Spectroscopy
Method No. 71-15
Job No. 8502009

CHARACTERIZATION OF PRECIPITATES FROM AROCLOR 5432 OR FROM FLUIDS FORMULATED WITH AROCLOR 5432

SCOPE

A white precipitate occasionally forms in Aroclor 5432 or in fluids which contain Aroclor 5432. In several instances this precipitate has been identified as one isomer of dichloroterphenyl (DCT). This method provides a means for determining whether a precipitate is this DCT isomer.

PRINCIPLE

The undesirable DCT isomer, if present in the precipitate, can be isolated and detected by flame ionization gas chromatography. Comparison of the sample chromatogram with a reference chromatogram run under the same conditions allows both identification and quantitation of this component. If the precipitate is not this DCT isomer, other methods of identification will be required.

EXPERIMENTAL

A. Sample Preparation

1. The precipitate to be characterized must be acetone washed and dried. This can be accomplished conveniently on the filter paper used to isolate the solid particles from the fluid. (If the precipitate dissolves during this step, it is not DCT.)

2. Weigh approximately 15 mg. of precipitate, to the nearest 0.1 mg, into a 10 ml volumetric flask. Dilute to the mark with n-butyl phenyl ether (Aldrich Chemical Co.). Allow the solids to dissolve. Dissolution time can be shortened to about one hour by warming the flask to 40-50° C. Cool before proceeding.

B. Gas Chromatographic Analysis

1. Gas chromatographic conditions are as follows:

detector	- flame ionization
column	- 2 in x 1/8 in stainless steel packed with 10% SE-52 on 80-100 mesh High Performance Chromosorb W
temperatures	
oven	- 280° C.
detector	- 300° C.
injection port	- 300° C.
flow rate	- 30 ml helium/min.

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2. "Dichloroterphenyl Reference Solution" was prepared by dissolving 2.8 mg of solid DCT in 2.0 ml butyl phenyl ether. (Reference DCT is available on request.) If a precipitate has formed in the solution, warm to dissolve solids. Inject a 2 µl aliquot of "Dichloroterphenyl Reference Solution" into the chromatograph and record the chromatogram. This DCT isomer should elute in about 6 minutes under these conditions. Attenuate so the DCT peak is at least half scale.

3. Chromatograph a 5 µl portion of the dissolved precipitate, using the same attenuation as in B-2. If a peak is observed with the same retention time as the DCT reference peak, the precipitate is DCT.

4. Calculate the percentage of DCT in precipitate using the following equation:

$$\text{Weight \%} = \frac{14}{\text{Sample Weight (mg)}} \times \frac{\text{Peak Height (unk)}}{\text{Peak Height (ref)}}$$

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9/71 - J. P. Mieure, M. W. Dietrich

INDUSTRIAL CHEMICALS CO.

RESEARCH & DEVELOPMENT

Report

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ON

PHYSICAL CHEMICAL [redacted] - 1971

September 1972 WRITTEN BY - E. M. Emery, et al

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ST. LOUIS TECHNOLOGY DEPARTMENT

St. Louis Research and Development Department Report No. 3784

FINAL REPORT
ON
SPECIAL PHYSICAL CHEMICAL STUDIES - 1971

Job No. 2-21-760.01-8503022

Date: September 1972

Written by: E. M. Emery
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*Appreviated report containing only the index, section titles, introduction and summary

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INTRODUCTION

All of the special studies in physical chemistry done in the Monsanto Industrial Chemicals Co., St. Louis Research and Development Laboratories, during 1971 are compiled in this report.

SUMMARY

Following is a listing by title of all of the special study reports compiled in this Final Report:

Special Study No.	Title
71-1	Microscopic Comparison of Recent Vulcan Stannous Oxalate Catalyst Crystals with Older Production
71-2	Thermal Stability of Benzoic Acid with Triethylamine (1.0 to 0.025 Molar)
71-3	Composition of Competitive Polychlorinated Biphenyl Products
71-4	Thermal Stability of AMAC Process (L-DOPA Intermediates)
71-5	Application of Gamma Radiation Transmission Techniques to Density Measurement of WGK PNA Continuous Process Stirred Reactor
71-6	Thermal Stability of Azo Methine Residues (Lasso Process)
71-7	Thermal Stability of 3,4-Dichloroaniline (DCA) Still Residue (12/17/70)
71-8	Thermal Stability of NCP, CPDPP and o-XDP
71-9	Thermal Stability of Aroclor 5060/NaOH/Lime (Low Color Aroclor 5460)
71-10	Thermal Stability of M-5538 (Ethylene-bis-Isopropyl/ Isobutyl Xanthate)
71-11	Composition of Chlorinated Biphenyl Products in the Range from 8 to 42% Chlorination Level
71-12	Thermal Stability of a 75/25 Mixture of Phthalyl Chloride and Therminol 66
71-13	Thermal Stability of MDP and Santicizer 141 Solvent Still Residues with Calcium Oxide

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Special Study No.	Title
71-14	Mercury Inventory, WGK Chlor-Alkali Department, March 1971
71-15	Mercury Inventory, Alcoa Chlor-Alkali Plant, Point Comfort, Texas, March 1971
71-16	Thermal Stability of Hitec Salt from Phthalic Anhydride Reactor R1-4 (5/6/71)
71-17	Thermal Stabilities and Heat of Reaction for New L-DOPA Catalyst System
71-18	Thermal Stability of Hydrogenated Linear α-Methyl Styrene Dimer (HLD) Process Streams
71-19	Thermal Stability of Salicylic Acid (LPC) Mother Liquor Fractionation Residues
71-20	Thermal Stability of Dichloroaniline (JFQ Surge Tank, 3/10/71)
71-21	Thermal Stability of L-DOPA and DAMT
71-22	Physical Properties of o-Chlorobiphenyl
71-23	Thermal Stability of ODCB Reactor Heel/Therminol 66
71-24	Thermal Stability of Maleic Anhydride Still Residues
71-25	Viscosity of HB-30
71-26	Santicizer 140 Crystallizing Points
71-27	Dicyclohexylphthalate - Microscopy Studies
71-28	Thermal Stability of Maleic Anhydride with Phthalyl Chloride Color Stabilizer
71-29	Electrodeless Conductivity Meter for the LPC Salicylic Acid Process
71-30	Thermal Stability of Partly Decomposed Azo Still Residue (Lasso Process)
71-31	Benzyl Salicylate Heat of Reaction by DSC
71-32	Thermal Stability of Santoflex 13 and 77 Finished Batches
71-33	Physical Properties of Aroclor 6000 Blends

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Special Study No.	Title
71-34	Thermal Stability of Santicizer 8 (o/p Mix of N-Ethyl Toluenesulfonamides)
71-35	Thermal Stability of a Mixture of Benzyl Chloride and Therminol 66
71-36	Thermal Stability of Trimethoxy Benzaldehyde Process Streams
71-37	Mercury Inventory, B. F. Goodrich Company, Calvert City, Kentucky, August 1971
71-38	Thermal Stability of Ortho and Para-Nitroanisoles
71-39	Thermal Stability of Methyl Salicylate Still Residues (JFQ)
71-40	Thermal Stability of Monomethylammonium Citrate and Glutarate Solutions
71-41	Heat of Ozonolysis of Maleic Acid (Riedel Ethavan Process)
71-42	Mercury Inventory, Alcoa Chlor Alkali Plant, Point Comfort, Texas, September 1971
71-43	Examination of Soda Springs, Idaho Plant Water Residue for Natural Radioactivity
71-44	Heat of Reaction of Methyl Salicylate and Aqueous Ammonia (Salicylamide Process)
71-45	Thermal Stability of Glyoxylic Acid (Riedel Ethavan Process)
71-46	Thermal Stability of Parachloroaniline (Catalytic Reduction Process)
71-47	Mercury Inventory, W. G. Krummrich Chlor Alkali Department, July 1971
71-48	Mercury Inventory, W. G. Krummrich Chlor-Alkali Department, November 1971
71-49	Investigation of Continuous Process p-Nitroaniline Quality Problem by Microscopy and Thin Layer Chromatography
71-50	FDA Extraction Work on ^{14}C Tagged RPC 1604 and RPC 1108
71-51	Thermal Stability of 4-NDPA Feed to Luwa #1 (Yield Loss Study)

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