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TENTATIVE PROCESS FOR MCS 717 (Low Temperature Capacitor Fluid)

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INTRODUCTION

This report is a Tentative Process for the manufacture of MCS 717, a low temperature capacitor fluid, prepared by the isomerization of a selected chlorinated biphenyl. It will supercede the process for MCS 717 (Report #P-1501) which was successfully demonstrated in the JFQ Pilot Plant during October, 1968. Demands for larger quantities of this product necessitated the modification of the process so that the reaction could be adapted to existing Aroclor production facilities at WGK and/or Anniston.

SYNOPSIS OF PROCESS

Crude undegassed Aroclor (spec. grav. at 25°/25°C-1.3450 to 1.3500) is isomerized with $AlCl_3$ (1%-175°C-2 hrs), degassed with plant air (1.5 NN max.), neutralized with calcium hydroxide and straight-takeover distilled. The distillate is treated with Attapulgus earth (0.5%-2 hrs-60°C) followed by a Porocel filtration* into thoroughly cleaned and rinsed double phenolic lined drums.

* see Bill of Raw Materials

BILL OF RAW MATERIALS

The following weights of raw materials are required to produce 100 pounds of MCS 717.

	Pounds	Gallons	lbs/gallon
Crude Aroclor	110	9.82	11.20
Aluminum Chloride	1.1		
Calcium Hydroxide	0.5		
Attapulgus Earth	0.5		
Porocel-60 Mesh*	1.0		

* This medium is used to pack the filter for the removal of Attapulgus earth from finished product.

PROCESS IN DETAIL

(1) Isomerization

To a reactor, equipped with a stirrer and a condenser leading to a HCl scrubber, is charged 110 pounds of crude undegassed Aroclor (1.3450 to 1.350 at 25°/25°C) and 1.1 pounds of dry $AlCl_3$. The reaction mixture is held at 175°-180°C for 2 hours to accomplish the isomerization. If heating facilities are not available to attain these isomerization temperatures, the chlorination of the original biphenyl can be finished-off at 180°C to 190°C to supply residual heat for the hold period.

(2) Degassing

The batch is then cooled to 150°C and degassed with the introduction of plant air until the acidity of the crude is reduced to 1.0 NN-1.5 NN. This was accomplished in the laboratory, in 2 to 3 hours at flow rates equivalent to 1.0 cu.ft/hr/lb to 0.5 cu.ft/hr/lb. After the batch has been degassed, the crude is transferred to the distillation equipment.

(3) Neutralization

After the transfer of the crude to the distillation still is complete, 0.5 pounds of calcium hydroxide is slurried into the batch at 150°C. The mixture is stirred at 150°C until the acidity of the crude is reduced to 0.5 NN maximum. In the laboratory, this reduction in acidity was accomplished in less than an hour, under these conditions.

(4) Distillation

The crude is distilled into a stirred receiver, equipped with a constant recording specific gravity device. The distillation is continued until the specific gravity of the product approximates the specific gravity of the starting crude within + 0.005 units at 25°C/25°C. In the final stages (10% to 15%) of the distillation, the temperature of the condensing water is increased to 60°-65°C to allow the more viscous distillate to flow into the stirred receiver.

In the laboratory, this material was distilled through a 1 1/2" punched column in a pot temperature range of 207° to 230°C at 30 mm. The vapor temperature at the top of the punched column varied from 190° to 225°C at 30 mm during this distillation. A spray-trap type column is sufficient for this operation if the last 10% to 15% of the product is carefully distilled to prevent excessive entrainment of high polymeric materials from the pot residue.

Laboratory yields of distilled product ranged from 90 to 94% of the still charge. The residue from this isomerized crude contains approximately 3 times more solids than the residue from a regular Aroclor distillation.

(5) Attapulgus Earth Treatment

To the well mixed distillate is charged 0.5 pounds of activated Attapulgus earth and the resulting slurry is stirred for 2 hours at 60°C prior to a Porocel filtration* at 60°C to 65°C. The Attapulgus earth and the Porocel, used in this treatment, are activated by roasting the absorbents for at least 4 hours in a 250°C oven.

* see Bill of Raw Materials

(6) Packaging

The filtrate represents an electrical grade fluid and extreme care must be exercized in its handling, sampling, packaging and storage. Trace contaminations, especially ionic impurities, will completely deteriorate the electrical properties of the fluid and make it unacceptable for the application.

Transfer lines for the filtrate must be thoroughly cleaned, dried and flushed with the finished fluid. The same successive flushes may be used to rinse the double phenolic lined drums for fluid storage. The rinses are reintroduced into the process during a successive Attapulgus earth treatment.

If the finished product does not attain the specifications of an electrical Aroclor (power factor and resistivity) the fluid is reexposed to another Attapulgus earth treatment. When the drums are transferred to storage, care must be taken not to chip the internal phenolic coating by rough handling.

DISCUSSION OF IMPORTANT VARIABLES

(1) Isomerization

The main feature of this new capacitor fluid is a 10° to 15°C reduction in the pour point for low temperature applications. Because of this specification, it is important that the directly chlorinated Aroclor has a specific gravity of 1.3450 to 1.3500 at 25°/25°C before using the raw material for this process. Chlorinated biphenyls, adjusted to the desired specific gravity by the "back-blending" of Aroclors, cannot be used in the preparation of MCS 717. Specific gravities on the well mixed crudes are determined in standardized pycnometers.

