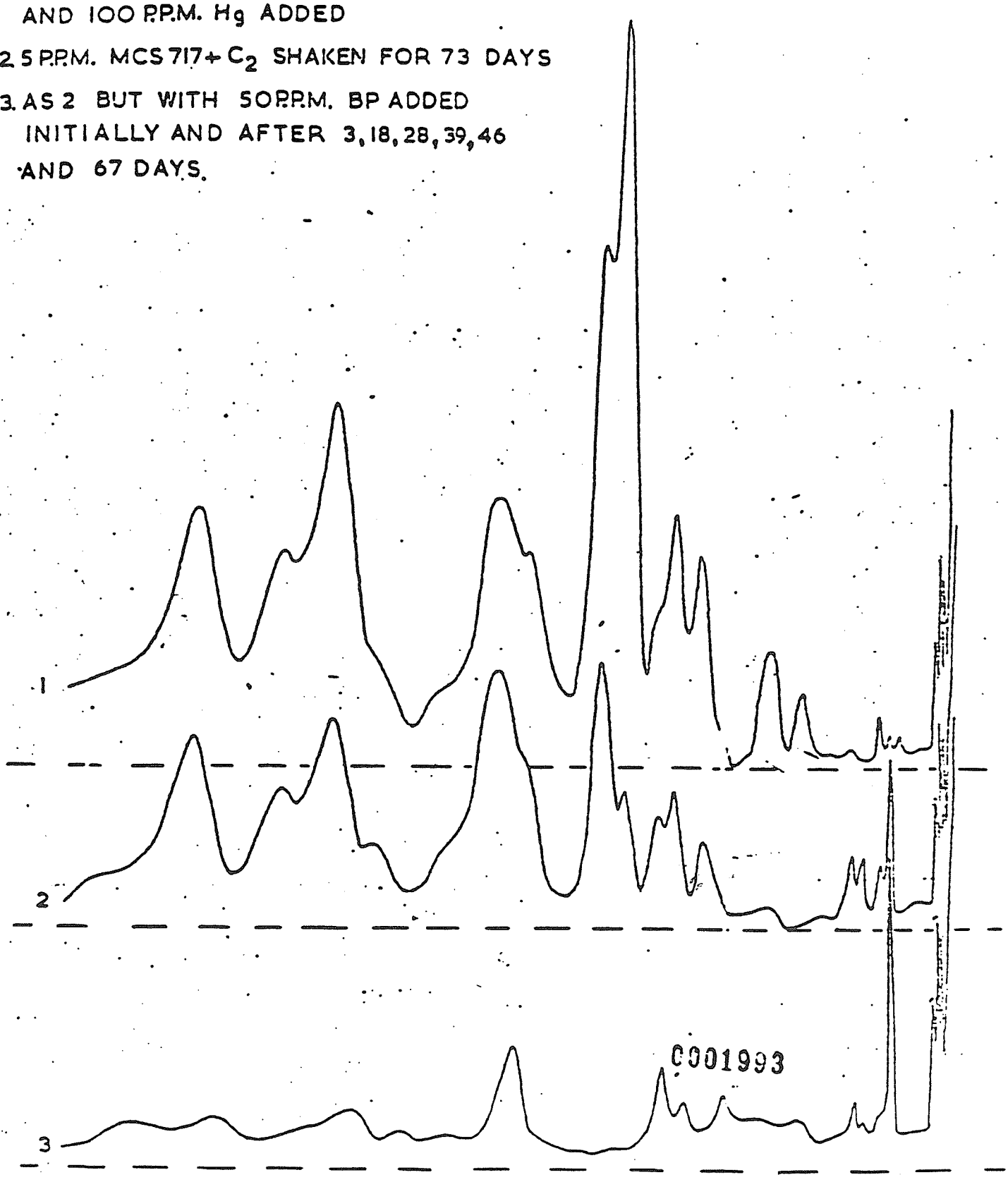


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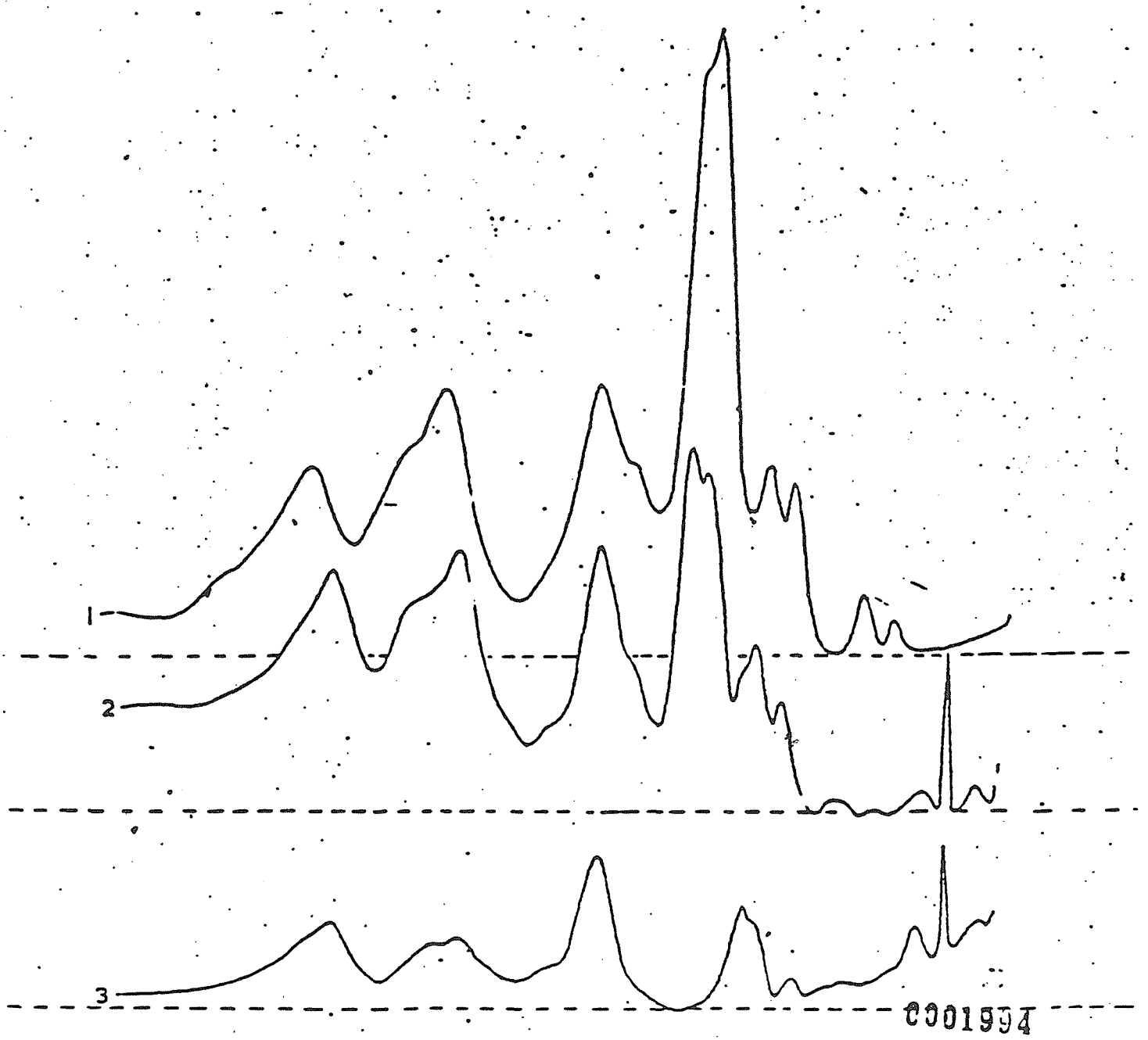
FIG 15 DEGRADATION OF MCS 762 BY CULTURE $C_2$

1. CONTROL Ie AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. MCS. 762 + $C_2$ SHAKEN 13 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P. ADDED INITIALLY AND AFTER 3 DAYS




C001994

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# IG16 DEGRADATION OF MCS 762 BY CULTURE $C_2$

1. CONTROL i.e. AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. MCS 762 + $C_2$ SHAKEN 38 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P. ADDED INITIALLY AND AFTER 3, 18 AND 28 DAYS

