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AEC RESEARCH AND DEVELOPMENT REPORT

CURIUM PROCESS DEVELOPMENT

III - 5. QUANTITATIVE DETERMINATION OF DIETHYLBENZENE IN AIR

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III-5. Quantitative Determination of Diethylbenzene in Air

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ABSTRACT

A gas chromatographic method was developed to determine the diethylbenzene concentration in the atmosphere of work areas in which this solvent is handled. In this method, an air sample was injected into a gas chromatographic column packed with diatomaceous earth coated with a silicone oil. The diethylbenzene was separated and determined quantitatively with a flame ionization detector. Diethylbenzene concentrations as low as 0.2 ppm were detected.

FOREWORD

This report is one in a series that describes the development of separations processes for purifying ^{244}Cm produced in Savannah River reactors. The series is being issued under the general title *Curium Process Development*. Following the general title, a roman numeral designates the subject area of the report and an arabic numeral designates the series report number in that subject area. A subtitle describes the content of each report. Subject areas foreseen for this series are:

I. General Process Description
II. Chemical Processing Steps
III. Analytical Chemistry Support
IV. Equipment Development and Testing

Reports issued in this series include:

I. *General Process Description* by I. D. Eubanks and G. A. Burney (USAEC Report DP-1009).

II-1. *Separation of Americium from Curium by Precipitation of* $K_3 AmO_2 (Co_3)_2$ by G. A. Burney (USAEC Report DP-1109).

III-1. *Analytical Techniques for Characterizing Solvent* by R. Narvaez (USAEC Report DP-1010).

III-2. *Identification of Solvent Degradation Products* by D. L. West and R. Narvaez (USAEC Report DP-1016).

III-3. *Analytical Control* by E. K. Dukes (USAEC Report DP-1039).

III-4. *Evaluation of Potential Hazards from Chlorination of Amines and Ammonia* by D. L. West, M. L. Hyder, G. A. Burney, and W. E. Prout (USAEC Report DP-1142).

IV. *Equipment Development and Testing* by A. A. Kishbaugh, H. Bull, III, G. W. Gibson, Jr., and L. F. Landon (USAEC Report DP-1146).

INTRODUCTION

Diethylbenzene (DEB, mixed isomers) has been used as the diluent for tertiary amines in the solvent extraction system for processing ^{244}Cm at the Savannah River Laboratory.[1] The solvent extractant is normally contained completely within the process equipment, but feed makeup operations and occasional spills can expose personnel to DEB vapors.

DEB is only a moderate health hazard.[2] Studies with animals indicated that DEB has a low oral toxicity with the LD_{50} value being greater than 1-2 g/kg, and that DEB produces adverse systemic effects only when exposures are prolonged and repeated. Although DEB does not appear to constitute a serious health hazard, it should be handled with caution because its toxicity has not been investigated completely. On the basis of the studies with animals, workmen should not be exposed repeatedly to DEB concentrations in air greater than 100 ppm, and the concentration should not exceed 50 ppm if such exposure is to be prolonged.

A gas chromatographic method for the quantitative determination of DEB in air is described in this report.

PROCEDURE

In studies to develop a method for analyzing DEB vapors in air, samples were collected in 0.5-liter glass bulbs, which were first purged for several minutes with a small air pump. These bulbs were previously coated with "Desicote"* to minimize adsorption of DEB to the wall; erratic results were obtained with uncoated bulbs.

A Perkin-Elmer Model 811 dual column gas chromatograph was used for the analysis with two 6-ft-long by 1/8-in-ID glass columns, packed with diatomaceous earth (120-140 mesh, "Gas Chrom Z"**) and coated with 5 wt % silicone gum rubber (General Electric SE-30).

To demonstrate the method, a 10 cm^3 aliquot of a collected air sample was injected into the gas chromatograph with a gas syringe. The sample port and detector block temperatures were maintained at 200°C. The column temperature, initially at 125°C, was programmed to increase to 175°C at 16°C/min after sample injection. The carrier gas was helium. Under these conditions DEB was separated from air and was detected quantitatively with the flame ionization detector. A chromatogram of a sample containing 3 ppm DEB in air is shown in Figure 1.

Unknown samples were quantitatively analyzed by comparing the DEB peak area to a calibration curve of standards. The minimum concentration of DEB in air which was detected by this method was ∿0.2 ppm. The analytical precision near this concentration was ∿±100%; the precision in the 0.5 to 100 ppm range was ∿±15%. This precision was attained in the absence of other interfering organic compounds.

