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ABSTRACT:

This report describes a study on "Continuous Flash Distillation" of crude Monoisopropyl-biphenyl (MIPB). MIPB is a component in MCS-1238, a potential replacement for Aroclor.

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Progress Report No. 1
MIPB Distillation Studies
Job No. 1615103

INTRODUCTION

The use of polychlorinated biphenyls (PCB) for capacitor dielectric fluids may eventually be discontinued due to environmental considerations. MIC R&D is currently developing substitute, non-PCB, fluids for capacitor use. A blend of tolyl xylyl sulfone (TXS) and monoisopropylbiphenyl (MIPB) is one fluid being considered. MIPB however, must be refined prior to blending as prescribed in PED document P-1797, "Blending, Refining, & Drying of MCS 1238 - A Dielectric Fluid". Work described in this report concerns MIPB refining by continuous flash distillation. The study was carried out during Jan./Feb., 1976 by the WGK Plant Process Research group. The work was done primarily to determine boiling point rise versus through-put. This information was needed to determine if similar type refining might be possible in current WGK Aroclor equipment using 200 lbs. steam as heat source.

SUMMARY

Continuous flash distillation of crude monoisopropylbiphenyl (MIPB-2) obtained from Dixie Chemical Co. was carried out in a laboratory apparatus. The unit was operated at 25 mm Hg pressure. High boiler residue was allowed to concentrate in the pot during the distillation while maintaining a fixed pot volume. Pot temperature rose from 183°C to 225°C after about 250 through-puts. This translates to $<$0.5% residue or a yield loss of less than 0.5 lbs./100 lbs. crude feed.

Some foaming was observed, particularly when feeding subsurface. Good agitation is essential, or foaming and bumping are intolerable. Distillate color darkened gradually with through-puts as well as with increased rates, indicating some entrainment. A demister in the vapor duct significantly improved distillate color. Corrosion to a steel coupon in the pot was nil (39 hrs.). There was no evidence of insolubles (inorganic salts) buildup in the pot residue or distillate lines. Product distillate was blended with TXS by R&D at G. O. Acceptable quality MCS-1238 resulted. Pot residue was a dark brown liquid, reasonably mobile at room temperature. The residue has been sent to R&D for further evaluation.

Use of current Dept. 248 equipment for similar refining, but with 200 lbs. steam heat, does not appear feasible. Heat from 200 lb. steam would not be adequate to achieve the temperatures required.

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DETAILS

I. EXPERIMENTAL EQUIPMENT

A schematic of the apparatus used in the distillation studies is shown on Figure No. 1. Material was fed via a microbellows pump which was actuated "ON/OFF" by the photocell level controller. The photocell was triggered ON/OFF by a float on the surface of the liquid in the level controller tube. A spring loaded relief valve, set to open at pressures above 17 lbs. was placed in the feed line between the pump and the distillation system. This prevented material from being drawn into the system unless the feed pump was operating. Except for the final part of the run, the pot level was maintained at 125 ml by height adjustment of the level controller. There was no measurable pressure difference between the pot and condenser exit.

II. GLC METHOD OF ANALYSES

Hewlett Packard Model 5712A Gas Chromatograph equipped with TC detector and appropriate integrator was used for semi-quantitative analyses of feed, distillate and residue samples. Conditions were as follows:

Column	6' x 1/8" s.s. - 10% W 98 on Chrom W, 80/100 mesh
Detector	350°C.
Injection	280°C.
Oven	140°C isothermal 4 min, 8°C/min → 320°C and hold 16 min.
Carrier	Helium - 30 ml/min.

1.0 µl Injection of "as is" sample.

Attenuation 8 X1
Bridge Current 110 ma

Quantitation is by normalization of total area of all eluting components. No compensation is made for possible non-eluting compounds or variation in response for the individual components.

III. DISCUSSION

A. SYSTEM START-UP AND OPERATION

In start-up, the system (Figure No. 1) was first evacuated to 25 mm Hg pressure. The feed pump was started and 125 ml pumped into the distillation pot. Heating was then started. When distillate began to come over, automatic feeding was started. Pot and distillate lines were insulated with three layers of insulation. Pot heat was supplied by resistance wire and regulated by powerstat variable transformer. Temperatures in the pot (vapor & liquid) and distillate vapors (head) were monitored by thermocouple and plotted vs time by multipoint recorder.

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III. DISUCSSION (Cont'd.)

The system was operated at various feed/distillate rates. Operating characteristics and distillate quality were observed at the different rates.

Tempered water was used on the condenser in order to get only partial condensation of water vapor from the distillate, and thus a lower moisture content in the product.