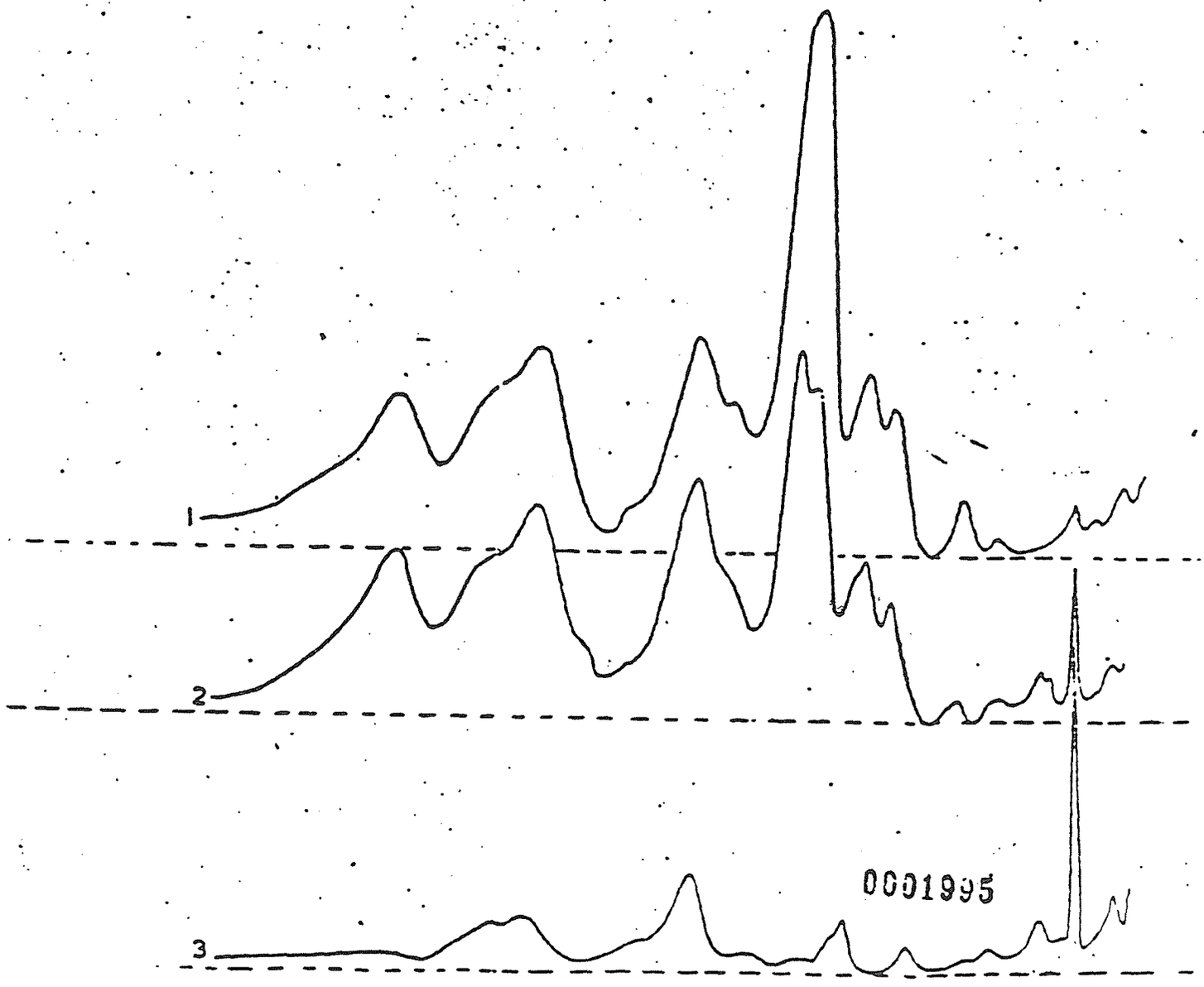


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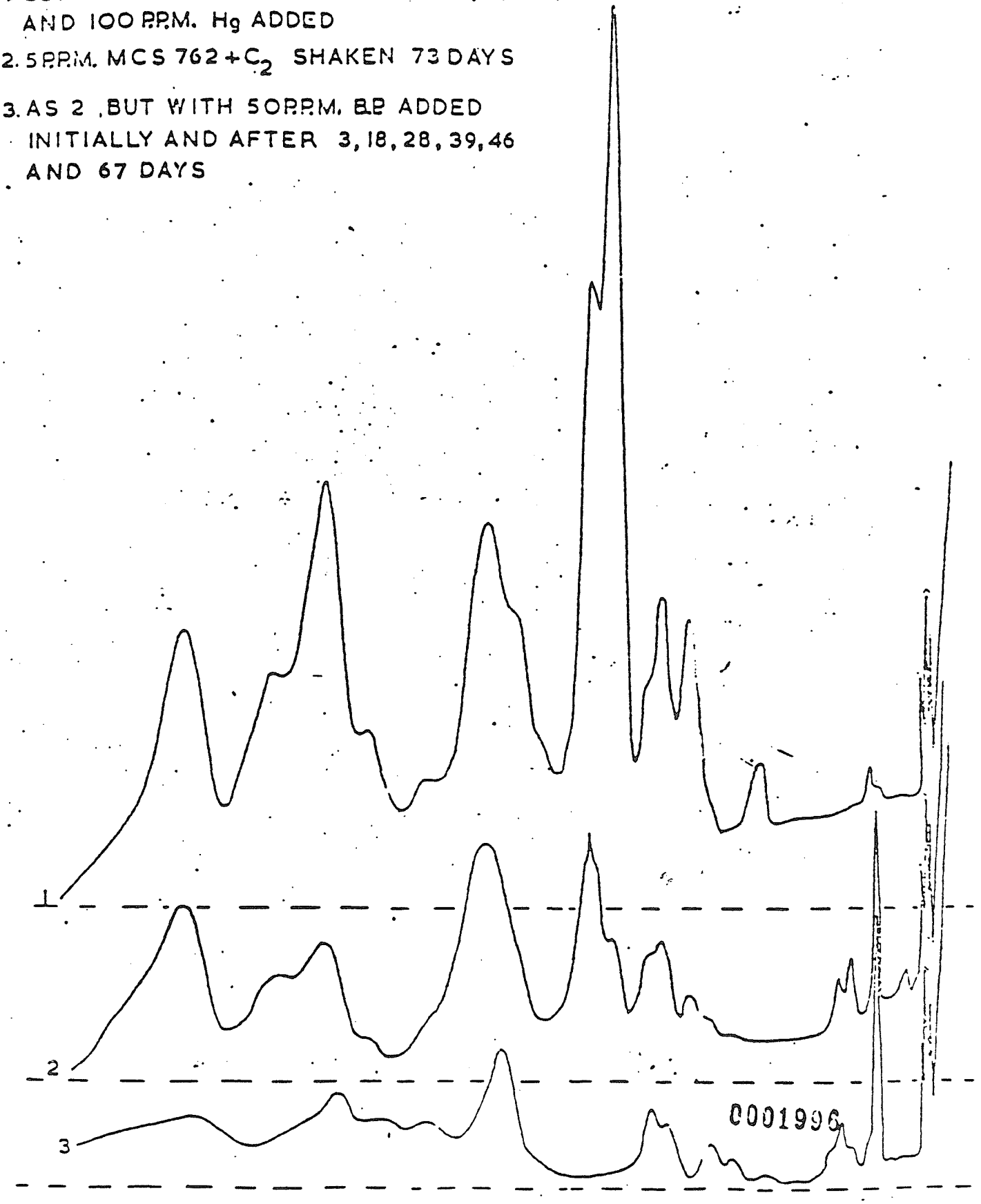
FIG 17 DEGRADATION OF MCS 762 BY CULTURE $C_2$

1. CONTROL ie. AS 2 BUT WITH 50 P.P.M. BP AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. MCS 762 + $C_2$ SHAKEN 73 DAYS
3. AS 2 BUT WITH 50 P.P.M. BP ADDED INITIALLY AND AFTER 3, 18, 28, 39, 46 AND 67 DAYS




C001996

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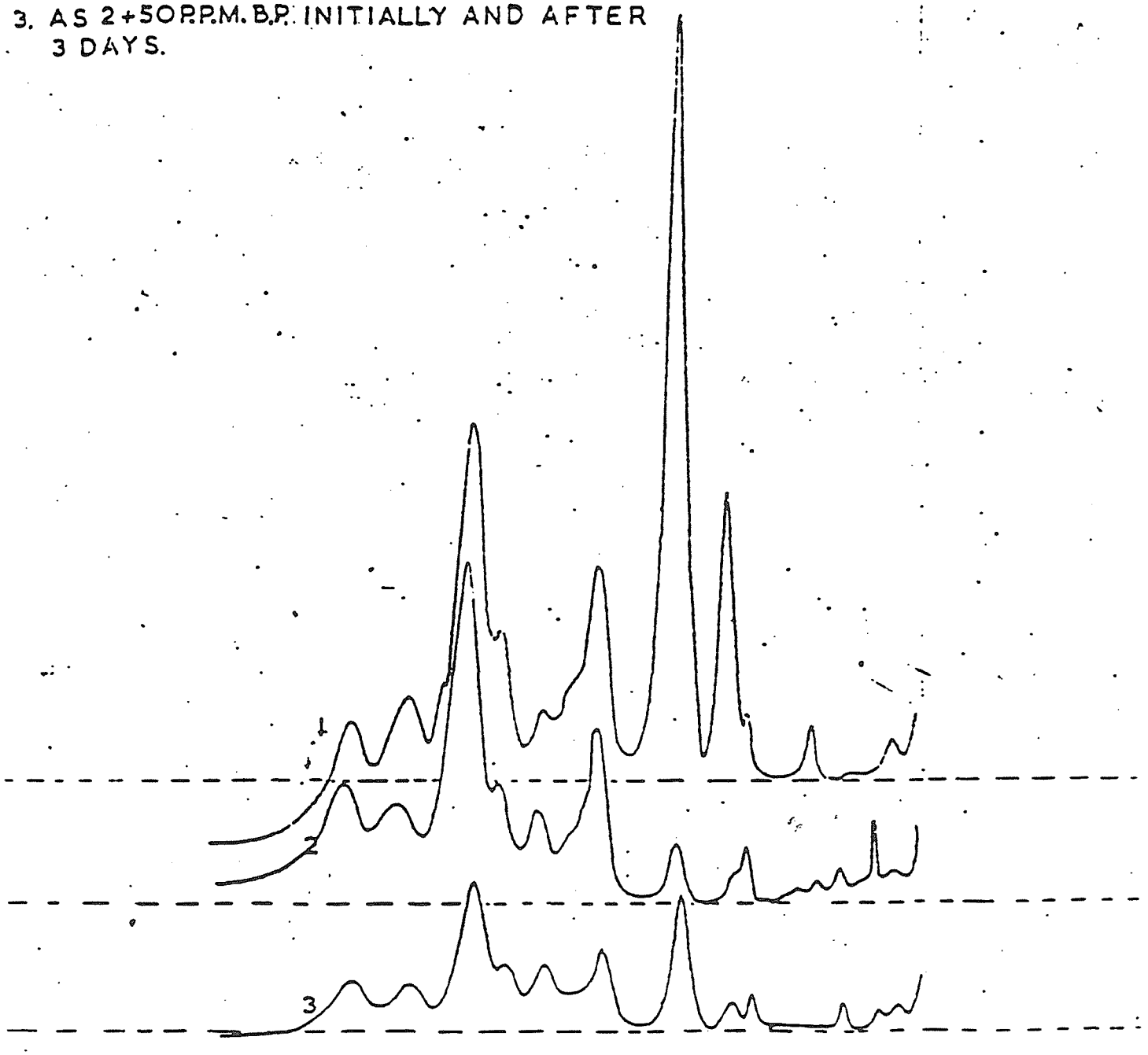
FIG 18 DEGRADATION OF AROCLOR 1242 B (MCS 1016) BY CULTURE $C_2$

