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GENP 010324

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ANALYTICAL OR PHYSICAL MEASUREMENTS SERVICE REPORT

Customer's Reference: P.O. #002-129982

Du Pont No. 774-81

Date Received 6/1/77

CHLORODIBENZOFURANS IN POLYCHLORINATED BIPHENYLS

A sample of polychlorinated biphenyl (PCB) with General Electric Company's designation A.R. No. 88118, Sample 8111453, was sent to us for analysis of chlorinated benzofuran (CDBF) content. We agreed to attempt to perchlorinate the mixture and look for octachlorodibenzofuran in the presence of decachlorobiphenyl.

We have made two attempts to (1) separate the chlorinated dibenzofurans from the PCB's by liquid chromatography, (2) form the octachlorodibenzofuran derivative by perchlorination of the residue from the liquid chromatography fraction and (3) detect the presence of the octachlorodibenzofuran in the perchlorination mixture. However, from the data available we have not been able to determine if the perchlorinated dibenzofuran exists in the reaction mixture.

The procedure used for separation, perchlorination and detection was based on information in References 1, 2 and 3. One gram of the PCB mixture was taken up in hexane and added to a 1.2 cm liquid chromatographic column containing 20 grams of preactivated

Alton J. Dahl
Senior Supervisor

12/9/77

Signed

Date

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Florisil. The column was washed with 200 mL of hexane, 50 mL of hexane/acetone (95/5) and finally with 100 mL of acetone. The acetone fraction presumably held any chlorinated dibenzofurans as well as any other chlorinated oxygen containing organic compounds which were present in the original PCB mixture. The PCB's should have been removed from the column by the hexane.

The acetone column wash was reduced to 0.5-1.0 mL with a Kuderna-Danish evaporator. Low steam heat and slow nitrogen gas flush were used for the evaporation. The concentrated acetone fraction was transferred to a Carius tube. The acetone was removed with a slow nitrogen purge through a narrow tube inserted in the Carius tube. No heat was applied to the tube. The residue was perchlorinated with antimony pentachloride according to the procedure in Reference 1. Hexane instead of benzene was used in the final work up of the reaction mixture.

The hexane solution of the perchlorination reaction was analyzed by gas chromatography with an electron capture detector. The GC column was a stainless steel tube, 180 cm x 3 mm, packed with 3% OV-17 on 60-80 mesh Gas Chrom Q. The column temperature was 200°C; the helium carrier gas flow was 30 mL/minute.

Two control experiments were run. Dibenzofuran was perchlorinated and dibenzofuran was added to a portion of the PCB mixture before liquid chromatography separation. Also, acetone was added to a Carius tube and evaporated; then a perchlorination reaction was run on the acetone residue.

The reaction products from the perchlorination of the acetone solvent residue were very similar to the products from the perchlorination of dibenzofuran and the LC fraction from the PCB mixture. However, examination of the GC charts reveals several GC peaks which are not attributed to reaction products from the residue of acetone. The peak areas and retention times of these components are shown in the attached table. The chromatograms are also attached.

At the request of Don Wilkins the final reaction product mixture from the dibenzofuran and the acetone LC fraction of the PCB mixture are being sent to General Electric.

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GC PEAK AREAS AND RETENTION TIMES FOR
COMPONENTS FROM PERCHLORINATION REACTIONS

(Area units are arbitrary integrator units divided by 10^6)

Reaction Mixture Code	Starting Material	Retention Times (Minutes)					
		<u>0.41-0.45</u>	<u>0.42-0.47</u>	<u>3.16-3.17</u>	<u>3.23-3.1</u>	<u>8.25-8.49</u>	<u>8.73-8.88</u>
)-6-2A	Dibenzofuran	-	-	-	0.4	-	0.04
)-6-4A	LC Fraction of PCB Mix	-	-	-	35.6	6.4	10.5
)-12-3A	Dibenzofuran	12.5	12.5	0.5	0.5	0.4	0.3
)-12-2A	LC Fraction of PCB Mix Spiked with Dibenzofuran	19.2	20.5	1.6	1.7	0.7	0.7

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