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GAS CHROMATOGRAPHIC STUDIES OF SULPHUR CONTAINING COMPONENTS
OF ATHABASCA BITUMEN DISTILLING AT 318°C

by

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INTRODUCTION

This is a preliminary report of the work on the fraction distilling at 318°C.

The procedure is similar to work described in the two previous reports, but the column was changed in such a way as to eliminate the occurrence of carrier gas bubbles forming in the column packing. Also a cut from this fraction was obtained by redistillation.

Experimental

This sample was obtained from the batch of molecular distilled pentane extract of bituminous sand and distilled in the spinning annular still at $42\ \mu$ over a temperature range $81\ 1/4 - 83\ 1/4^\circ\text{C}$. A portion of this was redistilled at $17\ 1/2\ \mu$ at $71\ 1/2^\circ\text{C}$ and this redistilled material was chromatographed as described in this report. The sulphur content was 2.08%.

The preparative gas chromatographic column for this work consisted of 20 ft x $1/2$ inch OD glass tubing packed with 20% Hyprose [octakis (2 - hydroxypropyl) sucrose] on acid washed, 60-80 mesh Chromosorb W. Both inlet + outlet of chromatograph were kept at 210°C . The Helium carrier gas had a flow rate of 380 ml/min. At the outlet $1/30$ of the flow was diverted to the detector. The sample size was $10\ \mu\text{l}$ and 5 runs were made. The sample was injected at 70°C and then increased at $1^\circ/\text{min}$ to 190°C and held at this temperature. In this column the carrier gas always flowed downward through the column packing. Samples were collected on 50 mg of packing of 10% carbowax 20 M on Chromosorb W. This was one fifth of the amounts that were used in traps for previous runs.

Discussion and Results

The chromatogram showing the Melpar sulphur trace that was obtained with the preparative column is shown in the figure. Fractions were trapped out as indicated. The resolution obtained here is better than that obtained before on any preparative column. With the carrier gas

flowing downward through the column packings there was no change of any bubbles of carrier gas developing in the packing.

Characterization of the sulphur compounds in the fractions that were collected will be described in a later report.