1. CONTROL AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M. Hg ADDED
2. 5 P.P.M. AROCLOR 1242 B + $C_2$ SHAKEN 13 DAYS
3. AS 2 + 50 P.P.M. B.P. INITIALLY AND AFTER 3 DAYS.




0001997

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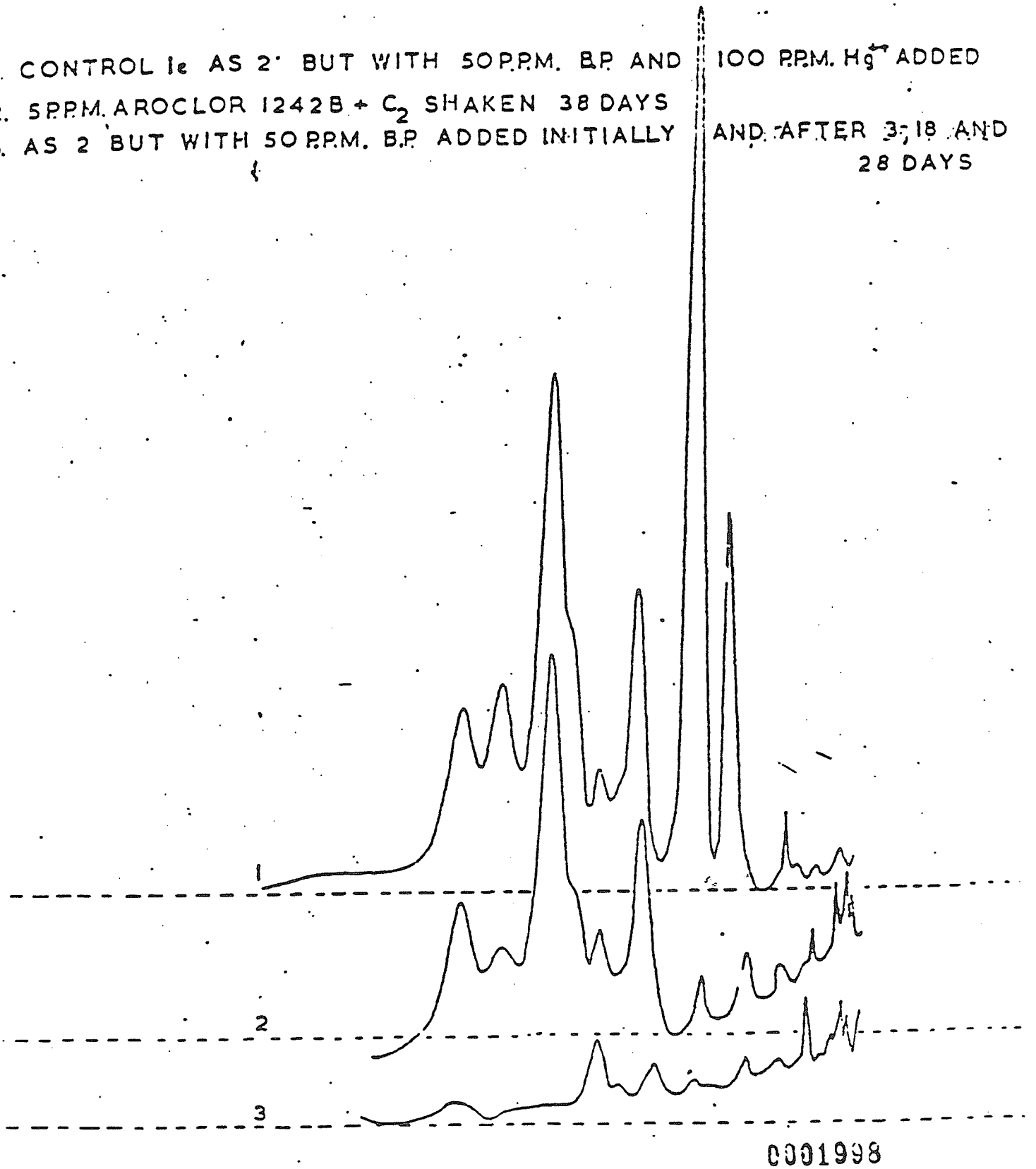
FIG 19 DEGRADATION OF AROCLOR 1242 B (MCS 1016) BY CULTURE $C_2$

1. CONTROL ie AS 2 BUT WITH 50 P.P.M. B.P AND 100 P.P.M. $Hg^{++}$ ADDED
2. 5 P.P.M. AROCLOR 1242 B + $C_2$ SHAKEN 38 DAYS
3. AS 2 BUT WITH 50 P.P.M. B.P ADDED INITIALLY AND AFTER 3, 18 AND 28 DAYS

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MG20 DEGRADATION OF AROCLOR 1242 B (MCS 1016) BY

