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GAS CHROMATOGRAPHIC STUDIES OF SULPHUR CONTAINING COMPONENTS OF ATHABASCA BITUMEN DISTILLING AT 300°C

by

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INTRODUCTION

This is a preliminary report of the work on the fraction distilling at 300°C and is a continuation of the work on sulphur containing components in the Athabasca bitumen. In the initial report of the work that had been done on the fraction distilling at 333°C was described. The second band of gas chromatographic peaks which was fairly well resolved had been investigated but nothing had been done on the poorly resolved first band. In this work much better resolution was obtained. Also, this material had recently been obrained from bituminous sands and had undergone no previous fractionation other than distillation.

Experimental

A pentane extract that had been obtained by extraction of bituminous sand was distilled in a molecular still at 100°C and at a pressure range of 22-80 μ .

During distillation in a spinning annular still the fraction discussed here was obtained at a temperature of 75-75 7/8°C at a pressure of 67 μ . In both these stills the material was in contact only with glass and teflon. A microcoulometric determination revealed this fraction to contain 1.68% sulphur.

Two gas chromatographic columns were used; one analytical and the other preparative.

The analytical column consisted of a 20 ft x 1/8 inch O.D. glass column packed with 12% Hyprose [octakis (hydroxypropyl) sucrose] on acid washed, 60-80 mesh Chromosorb W. Helium was the carrier gas and its flow was 60 mls/min. The detector and inlet temperature were both 200°C. The 0.5 μl samples were injected at ambient temperature. The column was held at ambient temperature for 20 minutes and then increased at 0.8°C/min.

The preparative column consisted of 20 ft x 1/2 inch O.D. glass packed with 12% Hyprose on acid washed, 60-80 mesh Chromosorb W. Also an after column of 2 1/2 ft x 1/2 inch O.D. glass tubing packed with

10% carbowax 20M on Chromosorb W was added. The carrier gas was nitrogen and its flow rate was 330 mls/min. The carrier gas was split at the outlet and 1/30 was passed to the Melpar detector. Both inlet and outlet temperatures were 210°C. The 10 μl samples were injected at a column temperature of 75°C and then the temperature was increased at 10/min

The fractions were collected as reported in the report dealing with the fraction distilling at 333°C.

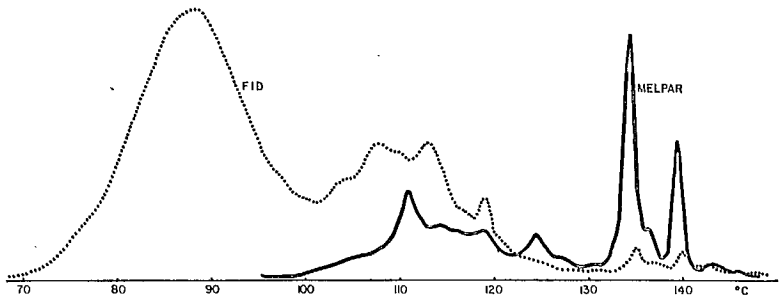
DISCUSSION AND RESULTS

The chromatograms obtained using the columns are shown in figures 1 and 2.

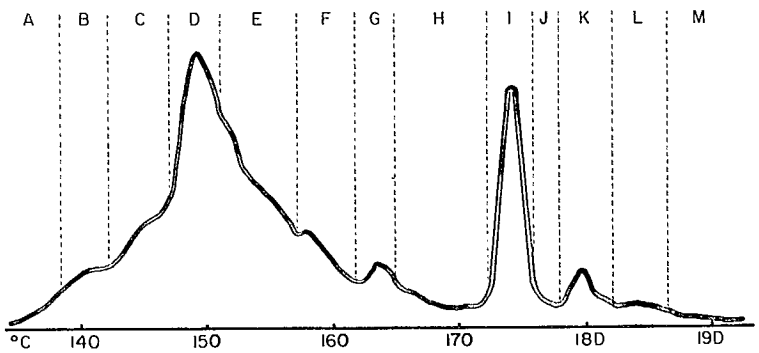
Figure 1 shows both the Melpar sulphur and flame ionization traces that were obtained with the use of the analytical column. Figure 2 shows the Melpar sulphur trace obtained with the use of the preparative column. The fractions obtained during use of the preparative column as shown in Figure 2 were analyzed for sulphur and percentages of sulphur in the original sample are as follows:

A	1.87	H	2.29
B	4.57	I	4.88
C	2.41	J	1.93
D	14.56	K	3.75
E	8.72	L	1.12
F	3.64	M	~~interminable~~ indeterminable
G	2.07		

Further characterization of these fractions will be described in a later report.



CHROMATOGRAPHIC SEPARATION ON ANALYTICAL HYPROSE COLUMN



CHROMATOGRAPHIC SEPARATION ON PREPARATIVE HYPROSE COLUMN