Physical Chemistry
Method No. 70-15
Job Nos. 0435122
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DETERMINATION OF POLYCHLORINATED BIPHENYLS IN AROCLOR 5460 BY CARBON SKELETON CHROMATOGRAPHY

INTRODUCTION

Aroclor 5460 is made by chlorinating Santowax R, a mixture of terphenyl isomers. In order for Aroclor 5460 to be used as a hot melt ashesive in food packaging, the level of polychlorinated biphenyls (PCB) in this product must be monitored to know that they are below a given level. PCB's result from residual biphenyl in the Santowax R.

PRINCIPLE

Direct gas chromatography cannot be used to monitor the PCB's because of overlap on the chromatogram between the higher chlorinated biphenyls and the lower chlorinated terphenyls. Therefore, carbon-skeleton chromatography is used, in which a micro catalytic hydrogenation unit is attached to the inlet of the gas chromatograph. All of the chlorines on the terphenyls and biphenyls in the sample are stripped off by this unit and the hydrogenated biphenyl is then separated from the hydrogenated terphenyls by the gas chromatograph.

APPARATUS

1. Perkin -Elmer Model 900 or equivalent programmed temperature gas chromatograph with flame ionization detector.

2. Modified Beroza Carbon Skeleton Determinator (Lab-built from design supplied by Ethyl Corporation).

3. Palladium Chloride Catalyst (Cat. No. 3-8010 Catalyst Charge, $2.50/each, National Instruments Laboratories, Inc., 12300 Parklawn Drive, Rockville, Maryland).

4. 1/8" O.D. x 2 meter column of 10% SE-52 Silicone Gum Rubber on High Performance Chromosorb W, 60-80 mesh.

PROCEDURE

The Beroza Carbon Skeleton Determinator (CSD) is filled with several catalyst charges and attached to the inlet of the chromato-

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graph by inserting the large needle on the end of the CSD through the septum on the chromatograph. The carrier gas line is disconnected from the injection port inlet and rerouted to the front of the CSD. The injection port inlet is capped off. Hydrogen must be used as the carrier gas. Therefore, to provide optimum operation of the flame ionization detector, auxilary helium gas is added to the hydrogen carrier flow going into the detector. A helium flow about equal to the hydrogen carrier flow is suitable.

The CSD temperature is maintained at 280°C by connecting the heater cord to a Variac with temperature readout on a pyrometer connected to the thermocouple. The hydrogen carrier flow rate is 35 ml/min with helium auxilary flow rate of about 35 ml/min. The column is held at 135°C for 2 min. after sample injection and then programmed to 300°C at 10°C/min. The injection port temperature is 315°C and detector 300°C.

The sample is dissolved in cyclohexane to about a 50% solution and 0.5 ul injected into the CSD unit. They hydrogenated biphenyl, terphenyl and quatraphenyl peak areas are normalized assuming equal response for all three. A portion of Aroclor 5460 was purified by preparative gas chromatography to remove all biphenyls. A mixture of this material containing a known amount of Aroclor 1260 was run through this procedure to confirm that the assumption of equal response in the flame ionization detector for all hydrogenated species is valid.

REFERENCES

Literature references to the operation and application of the Beroza Carbon Skeleton Determinator are as follows:

(1) Beroza, M., Anal, Chem. 34, 1801 (1962).

(2) Beroza, M. and Sarmiento, R., Anal. Chem. 35, 1353 (1963).

(3) Beroza, M. and Sarmiento, R., Anal. Chem. 37, 1040 (1965).

APPENDIX

Details of the construction of the modified CSD unit are given in figures 1 and 2. These are copies of two diagrams provided by

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Ethyl Corporation. A sample chromatogram is given in Figure 3.