CULTURE $C_2$

1. CONTROL ie. AS 2 BUT WITH 50 P.P.M. B.P. AND 100 P.P.M. Hg ADDED

2. 5 P.P.M. AROCLOR 1242 B + $C_2$ SHAKEN 73 DAYS

3. AS 2 BUT WITH 50 P.P.M. B.P. ADDED INITIALLY AND AFTER 3, 18, 28, 39, 46 AND 67 DAYS

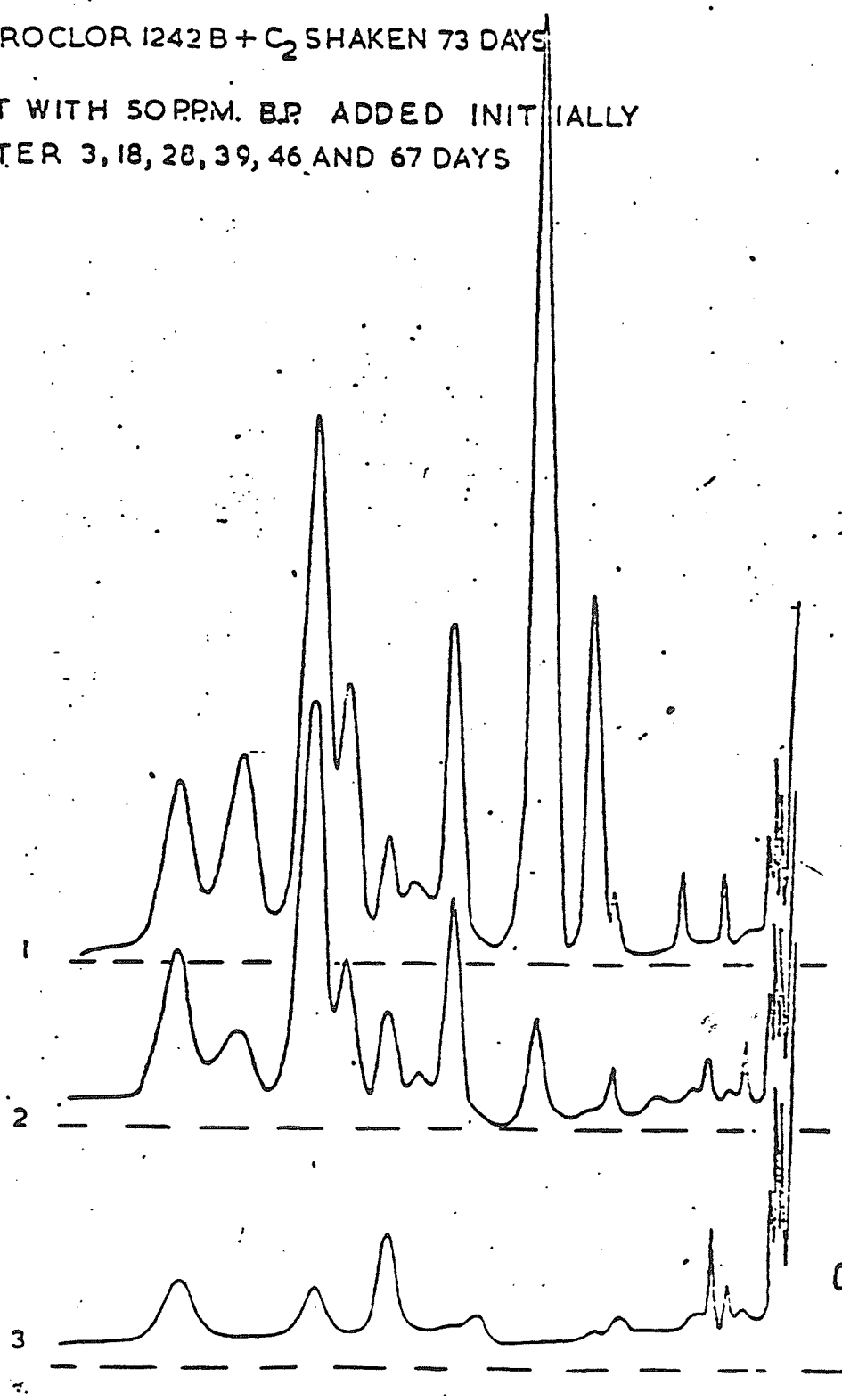


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FIG 21 COMPARISON OF THE DEGRADATION OF FOUR COMPLEX P.C.B. MIXTURES

1. AROCLOR 1242
2. MCS 762
3. MCS 717
4. AROCLOR 1242 B (MCS1016)

5 P.P.M. + $C_2$ SHAKEN 73 DAYS.

50 P.P.M. B.P. ADDED INITIALLY AND AFTER 3, 8, 18, 28, 39 46 AND 67 DAYS

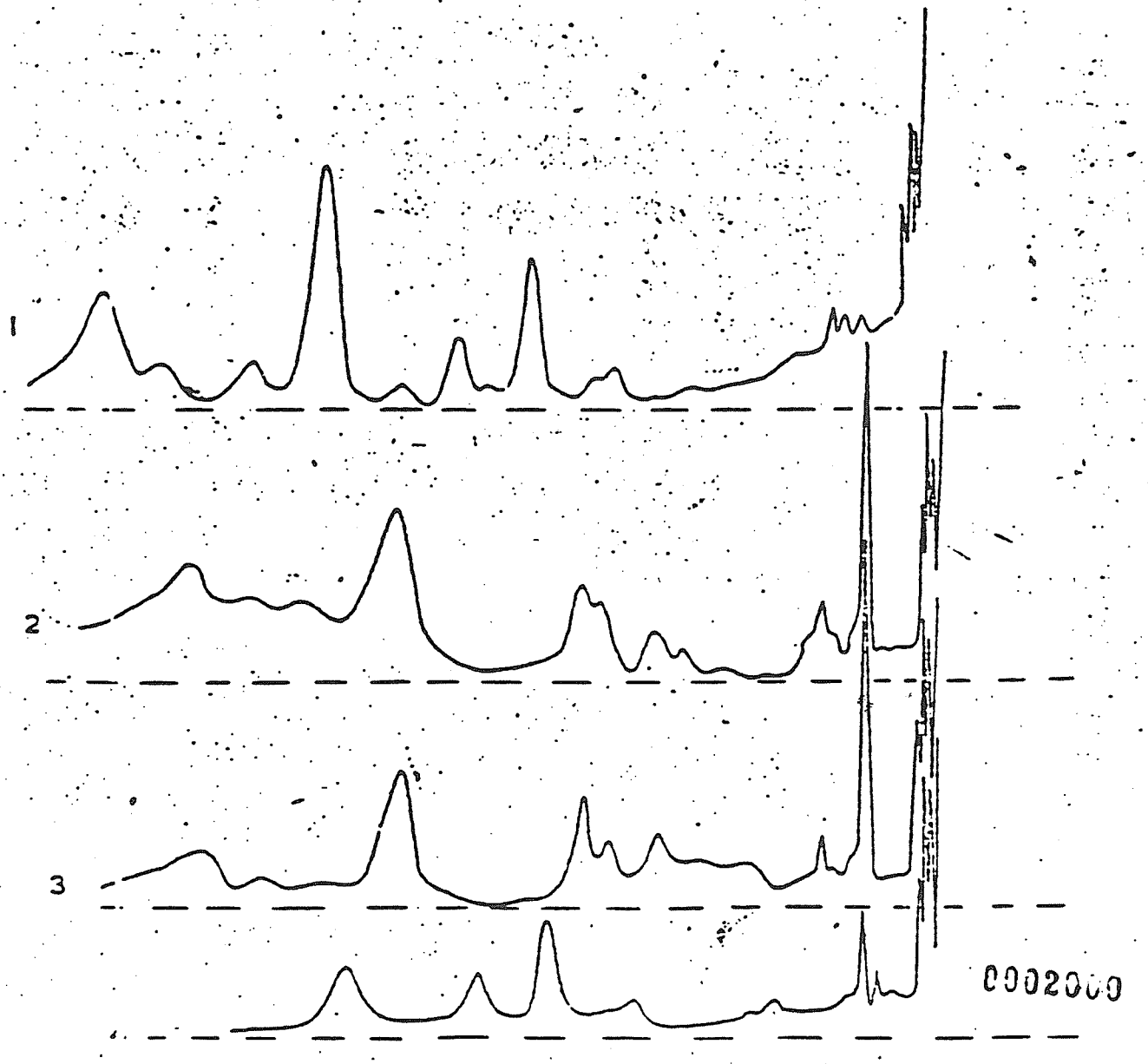


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FIG 22 DEGRADATION OF HB40 BY CULTURE $C_2$

1. CONTROL i.e. AS 2 BUT WITH 50 PPM BP AND 100 PPM Hg ADDED.
2. 5 PPM HB 40 + $C_2$ SHAKEN FOR 117 DAYS.
3. AS 2 BUT WITH 50 PPM BP ADDED INITIALLY.

