WGK ANALYTICAL RESEARCH AND DEVELOPMENT REPORT No. 1771, November 19, 1970

OTHER LOCATIONS	GENERAL OFFICE	W. G. KRUMMRICH PLANT
		B. Corlew R. McCutchan D. Armstrong W. Engman W. Krull D. Jackson R. Blowers

TITLE: Aroclor 1260 Quality Problem. Status Report No. 2

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PROBLEM: Aroclor 1260 produced by Dept. 246 is out-of-specifications on after corrosion chlorides.

JUSTIFICATION: To produce quality 1260 Aroclor and retain sales.

SUMMARY: A series of chlorinations in a laboratory scale unit using various sources of raw material were performed. Also side by side chlorinations were made with the plant equipment and the laboratory unit in the department. No positive solution has been determined to date. A designed experiment of the plant chlorinators and specified variables has been started.

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Department 246 has encountered problems in production of Aroclor 1260. The material is out-of-specifications on after corrosion chloride and sometime out-of-specifications on the Monsanto stability.

To determine where in the process the bad material was made, crude 1160 from each of the batch chlorinators was distilled and porocel treated in the laboratory. All five were out-of-specification on after corrosion chloride but good on Monsanto stability. This indicated that the problem was not in the distillation step but either the chlorinator or raw materials.

To evaluate raw materials, a laboratory scale chlorinator was charged with plant biphenyl and catalyst. Bottled chlorine was used. The material produced was good quality 1260. A second chlorinator run made using biphenyl purchased from Fisher Scientific and bottled chlorine also produced good quality 1260. This would indicate that the biphenyl raw material was satisfactory to produce quality 1260.

To evaluate the chlorine supply, the laboratory chlorinator was moved to the department and connected to the plant chlorine supply. The chlorinator was charged with crude 1154 and chlorination continued to 1160. Laboratory distillation and porocel treatment produced in-specification material. The plant-produced material was out-of-specification.

A side-by-side run of a plant chlorinator and the laboratory scale chlorinator was performed. The plant chlorinator was charged with biphenyl and catalyst and mixed for 10 minutes. This material was sampled and used to charge the laboratory chlorinator. Materials from the chlorinators were distilled and treated with porocel in the laboratory. Again the laboratory scale chlorinator produced quality material and the plant out-of-specification.

Comparison of the variables common to both chlorinators showed three areas of difference. (1) Starting temperature of chlorination: plant was 110°C; laboratory 150°C; (2) Chlorine addition rate: plant was stepwise of 400, 800 and 1200#/hr; laboratory was constant; (3) Demister: plant chlorinator has one, laboratory did not.

The laboratory chlorinator was adapted with a demister and a second side-by-side run performed. The laboratory chlorinator was controlled in starting temperature and chlorine addition rate to correspond with the plant. Laboratory distillation and porocel treatment produced in-specification material for the plant and out-of-specification for the laboratory chlorinator. Plant distillation and treatment did not produce in-specification material.

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A designed experiment involving the four operating plant chlorinators is planned. It will include the changing of the following variables.

(1) Doubling the charge of $FeCl_3$ catalyst.

(2) Increasing the starting temperature to 150°C.

(3) Increasing the finishing temperature to 190°C.

(4) No change - to establish a base.

This work is currently being performed and will be reported in the next status report.

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