I50 NOTES

Quantitative separation of cymene isomers by gas chromatography

Separation of o-, m- and p-xylenes has been reported¹; also 2,6- and 2,7-dimethylnaphthalenes have been separated², using a Bentone-SE 52 column. During the course of an investigation in this laboratory it became necessary not only to detect the three isomers of cymene (isopropyltoluene), but also to analyse them quantitatively. The most closely related work published to date is that of VAN DER STRICHT AND VAN RYSSELBERGE³, who by using a Bentone-34 column were able to separate the three isomers, but not sufficiently for quantitative work.

The work of the above-mentioned workers was taken as a starting point, and columns (6 m × 5 mm) packed with Bentone-34, SE-30, and didecyl phthalate (DDP) and mixtures thereof on 60/80 chromosorb W were tried. A PE-116 apparatus* (Thermister detector) was used, and the method of preparing the packing was similar to that of Mortimer and Gent¹. Columns with the following different stationary phases were tried:

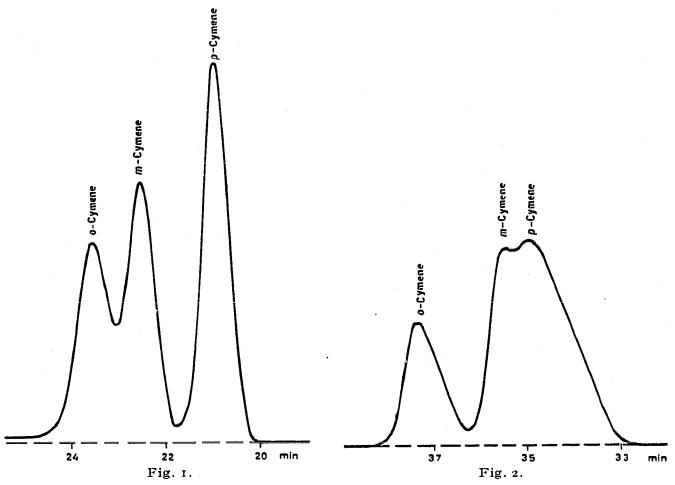


Fig. 1. Column packing: 5% SE 30-15% Bentone-34; column temperature: 161°; carrier gas: hydrogen; inlet pressure: 2.5 kg/sq.cm; sample size: 4 μ l.

Fig. 2. Column packing: 10% DDP-20% Bentone-34; column temperature: 144°; carrier gas: hydrogen; inlet pressure: 2.5 kg/sq.cm; sample size: 4 μ l.

^{*} Perkin Elmer Fractometer Model 116.

- (1) 20 % SE 30.
- (2) 5 % SE 30-15 % Bentone-34.
- (3) 15 % DDP- 5 % Bentone-34.
- (4) 10 % DDP-15 % Bentone-34.
- (5) 10 % DDP-20 % Bentone-34.

Results obtained with columns 2 and 5 were best. As can be seen from Fig. 1, stationary phase 2 gives a satisfactory separation of the *meta* and *para* isomers, but there is tailing between *ortho* and *meta*. Fig. 2 where column 5 is used shows that the tailing between *ortho*- and *meta*-cymene has been eliminated, but there is a poor separation of *para* from *meta*. To overcome this difficulty the two columns (total length 12 m) were used in series with each other, and the chromatogram obtained is shown in Fig. 3.

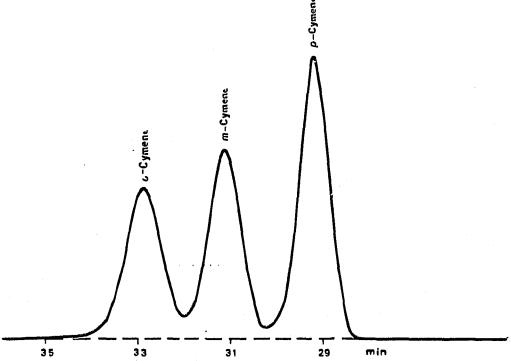


Fig. 3. Column packing: (I) 5% SE 30-15% Bentone-34; (II) 10% DDP-20% Bentone-34 column temperature: 155°; carrier gas: hydrogen; inlet pressure: 2.5 kg/sq.cm; sample size: 6 µl

In addition a single column was tried combining the three stationary phases as follows: 5 % SE 30-10 % DDP-20 % Bentone-34. The separation was not as good as the one obtained by using the two columns in series, possibly because of the shorter length of this column.

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¹ J. V. Mortimer and P. L. Gent, Nature, 197 (1963) 789.

² Aerograph Res. Notes, Summer Issue, 1963.

³ M. VAN DER STRICHT AND J. VAN RYSSELBERGE, J. Gas Chromatog., 1, Aug. No. (1963) 29.