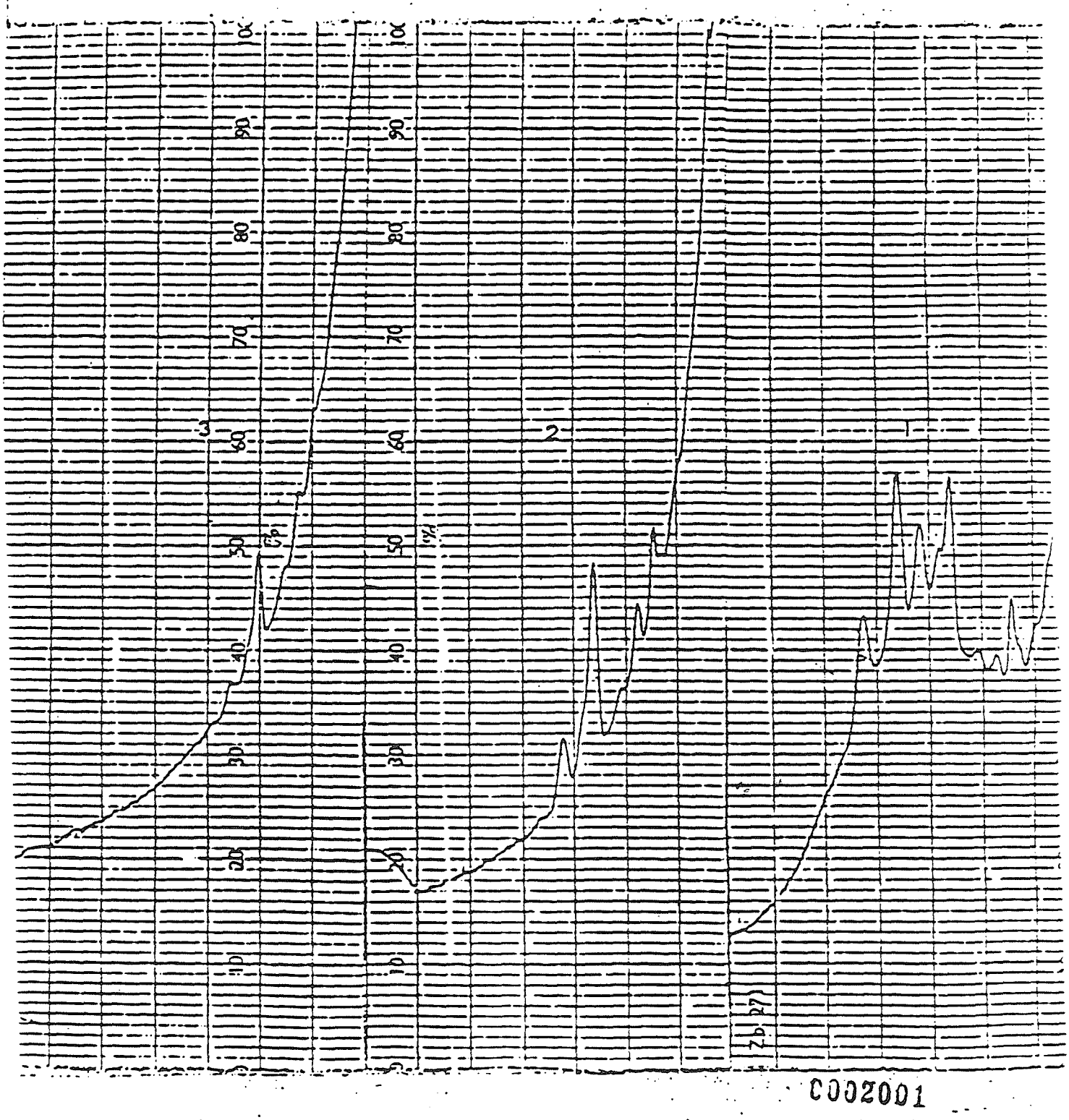


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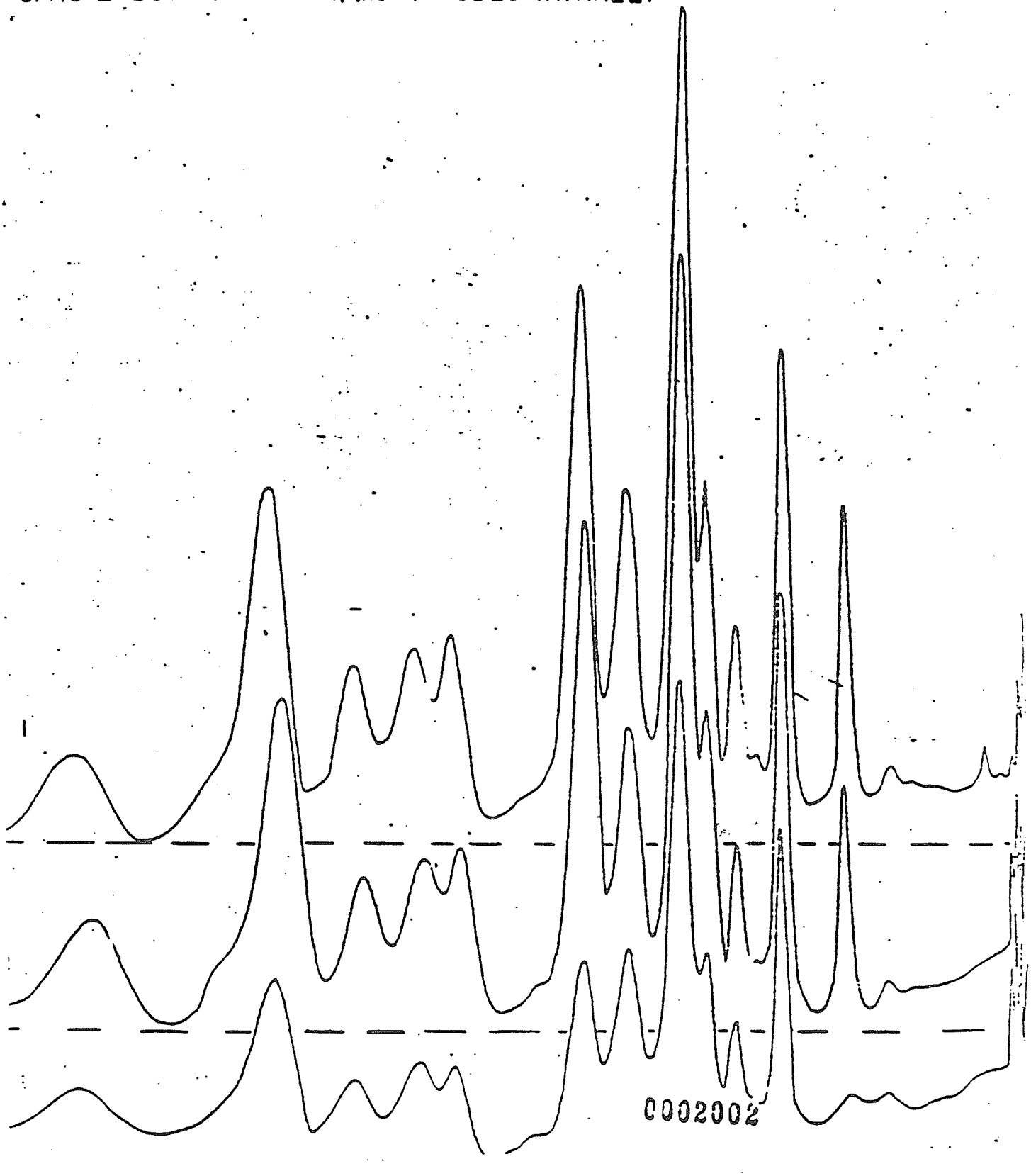
FIG 23 DEGRADATION OF AROCLOR 1242 BY CULTURE $C_2$

1. CONTROL ie AS 2. BUT WITH 100 P.P.M. Hg ADDED
2. 5 P.P.M. AROCLOR 1242 + 100 ml RIVER WATER SHAKEN FOR 180 DAYS
3. AS 2 BUT WITH 50 P.P.M. BP ADDED INITIALLY

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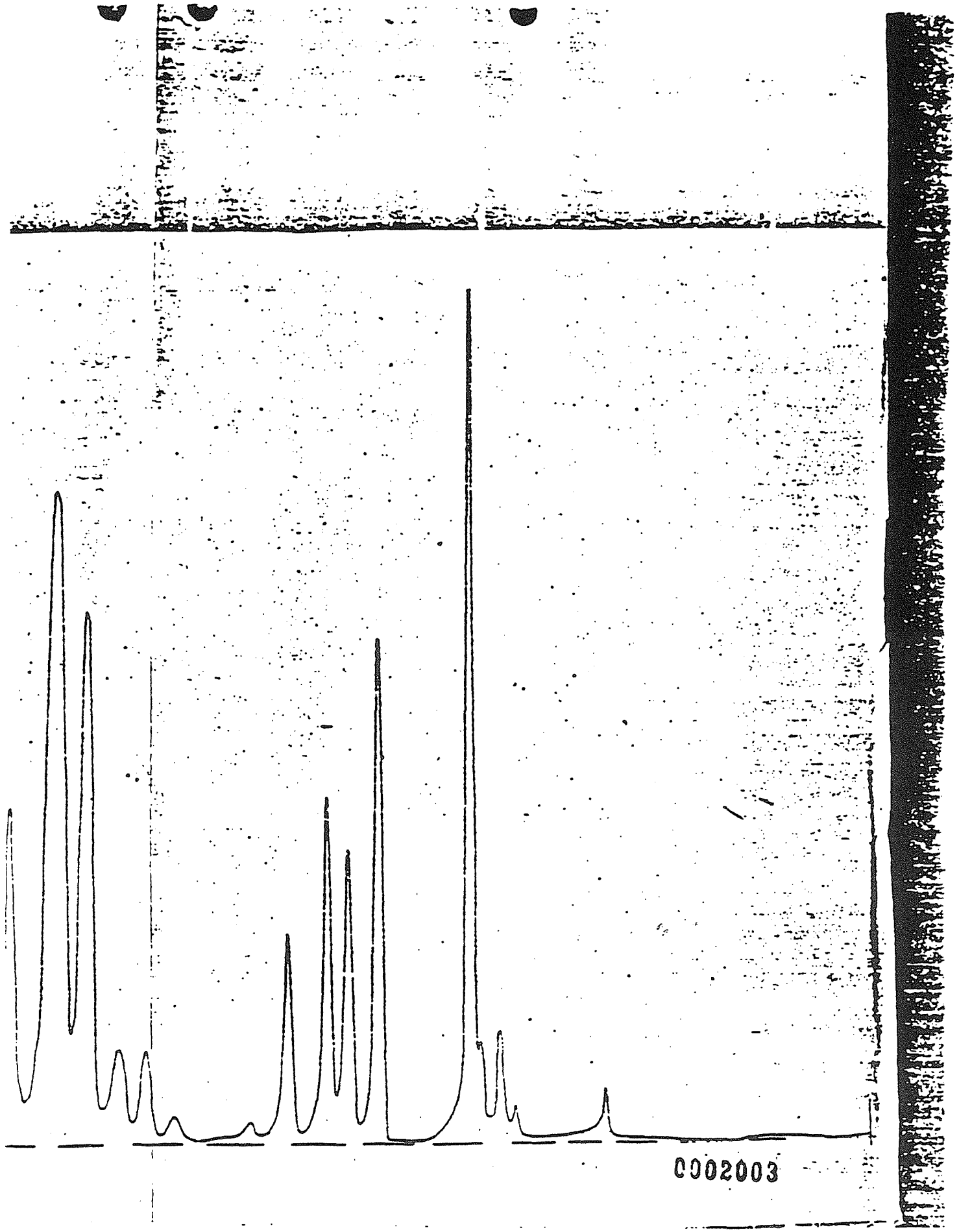
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AROCLOR 1242 (CAPILLARY COLUMN)

