

**O R G A N I C
C H E M I C A L S D I V I S I O N
R E S E A R C H**

Report

REPORT NO. 1-21-60-1630043
FOR
MOS 747
(Temperature Controller Fluid)

DATE - January 27, 1969 WRITTEN BY - J. D. Sullivan



ST. LOUIS, MO.

NEV 024991

PLAINTIFF'S
EXHIBIT

1464

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The fluidity of the finished product has not been firmly established but reasonable ranges for viscosities and pour points are listed above. These ranges were determined by laboratory runs and pilot plant demonstrations.

The effects of higher boiling Aroclor isomers on the fluidity of the product increases as the temperature is reduced. However, low temperature viscosities (i.e. +20°F) have been very consistent with pour point determinations. Furthermore, low temperature viscosities are more accurate, more reproducible and more meaningful than absolute pour point determinations.

Pour points were determined in regular ASTM tubes suspended from a specially designed holder, which was mounted inside a frost free cold chest (Thermotron Corp.), equipped with accurate temperature controls. By means of an external rotating device, the tubes were rotated, through a 45° angle, from the vertical position to a horizontal position and the timing for the test was started. Flowing motions, on the surfaces of the constant temperature fluids, were observed through a frost-free sight glass. The lowest temperature, at which flow was observed within 5 seconds (ASTM), was designated as the pour point of the fluid.

TOXICITY AND HAZARDS

Aroclor is a liquid, under normal conditions, having a medium toxicity range for liquid ingestion and a high toxicity range for vapor inhalation. The maximum allowable concentration is 1 mg/cubic meter. This material can cause dermatitis, systemic poisoning from the fumes and yellow atrophy of the liver. There are skin, mucous membranes and eye irritation encountered in handling Aroclors. However, the crude Aroclor, used as the starting material in this process, is saturated with HCl. Hydrogen chloride is a gas and is very toxic. It is hazardous to teeth, skin, eyes and mucous membranes. Aroclors offer no fire hazard.

Aluminum chloride reacts with small amounts of water with such violence as to start a fire in the presence of organic materials. Containers of this material should be stored completely enclosed or under a dry inert atmosphere. It reacts with moisture in the skin or mucous membranes to form a strong acid which can result in severe burns. Avoid inhaling the dust and wash affected areas with large amounts of water. Wear rubber gloves and goggles when handling this material.

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Calcium hydroxide is not dangerously toxic but is very hazardous to the eyes. It will burn the skin and the dust will affect the mucous membranes. In general, Attapulugus earth is not toxic. Persons handling large amounts of this absorbent should wear a respirator to avoid inhalation of the dust which contains silica.

POLLUTION CONTROL

The following weights of waste materials will have to be disposed of from the various process steps in the production of MCS 717.

Pounds of Waste Materials per 100# MCS 717

<u>Process Steps</u>	<u>Pounds</u>
Isomerization & Degassing	0.01# HCl
Neutralization	None
Distillation	5# to 10# Residue
Attapulugus Earth Treatment	0.5# Attapulugus
	<u>1.0# Porocel</u>
Filter Cake	1.5#

Residual HCl contained in the original crude and the small amount of HCl, evolved during the isomerization, will be dissolved in a water scrubber and sewerred. The still residue, including chlorination tars, isomerization polymers, iron, aluminum and calcium salts along with highly chlorinated biphenyl, is transferred hot to a suitable container and sent to the toxic dump. The filter cake, from the Attapulugus Earth treatment, will go to the solid disposal interment.

J. D. Sullivan

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1/17/69

File Copy Approved by
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W. R. Richard

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MONSANTO COMPANY
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May 13, 1969

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Tentative Amendment "G"

TITLE

Tentative Amendment "G" to the Standard Manufacturing Process for the manufacture of Aroclors, Department 246, dated October, 1966.

SUB-TITLE

Demonstrate quality improvement of Aroclor 1254, as measured by stability tests, by use of modified plant conditions.

PRESENT PRACTICE

Biphenyl is preheated to 120°C, charged to the batch chlorinator, 0.05% ferric chloride is added, and a controlled flow of chlorine is turned on. The reaction mass temperature increases to 150°C where it is controlled. When the proper specific gravity of the reaction mass is attained the chlorine flow is stopped.

The batch is pumped to an air blown tank where the residual hydrogen chloride is removed. It is then distilled from lime, earth treated and stored for sales.

CHANGES

It is the intent of this amendment to permit the demonstration of the effect of iodine as a co-catalyst in the chlorination of biphenyl. It is proposed that 0.05% iodine be added with the ferric chloride. All other conditions would remain the same.

PLAINTIFF'S
EXHIBIT

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JUSTIFICATION

It is the objective of the Functional Fluids Business Group to produce Aroclor 1242 with a thermal chemical chloride (TCC) test limit of less than 0.1 ppm. The use of the iodine-ferric chloride catalyst system is the only action which has consistently achieved this goal in the laboratory.

No improvement in the stability of plant produced Aroclor 1242 was experienced when iodine co-catalyst was used during four plant tests. It is thought that these failures were due to a masking effect by organic impurities in the chlorine from the Hooker chlorine facilities. These impurities have been reduced since the Hooker shutdown.

Aroclor 1254 was chosen because improvements similar to those of Aroclor 1242 have been seen in the lab work. Since plant produced Aroclor 1254 exhibits a higher TCC average compared to Aroclor 1242 (0.64 vs. 0.14 for first quarter 1969), the effect of iodine in the plant should be easy to document. If there is an improvement in Aroclor 1254 stability, a plant test in the continuous Aroclor 1242 line will be requested. If there is no improvement found in the Aroclor 1254 plant test, no further plant tests or laboratory work will be conducted until it is deemed necessary by the Business Group.

SOURCE OF DATA

The Process Research group at W GK has extensively investigated iodine as a co-catalyst in the Aroclor chlorination step. Variables investigated have included chlorination temperature, FeCl_3 content, iodine content and biphenyl variability. All the data indicates iodine improves the thermal chemical stability. The electrical properties are not affected.

SAFETY

Iodine is a severe irritant to the skin and lungs. Care should be taken to avoid skin contact or inhalation of the vapors.

The effect of iodine vapor upon the body is similar to that of chlorine or bromine, but it is more irritating to the lungs.

QUALITY

The batches made during the plant test will be processed as non-electrical grade Aroclor 1254. Laboratory chlorinated material using iodine as co-catalyst meets all specification properties and exhibited excellent stability.

QUALITY (Cont'd.)

Based on off-gas analyses during past iodine tests, the off-gas could contain as much as an average of 1-2 parts per million of iodine. This could result in approximately 1 ppm in the muriatic acid produced in Dept. 218. The acid will be analyzed for iodine and will be sewerred if it contains more than 5 ppm; it will be sold as industrial grade if it contains from 1 to 5 ppm and food grade if it contains less than 1 ppm. The test batches will be scheduled with Dept. 236 batches so that no Dept. 246 gas is transferred to Dept. 217.

EXTENT OF DEMONSTRATION

The demonstration will consist of three consecutive batches from the same chlorinator, each utilizing 0.05% iodine and 0.05% ferric chloride based on biphenyl. At least six normal batches will be taken as baseline. The batches will be sampled at the 1242 and 1254 stage and processed in the laboratory. All four stability tests will be measured for each sample in the analytical section.

CRITERIA FOR ACCEPTANCE

This amendment is to allow temporary demonstration of modified plant conditions and further demonstration would be necessary before adoption.

Written by: C. W. Hamlet, Jr.
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Approved by:

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Div. Resch. Group Leader Date

Manager of Research Date

Research Safety Review
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