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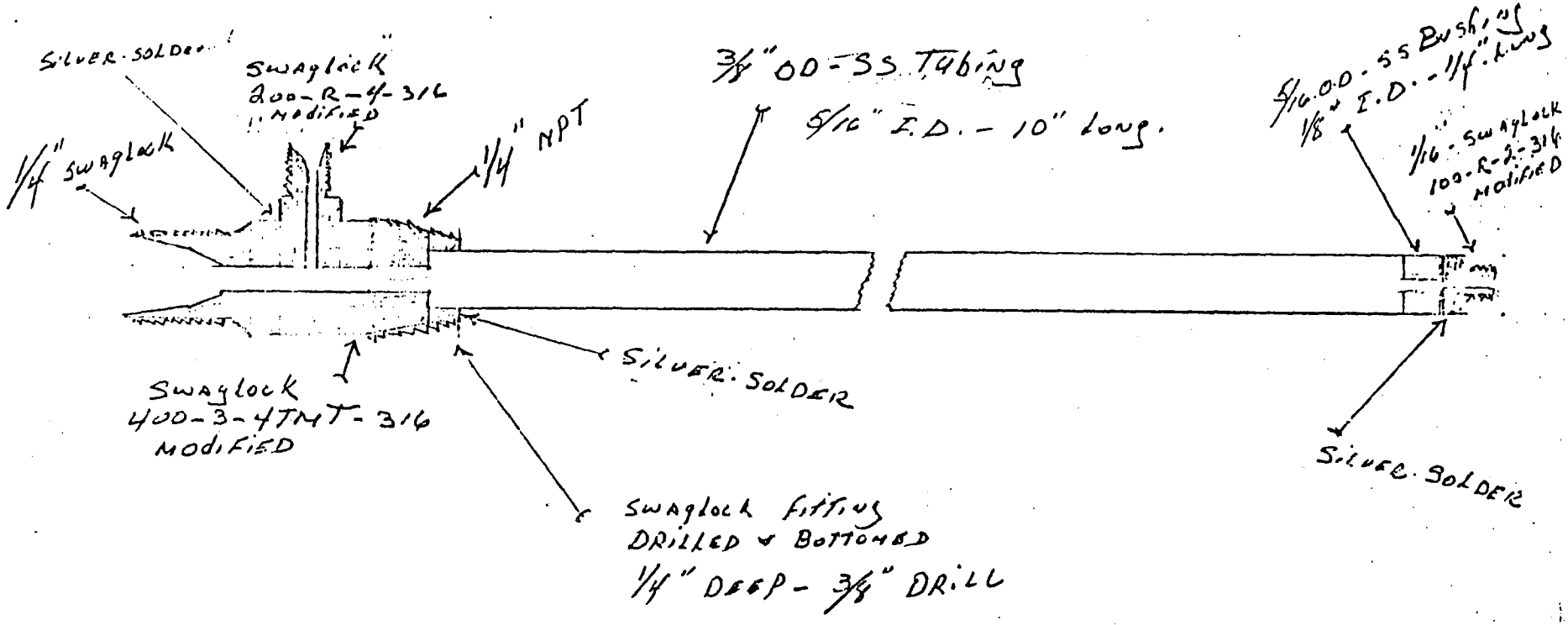
FIGURE 1
CATALYST TUBE



NOTE:- SWAGLOCK FITTING 400-3-4TMT-316, THE BRANCH 1/4" TUBE FITTING WAS CUT OFF AND A 1/4" HOLE 1/4" DEEP DRILLED TO RECEIVE SWAGLOCK FITTING 200-R-4-316, WHICH WAS SILVER-SOLDERED.

NEEDLE:-
#17 SYRINGE NEEDLE - 4" LONG WITH 1/8" SS. SWAGLOCK

NUT:-
1/4" SS SWAGLOCK MODIFIED

SEPTA BACKUP:-
SS - MACHINED TO SWAGLOCK TAPER.