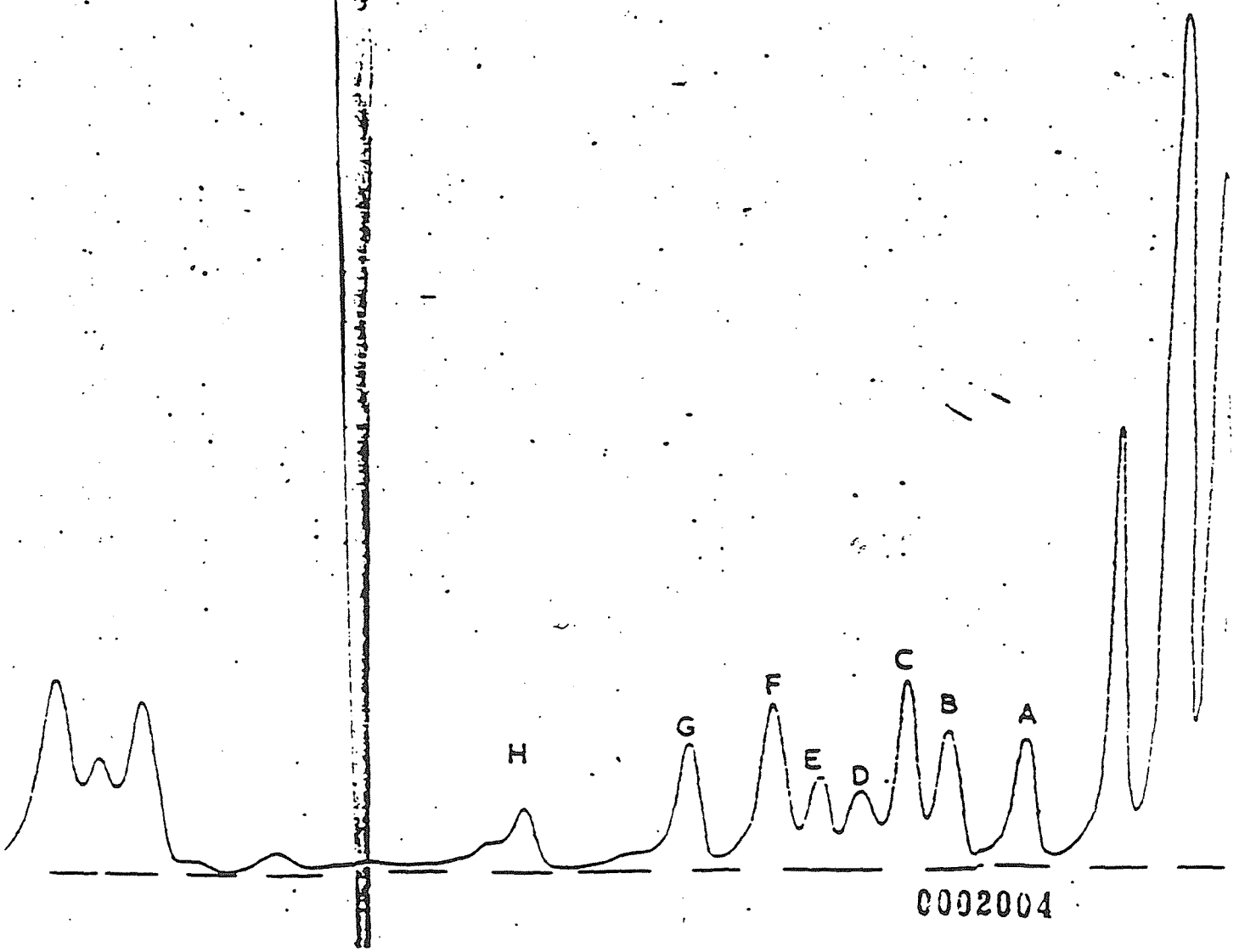


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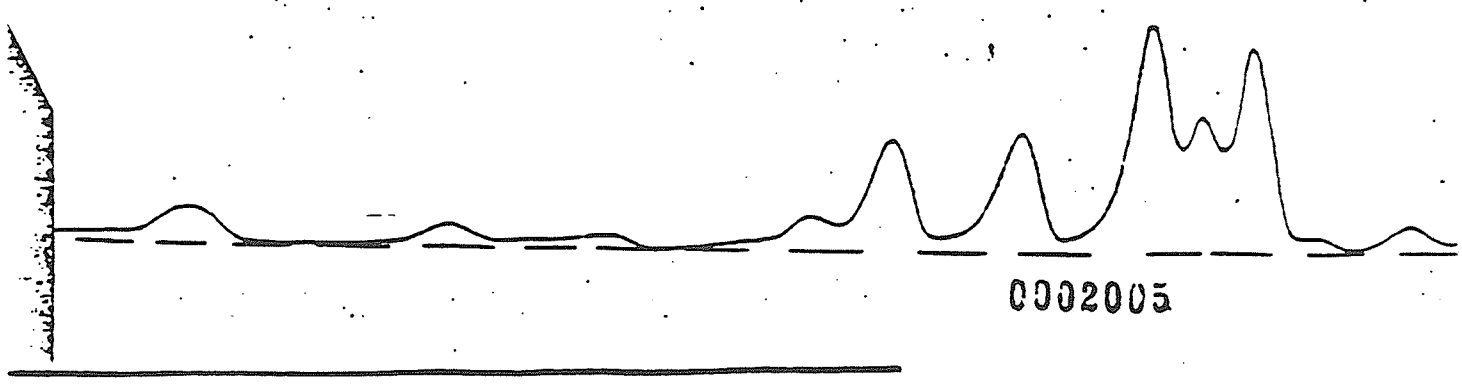
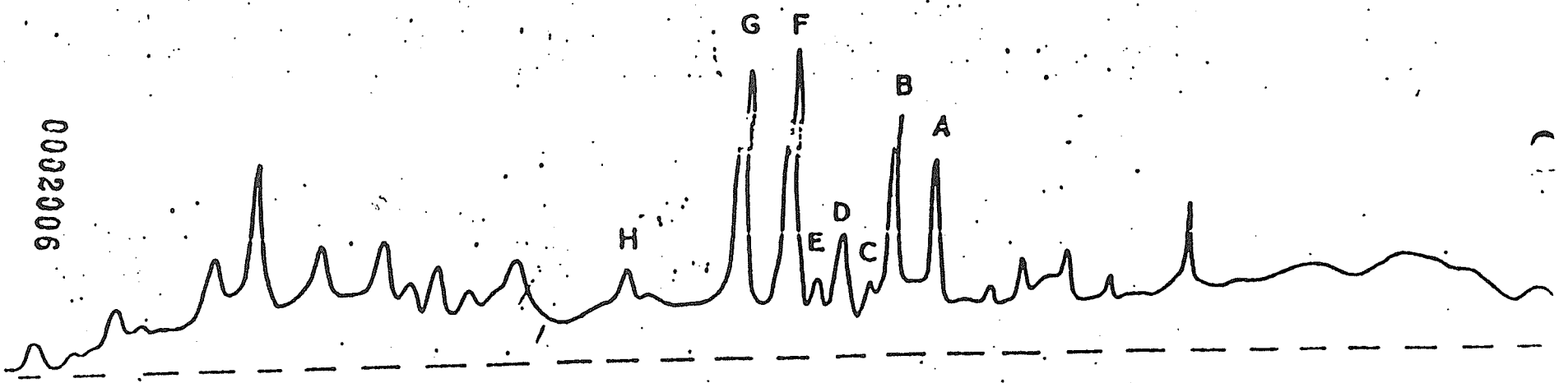
FIG 24 CHROMATOGRAM OF UNDEGRADED AROCLOR 1242 (C



0002005

FIG 25 CHROMATOGRAM OF DEGRADED AROCLOR 1242 (CAPILLARY COLUMN)




STLCOPCB4027340

Monsanto

FROM (NAME & LOCATION) E. S. Tucker - Technology Department - S. Second Street

DATE December 21, 1970 cc: R. E. Keller - Res. 1

SUBJECT

REFERENCE

TO : T. Bell
Anniston

Regarding our conversation on 12-18-70, it dawned on me after talking to you that I had recently run across an article in Analytical Chemistry which might be of help to you. Basically, it appears to be a rather simple method of increasing the sensitivity of the EC techniques you are currently using to monitor PCB's in water. If it works, it might allow you to detect extremely low levels of PCB's and hence shorten the time required to determine the point at which PCB's begin breaking through the carbon absorption columns you are currently evaluating.

If you do choose to evaluate this concentration technique, please let us know how well it works.

A copy of the article is enclosed.

→ E. S. Tucker

db
Encl.

TRAN 066313

IM 10 REV. 1-68

STLCOPCB4027341

**Monsanto**

FROM (NAME & LOCATION) W. R. Richard - Research Center

DATE December 22, 1970 cc

SUBJECT BIODEGRADABILITY OF POLYCHLORINATED BIPHENYLS

REFERENCE

TO : E. S. Tucker - S. 2nd St.

The subject report by J. H. Mainprize of Ruabon was issued December 2, 1970. A copy was sent to R. E. Keller, so you might find it convenient to take a look at his.