Condensate collected in the cold traps was not quantitated due to an upset which caused product to be drawn into the traps (receiver overflowed and backed into traps). Data and comments from the run are found on TABLE No. 1.

B. FOAMING/BUMPING

The pot was fed subsurface for the first 3 - 4 through-puts. Much foaming occurred. This condition was probably due to degassing of the feed as well as flashing of low boilers such as moisture. A degassing surge vessel might eliminate this problem. With surface feeding, foaming was negligible.

Good agitation is essential, or bumping is intolerable. This is true regardless of surface or subsurface feeding.

Although surface feeding was used for most of the run, boiling was observed over the entire surface area of the pot liquid. This indicates the feed material for the most part went into the pot liquid before boiling (e.g. pseudo-subsurface feeding).

C. BOILING POINT VS THROUGH-PUT

The initial boiling point of the pot material was 183°C. (25 mm Hg). The boiling point increase with through-put rose quite rapidly early in the run. Boiling point increase from 195°C. and up was quite slow (see TABLE 1 data). The distillate head temperature remained about 183°C., throughout the entire run regardless of through-put or rate. Early temperature readings of about 165°C. are throught to be in error due to inadequate insulation on the distillate lines.

D. PRODUCT COLOR

Three factors had a significant influence on distillate color. They were entrainment, vapor disengagement rate, and through-put (see TABLE 2). Product color slowly increased from 8 to 30 apha during the first 52 through-puts, while running at a constant disengagement rate of about 0.7 grams/min/in^2. Pot liquid also gradually darkened during this period, suggesting the product color darkening was probably due

III. DISCUSSION (Cont'd.)

to pot liquid entrainment. When the disengagement rate was reduced to about 0.2 grams/min/in^2, at about 55 through-puts, product color improved to 8 apha. There was probably less entrainment at the lower disengagement rate.

A "Goodloe" Type A Column, 25 mm diameter and 3 inches high, was installed in place of the "Ace" adapter distilling trap. Product color improved from 30 to 5 apha with the change. Disengagement rate was then increased to about 1.1 gram/min/in^2 . Product color darkened to 20 apha indicating continued entrainment, but not nearly as severe as when running with the Ace distilling trap. Product color did not appear to change with increasing through-put (due to pot volumn reduction) after installing the Goodloe column (compare samples 18 and 21, TABLE No. 2). The Goodloe column is packed with stainless steel gauze.

Distillate sample No. 15 was evaluated for acceptability in MCS-1238 by Functional Products R&D at G.O. The material was satisfactory in all respects.

E. FEED & DISTILLATE MOISTURE CONTENT

Moisture content of feed and distillate material were determined by Karl Fisher method using methanol as solvent.

	H_2O
Feed	125. ppm
Distillate Sample No. 3	$<$2.5 ppm
Distillate Sample No. 17	16 ppm

Distillate sample No. 3 may be erroneously low due to air leaks into the distillation system. The specification of $<$30 ppm is easily achieved.

F. CORROSION TO STEEL

The steel corrosion coupon positioned in the pot during the distillation showed essentially no corrosion for the duration of the run. (39 hours exposure at temperatures above 195°C.). There was no difference in corrosion to the coupon between vapor and liquid contact. Any effects the steel coupon may have had on product quality or residue characteristics are not known because no portion of the run was made without the coupon present. No gross effects were apparent however, since product quality was acceptable.

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III. DISCUSSION (Cont'd.)

G. RESIDUE AND SYSTEM YIELD

Residue collected, after boil down and operation at 225°C. pot temperature, was 33.0 grams. GC indicates the material to be 45-75% high boilers. The residue was dark brown in color and was still semi-mobile at room temperature. There was no evidence of any insoluble type substance, such as inorganic salts in the residue or distillate lines. Total through-put in the system was 8355. grams. At most, the high boiler content in the feed was therefore:

$$\frac{0.75 \times 33.0 \times 100}{8355.0} = 0.30\%$$

Yield loss as residue was:

$$\frac{33.0 \times 100}{8355.0} = 0.39\%$$

This represents a system yield loss of < 0.5 lbs./100 lbs. crude feed.

The residue has been given to Functional Products R&D for further evaluation.

H. GLC ANALYSES OF FEED, PRODUCT, & RESIDUE

TABLE No. 3 summarizes GC analyses of Feed, Product, and Residue. There was no appreciable change in distillate composition throughout the entire run. High boiler content gradually increased in the pot material (residue) during the run.

Some of the crude MIPB remaining in the residue at end of run may be recoverable by further boil down.

Chromatograms of crude MIPB, distillate, and residue samples are appendixed (Figures 2 thru 5).

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