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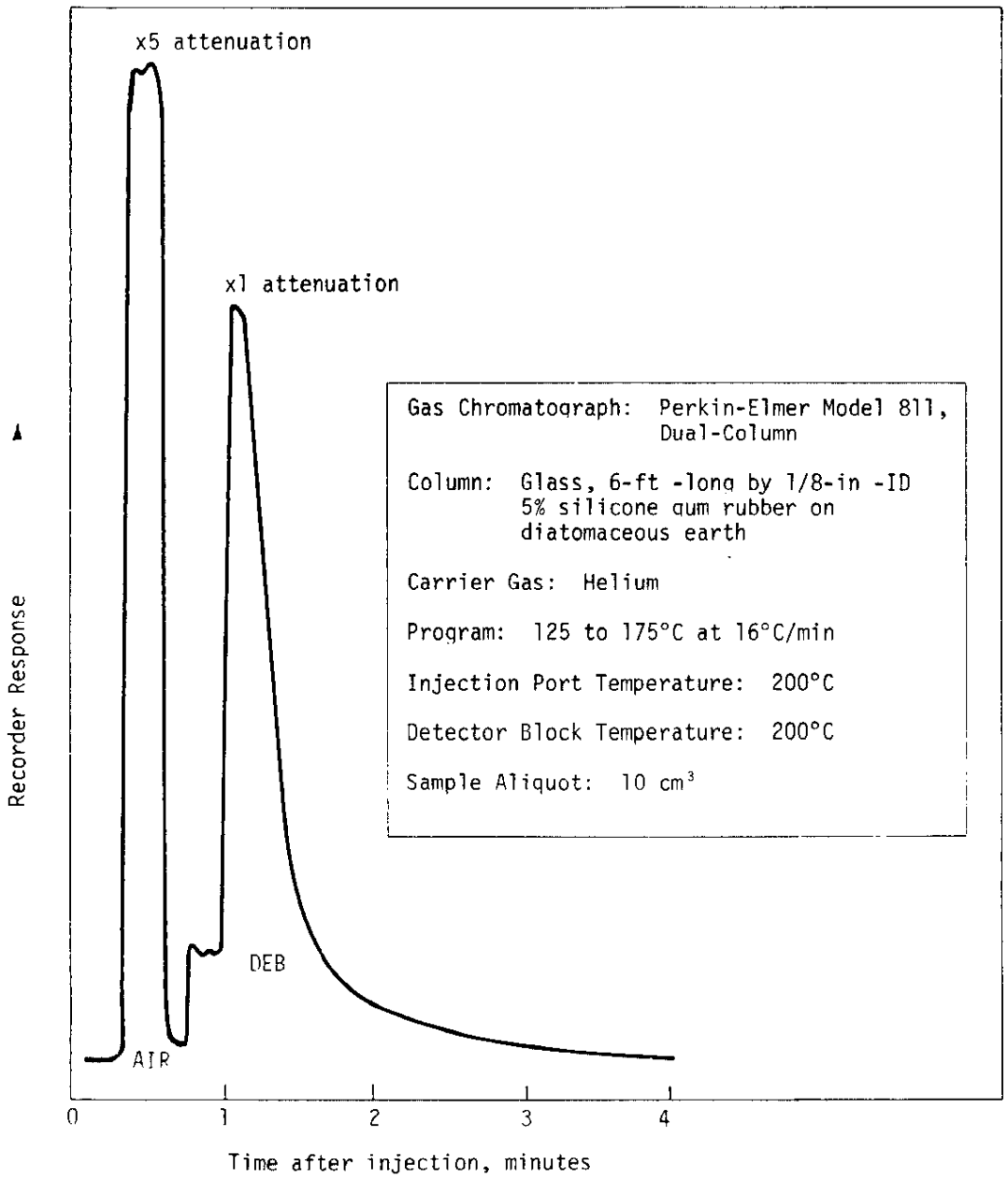

FIG. 1 GAS CHROMATOGRAM OF 3 ppm DIETHYLBENZENE IN AIR

REFERENCES

1. I. D. Eubanks and G. A. Burney. *Curium Process Development - I. General Process Description.* USAEC Report DP-1009, E. I. du Pont de Nemours & Co., Savannah River Laboratory, Aiken, S. C. (1966).

2. *Toxicological Properties of Diethylbenzene.* Bulletin issued by the Plastics Department of the Dow Chemical Company, Midland, Michigan.