WR.

W. R. Richard

/ls

TRAN 066312

FM 10 REV 11 68

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The following actions were taken or planned at the subject meeting involving review and projection of work by Applied Sciences.

BIODEGRADATION STUDIES

1. Primary biodegradation studies with the semi-continuous activated sludge test will be continued on a need basis for products from the business groups.

2. Current work will be directed to application of the above test to distillation cuts to show that MCS 1016 is a more biodegradable product than Aroclor 1242. This information will help to promote the substitution of MCS 1016 for Aroclor 1242 in the market place.

3. Future efforts must be directed toward development of meaningful guide-lines, acceptable to regulatory bodies, to better evaluate the biodegradation data in hand. For example, at what % biodegradation level in 24 hours should a product be considered not acceptable under any circumstances if released to the environment? Concensus on this point indicated that we should continue to obtain our primary degradation figures which would provide a base for future reference and decisions.

4. No immediate work is planned in St. Louis on the use of specific cultures for biodegradation studies. Outside research of this type, including that with $C_3$ cultures, will be continued in the UK until the end of 1971.

5. No imme ate work is planned on determining biodegradation routes or identifying end products.

PCB RESIDUES IN INDUSTRIAL BIO-TEST TISSUES

The following work will be carried out to provide data needed to

- determine the maximum body PCB residue level at the no effect level.
- determine the residue plateau level for body tissues.
- establish PCB residue levels after recovery period following exposure.
- establish placental transfer of PCB's.

The projects will be carried out in the order indicated with #1 being completed first.

1. Two-year chronic toxicity study in rats, #B7298.

   - Male and female composites of muscle, liver and fat from the 12 and 24 month studies will be analyzed for PCB's. Kidney tissues will be eliminated from the analytical program.



DEPOSITION EXHIBIT
PHS 308
6-12-92 SB9

- Rats will be placed on a recovery diet for 60 days, sacrificed and analyzed for PCB's.

- Analytical results from the 12 and 24 month studies will be reviewed and a decision made on need for analyses for the 6 month study.

2. Three-generation reproduction study in rats, #P7297.

   - Male and female composites of muscle, liver and fat from the above will be analyzed for PCB's. This includes

     . parents, F-3 generation, F-2b litter.

     . parents on recovery diet, 1/2 of parents above for 60 days.

     . pups, F-3 generation.

   Kidney tissues will be eliminated.

3. Two-year chronic toxicity study in dogs, #C7299.

   - Male and female muscle, liver and fat will be analyzed for PCB's. This includes

     . 24 month sacrifice animals.

     . recovery sacrifice animals, 1/2 of 24 month, 60 days.

   - Control samples for above will be composites of male and female.

   Kidney tissues will be eliminated.

4. Chicken toxicity, reproduction and meat and egg residue study, #J7300.

   - Male and female muscle, liver and fat will be analyzed for PCB's. This includes

     . test sacrifice chickens.

     . recovery sacrifice chickens.

     . 30-day chicks hatched from eggs from above.

   - Control samples will be composites of male and female.

   - All eggs will be analyzed.

   Kidney tissues will be eliminated.

5. Aroclor and tissues from the 24 month rat study will be given a brief analysis for trace mercury levels.



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6. Results from the preceding work will be published, if possible, to help provide the base needed to set guide-lines for safe levels of PCB's in the environment.

Details for the preceding work with Industrial Bio-Test tissues will be determined jointly between Messrs. Wheeler, Fancher, Tucker and Keller.

RK

R. E. Keller

db
5/17/71



STLCOPCB4027345

Monsanto

FROM (NAME & LOCATION) W. S. Clark - General Offices

DATE: May 22, 1970

SUBJECT: NCR MEETING TO DISCUSS AROCLORS, M1PB, HB-40

REFERENCE:

cc: P. K. Maier - Cincinnati
W. E. Schalk - WSCHA
J. E. Springgate - JSPRI
E. C. Wilde - Akron
J. D. Wright - JWRIG

TO:
E. Emery - JFQ
→ M. W. Farrar - JFQ
J. T. Garrett - JGARR
W. H. Hunt - WHUNT
R. E. Keller - RKELL
R. E. Kelly - RKELL
W. B. Papageorge - WPAPA
S. Tucker - JFQ
W. F. Waychoff
E. P. Wheeler - EWHEE

(NCR)

On June 1, 1970, Messrs. Taylor, Thacker and Hebert of NCR will be in St. Louis to discuss toxicity, biodegradation, and analytical techniques associated with Aroclor 1242, M1PB and HB-40. This meeting will be held at 9:00 AM in Room C-303 at the General Offices. The agenda for this meeting will be as follows:

| | |
|---|---|
| Analytical Techniques | Applied Science Group |
| Biodegradation | Applied Science Group |
| Toxicity | Medical |

In view of the importance of these products and this account, it would seem reasonable to review this presentation on Thursday, May 28, 1970. This review will be held in Room B-275 at 9:00 AM. Please notify me if you will not be able to attend the meeting on June 1 and the review on May 28.

Will Clark

Willis S. Clark

/dbw

TRAN 009721

DEPOSITION EXHIBIT
PHF 309
6-12-92

STLCOPCB4027346

NATIONAL CASH REGISTER

CUSTOMER VISIT

JUNE 1, 1970

| | | |
|---|---|---|
| NCR: | Gordon Taylor | Head of NCR Paper Research |
| | Jerry Taylor | Manager - Purchases & Technical Service |
| | Ned Thacker | Staff Engineer |
| Monsanto: | Ed Emery | Research Chemist |
| | Jack Garrett | Medical Department |
| | Bob Keller | Organic Research Manager |
| | Pete Maier | Field Sales |
| | Bill Papageorge | Manager - Environmental Control |
| | Scott Tucker | Research Chemist |
| | Walt Waychoff | Product Manager |
| | Elmer Wheeler | Medical Department |
| | Jim Wright | Field Sales Manager |
| | Will Clark | Product Specialist |

| | |
|---|---|
| 8:12 | NCR arrive St. Louis Airport, TWA Flight 443 with P. Maier, met by W. Clark. |
| 8:45 | Visit with Jim Springgate |
| 9:00 | Meeting on Toxicology, Biodegradation, Analytical Work as it relates to MIPB, HB-40, and Aroclor 1242. |
| 12:00 | Leave for Lunch |
| 1:30 | Leave for Airport |
| 1:45 | Arrive Airport |
| 2:15 | NCR leave St. Louis Airport, Allegheny Flight 883, with P. Maier. |

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TRAN 085270

DEPOSITION
EXHIBIT
Plf. 310
6-12-92

# MCR CUSTOMER VISIT

## JUNE 1, 1970

### Objectives

1. Review the current state of knowledge in areas of toxicology, biodegradation and analysis of MIPB, HB-40, and Aroclor 1242.
2. Provide reports as appropriate covering this information.
3. Review Monsanto Company's plans for future work in these areas.

### Meeting Agenda

| | | |
|---|---|---|
| 1. | Background - European Visit | Papageorge |
| 2. | Toxicology | Wheeler |
| 3. | Analytical | Tucker |
| 4. | Biodegradation | Emery |

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W. S. Clark

TRAN 085271

STLCOPCB4027348

DEPOSITION EXHIBIT
Plf's 311
6-12-92

P. O. Benignus - St. Louis General Office

April 17, 1970

WESTINGHOUSE — PCB PROBLEM DISCUSSION
ST LOUIS MEETING
TUESDAY, APRIL 21

D. A. Olson

Q. Thompson - QTHOM
R. S. Bergen - RBERG
W. R. Richard - WRICH
H. H. Munch - JHM
J. O. Bryant
E. Wheeler - EWHEE
R. Keller - JFQ
S. Tucker - JFQ
W. Papageorge - WPAPA
R. E. Kelley - RKELL
J. Savage - JSAVA

PLAINTIFF'S EXHIBIT
215
Monsanto 8-10-81

All of our Monsanto people receiving copy of this memo are invited to the meeting which will be held in room T-459 located in the research center.

Westinghouse will be represented by the following people:

Dr. T. K. Sloat -- Westinghouse, Sharon, Pennsylvania
L. Shoaff -- Westinghouse, Bloomington, Indiana
G. Wilburn or D. Kaiser -- Westinghouse, S. Boston, Virginia
A. Tanton -- Westinghouse, London, Ontario

These people will be arriving Monday evening, April 20 and stay at the Hilton Inn across from the airport.

<u>The Meeting Schedule</u>

We would like to call on our people in the following order:

9:00 a.m. - 10:30 a.m. — Mr. Elmer Wheeler, Presentation of the Overall PCB Pollution Problem

10:30 a.m. - 10:45 a.m. - Coffee Break

10:45 a.m. - 11:15 a.m. - Dr. R. Keller, Detailed Description of Analytical Procedures

11:15 a.m. - 12:00 p.m. - W. Papageorge, Current and Foreseeable Future Status of the Pollution Problem

12:00 p.m. - 12:30 p.m. - Dr. W. R. Richard, Methods for disposing Scrap Askarels, Including Distillation and Incineration. Biodegradeable Studies.