Hydrogen chloride is an initiator for the dry $AlCl_3$ catalyzed isomerization. The crude undegassed Aroclor is saturated with by-product HCl and no effort should be made to degas the material before the isomerization.

(2) Degassing

Regular plant air is used for degassing in the production of Aroclor. Laboratory experiments show that dry air, dry nitrogen or air saturated with water vapor may be used in this degassing operation.

(3) Neutralization

After the crude has been degassed and transferred to the still, calcium hydroxide is added to the batch to complete the neutralization and to protect the iron equipment during the distillation. Calcium hydroxide is also added to the still pot charge to improve the quality of the distillate by taking up any HCl which may form, due to slight decomposition of the fluid, during the distillation. Furthermore, the $Ca(OH)_2$ promotes the removal of any Cl-groups which have reacted by addition rather than substitution, in the original biphenyl chlorination. This reduction in labile chlorine improves the chloride stability of the finished fluid for electrical applications.

(4) Distillation

The most important factor in this distillation is the realization that Aroclors are not pure materials. Aroclor contains innumerable, closely boiling isomers having widely varying fluidities. As the distillation temperature increases, the chlorine content and the viscosity of the distillate increases. These are the reasons for preheating the condensing water and agitating the contents of the receiver. The higher boiling isomers affect the fluidity of the product throughout the entire viscosity range but especially at the pour point.

Isomerization substantially increases the specific gravity of the crude Aroclor even after the neutralization solids are removed. An additional purpose of this distillation is the separation of the higher isomer products from the polymerized materials formed during the isomerization of the crude Aroclor. Available spray-traps and/or demisters are recommended for this operation. If a highly effective column is used, representative isomerized Aroclor cannot be obtained at reasonable temperatures and pressures. Increasing the specific gravity of the starting raw material would not overcome this distillation end-point difficulty. This

alternate gives a higher viscosity fluid at the desired specific gravity level and is accompanied by substantial yield losses in the still residue.

During the isomerization and degassing, $AlCl_3$ as well as acid tars sublime onto the free-board of the reaction vessel. For this reason it is imperative to transfer the batch to another pot before distilling the crude. The crude can be distilled from the same reaction vessel if the batch is held at reflux, under reduced pressure, until the return flow is colorless and the acidity of the batch is 0.5 NN or less.

After the isomerization run is completed the plant equipment may be cleaned out with a regular run of Aroclor 1248. It is recommended that this batch of Aroclor 1248 be segregated from the regular Aroclor production and be used for the formulation of Pydraul fluids.

(5) Attapulgus Earth Treatment

Attapulgus earth treatments are very effective in lowering the power factor and increasing the resistivity of electrical grade Aroclor fluids. Samples of the finished fluid, submitted for electrical properties, must be withdrawn through specially cleaned pipettes or thiefs into specially cleaned amber bottles with aluminum foil-lined caps. The finished fluid as well as the inner surfaces of sampling devices and sample containers must be protected from outside contamination and must not be touched with "salty" human hands. If the finished goods is stored for a prolonged period of time in plant equipment, awaiting analyses and/or packaging, it is recommended that the batch be blanketed with a nitrogen atmosphere.

In the pilot plant demonstrations of MCS 717 the filters were lined with Porocel (60 mesh) to speed the flow rates and climinate possible contaminations by other suitable filter aids. In large scale production, the passage of the distilled material through a fixed bed of Porocel would be a suitable purification for this product.

(6) Packaging

The same precautions employed in handling and sampling of electrical fluids must be observed in their packaging and storage. Double phenolic lined drums are satisfactory for general storage and local hauling. For

long distant shipping, involving the usual rough handling, a double epoxy-phenolic lined drum is recommended. These drums may be purchased from Bradey-Voorman (Chicago), Nasco (Granite City) or Florida Drum Company (Pensacola, Fla.).

MATERIAL SPECIFICATIONS, ANALYTICAL METHODS

A. Material Specifications

(1) Raw Materials

(a)	Crude Aroclor	Specific Gravity 1.3450 to 1.3500 at 25°/25°C
(b)	Aluminum Chloride	Code #11560
(c)	Calcium Hydroxide (Hydrated Lime)	Code #38800
(d)	Attapulgus Earth	Code #19400
(e)	Porocel (60 mesh)	Anniston

(2) Finished Product

Specific Gravity at 25°/25°C within 0.002 to 0.005 units of crude

Viscosity at 210°F	1.9 to 2.2 cs.
at 100°F	10 cs to 11 cs
at +20°F	600 cs to 800 cs
Pour Point	-30°C min. (-32°C to -36°C typical)
Dielectric Constant @100°C	4.3 min.
Resistivity @100°C	5000 x 10^9 ohm-cm
Power Factor @100°C	60 cycles 0.3%
	100 cycles 0.1%

	WGK Method Number
Inorganic (Free) Chlorides-report	T2092
Hydrolysis Stability Test - 0.2 ppm (max)	T2087
Monsanto Stability Test - 0.02 ppm (max)	11,503
Thermal Chemical Chlorides - 0.5 (max)	12,722

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.

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, Attapulgus 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

Process Steps	Pounds
Isomerization & Degassing	0.01# HCl
Neutralization	None
Distillation	5# to 10# Residue
Attapulgus Earth Treatment	0.5# Attapulgus
	1.0# Porocel
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 sewered. 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 Attapulgus Earth treatment, will go to the solid disposal interment.

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