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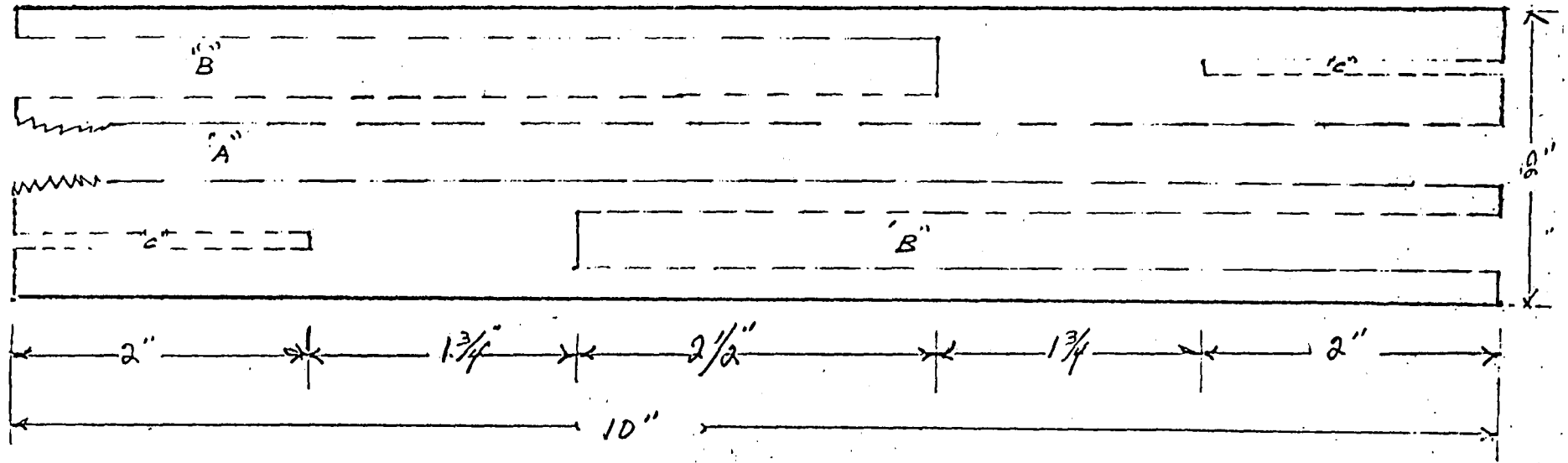
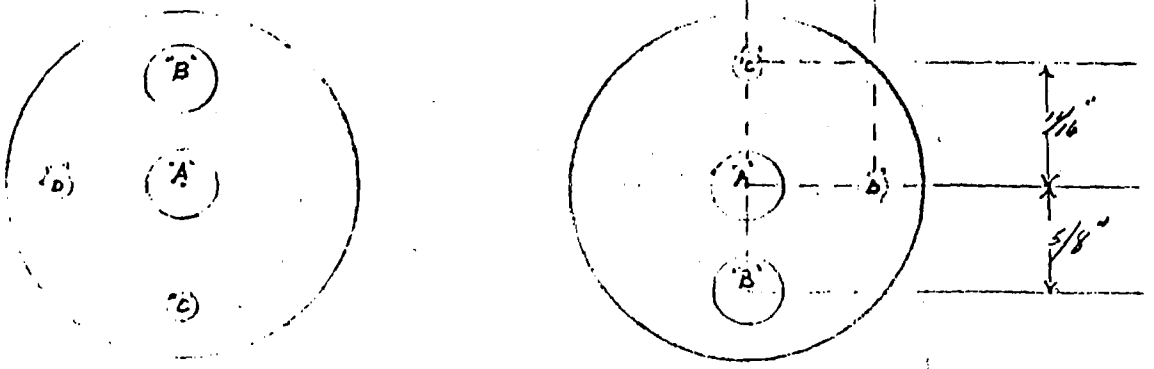
MATERIAL :- 2" ALUMINUM ROUND STOCK
HOLES :- "A" - 3/8" DRILLED & REAMED - THROUGH
" "B" - 3/4" DRILLED - BOTTOMED
" "C" - 1/8" DRILLED — BOTTOMED