12:30 p.m. - 1:30 p.m. -- LUNCH at Reserved Tables in our Cafeteria

1:30 p.m. - 2:30 p.m. -- Dr. H. H. Munch, Possible Replacement Fluids

2:30 p.m. - 3:30 p.m. -- General Summary

55

DEPOSITION EXHIBIT
192

P. O. Benignus

1154

/dn

STLCOPCB4027349

SEP 23 1968

C. Paton - General Offices

September 23, 1968

AROCLOR 5460/FDA PETITION

Dr. R. E. Kelly/Dr. W. H. Hunt
Dr. R. E. Keller
W. F. Waychoff

H. L. Williams - Anniston
G. W. Miller - Anniston

Further to our conversation, I am sending you several pieces of correspondence.

1. The letter from the FDA to Dr. Kelly indicating the deficiencies in our petition on Aroclor 5460.

2. The section of our 1968 petition on Aroclor 5460 (pages 10-13) which deals with the biphenyl and chlorinated biphenyl content of Aroclor 5460.

3. The GC and IR curves we attached to the 1968 petition.

4. The section of our 1966 petition on Aroclor 5460 which we withdrew without prejudice because we needed more feeding data.

5. The GC curve on Aroclor 5460 which was attached to the 1966 petition.

You will note:

1) Our manufacturing procedure has been described differently in the two petitions. After today's conversation, I understand the 1968 is the more correct of the two descriptions. The percentage biphenyl is determined on the Santowax R fed to the chlorinator. On distilling the chlorinated Santowax R, Montar 9 and Aroclor 5460 is obtained. No top-cut is taken in this distillation.

2) The questions raised by the FDA in their letter were answered, I think, by the GC curve submitted with the 1966 petition. The GC curve submitted with the 1968 petition does not give column details and other analytical conditions. It omits the factors necessary to calculate the percentage of each of the components. Can you let me have this information please.

TRAN 058001

DEPOSITION EXHIBIT
PHS 312
6-12-92 89

With regard to the polyphenyl composition, what maximum can we live with because we'll have to specify.

This will also be true for biphenyl content in the Santowax R. Can you live with 0.5%, 1.0% or what.

3. We need copies of the methods we could actually use for:

   Softening point
   Chlorine content
   Refractive index

   Bob Keller is attempting to determine a test method for chlorinated biphenyl content in Aroclor 5460.

4. I understand that AI 43 was the lot used in the feeding studies and we don't have a retained sample.

   Do we have any samples of lots prior to AI 43 and just after AI 43 so that we can determine:

   a) biphenyl content of Santowax R

   b) chlorinated biphenyl content of Aroclor 5460.

We must reply to the FDA by October 12 so these requests are rather urgent.

Cumming Paton

CP:dbw

TRAN 058002

STLCOPCB4027351

bcc: W. B. Papageorge
E. [illegible] Wheeler
W. [illegible] Richard
J. T. Garrett
M. W. Farrar
E. S. Tucker

PLAINTIFF'S EXHIBIT JS
223
8-11-87

Monsanto
COMPANY

JUN 9 197

ORGANIC CHEMICALS DIVI

1700 South Second Street
St. Louis, Missouri 63177
(314) MAin 1-4000

PCB - Governmental Coors

June 4, 1970

FW9A

Dr. A. F. Tabri, Chemist
Chemistry Unit
Department of the Interior
Federal Water Pollution Control Administration
5555 Ridge Avenue
Cincinnati, Ohio 45213

DEPOSITION EXHIBIT
PH: 313
6-12-92

Dear Dr. Tabri:

My apologies for not answering your letter of May 20 sooner. I have just recently returned from Europe and I am now finally catching up on my correspondence.

With reference to your question about solubility, we have the following information. We find that the solubility of our Aroclor 1242, 1248 and 1254 products is less than 300 ppb in water at 25°C. A rough order of magnitude for each is 50 ppb for Aroclor 1254, 100 ppb for Aroclor 1248 and 200 ppb for Aroclor 1242. We have biodegradability studies underway on the degradation of our Aroclor products. Two techniques have been employed to date; semi-continuous activated sludge and river water die-away. Early results indicate that a major number of the 2 chlorine and the 3 chlorine isomers degrade under these conditions. We hope to have a more complete picture on the polychlorinated biphenyl at higher chlorine levels in the not too distant future.

Your questions about toxicity and other additional information has been brought to the attention of Messrs. W. B. Papageorge and E. P. Wheeler of Monsanto for their comments. You can expect to receive an additional reply on these points.

We have no known isomer components available for analytical purposes at this time. For your information, we recently provided a few isomers on a "first come" basis to Dr. Dustman at the Department of the Interior, Laurel, Maryland. Unfortunately, our extremely small supply has been depleted. We assume that you have our product grade Aroclor materials and, if not, we will be happy to provide you with a set. Please let us know if we can be of further assistance.

Sincerely,

3485

Bloomington Dep 118 id
6/25/86 ag

Robert E. Keller, Manager
Applied Sciences [illegible]

UNITED STATES
DEPARTMENT OF THE INTERIOR
FEDERAL WATER POLLUTION CONTROL ADMINISTRATION

IN REPLYING ADDRESS:

5555 Ridge Avenue
Cincinnati, OH 45213

May 20, 1970

Mr. Robert E. Keller, Manager
Applied Sciences Section
Monsanto Chemical Company
1700 South Second Street
St. Louis, Missouri 63177

Dear Mr. Keller:

Thank you for your letter of April 27 and the enclosures on specific methodology for PCB Aroclors.

We would appreciate receiving any additional information if available, on such parameters as solubility, toxicity, degradation, etc., pertaining to these compounds.

Also, if available, we would appreciate having a few grams each of analytical samples for purposes of calibration.

Thank you in advance for your consideration of this request.

Sincerely yours,

A. F. Tabri, Chemist
Chemistry Unit
Water Sciences Section
National Field Investigations Center

3486

STLCOPCB4027353

Monsanto INDUSTRIAL CHEMICALS COMPANY

FROM (NAME & LOCATION) R. E. Keller - S. Second St. - St. Louis

DATE November 11, 1971

SUBJECT PCB'S IN PRODUCTS

REFERENCE

TO : W. B. Papageorge
General Offices

cc C. W. Roos - Res. 1
F. B. Zienty - GO
M. W. Farrar - Res. 1
W. R. Richard - GO
W. K. Johnson - GO
O. DeGarmo/J. F. Quinn - Res.
E. S. Tucker/J. P. Mieure - Res.

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Because of current work by regulatory agencies on probing for PCB's - we have looked at some of our key chemical products to determine if contamination exists. Overall, the products are within the very tight limits of 0.5 ppm max. PCB for food products such as eggs. On this basis, the following products, part of which are non-edible, would be expected to meet any rigid guide lines that might be set in the future.

Aspirin
Maleic Anhydride
Sodium Saccharin
Saccharin
Cresyl Diphenyl Phosphate
Santicizer 334F
Santicizer 711
Santicizer 160
Vanillin

Details of this work for 1971 production materials are summarized in the attached table. Results show that PCB's, typical of Aroclor 1242 and/or 1248, are found at levels less than 0.5 ppm in saccharin, cresyl diphenyl phosphate, Santicizer 334F, Santicizer 711, Santicizer 160 and vanillin.

Aspirin, maleic anhydride and sodium saccharin contain no detectable (<0.1 ppm) PCB.

An outside laboratory would likely find the PCB's reported in the above pharmaceutical and food ingredient products. We have to hope that the levels found would be put in proper perspective when reported to the press and others. The much lower body intake of these products relative to foods should provide for a much higher permissible level of PCB's.

We plan no additional work at this time.

REK

R. E. Keller

db
Attachment

DEPOSITION EXHIBIT
PH's 314
6-12-92

COMPANY CONFIDENTIAL

TRAN 021409

STLCOPCB4027354

Monsanto INDUSTRIAL CHEMICALS CO.

Aroclor S

FROM (NAME & LOCATION) E. S. Tucker - South Second Street Laboratories

DATE January 13, 1972

cc: R. E. Kellor - T1B

SUBJECT PCB'S IN DISHWASHER DETERGENTS

REFERENCE

TO W. B. Papageorge
B288S

Four samples of dishwasher detergents, manufactured by Economics Laboratory, were purchased from two different supermarkets and analyzed for PCB's as requested. PCB's were found in all samples at the levels shown in the following table.

| Sample Identification | PPM PCB's Found | Type of PCB's |
|---|---|---|
| Electra Sol, CO9141<br>3391-0520-0471<br>Purchased at Schnucks in Creve Coeur | 0.6 | Aroclor 1242 |
| Electra Sol, CO2501<br>3391-0518-1270<br>Purchased at Kroger's in St. Louis | 0.09 | Aroclor 1242 |
| Finish, 710051<br>6266-0500-0771<br>Purchased at Schnucks in Creve Coeur | 0.19 | Aroclor 1242 |
| Finish, "Special Formula for Water in This Area", CO8081<br>1035-0502-0565<br>Purchased at Kroger's in St. Louis | 1.17 | Aroclor 1254 |

E. S. Tucker

db

TRAN 023044

M-10 REV. 11-69

DEPOSITION EXHIBIT
Plf's 315
6-12-92

STLCOPCB4027355