HOLES :- "D" - 1/8" DRILLED - THROUGH
THREADS :- DRILL & TAP 1/4 NPT





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HYDROGENATOR HEATER BLOCK

FIGURE 2

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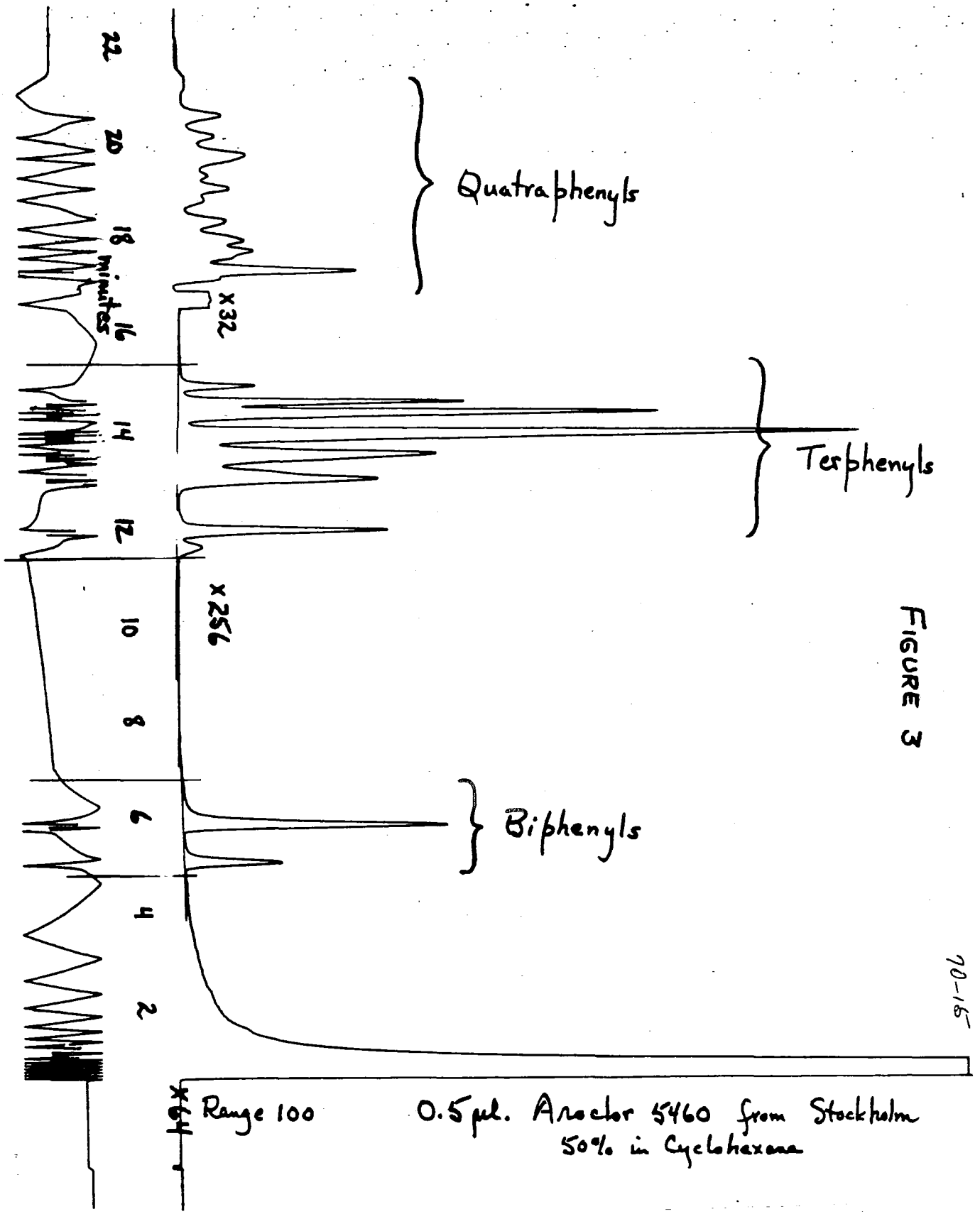

FIGURE 3

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