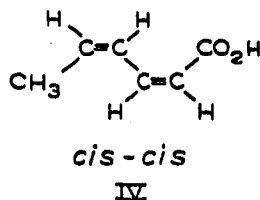
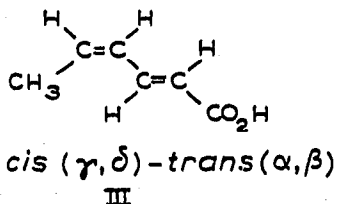
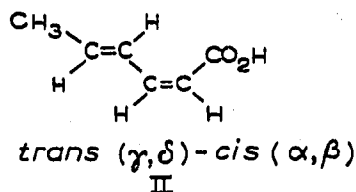
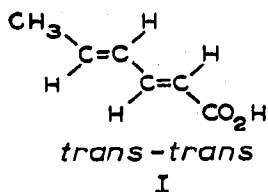


CHROM. 5470

## Determination of the isomers of sorbic acid by gas chromatography

Sorbic acid (2,4-hexadienoic acid) can exist in four possible steric configurations. The commercially available acid, which is used as a food preservative, has the *trans-trans* configuration (I). Several workers<sup>1-4</sup> have developed spectrophoto-



metric methods of determining sorbic acid content in foods. FLOYD<sup>5</sup>, using UV spectrophotometry, determined 0.005% sorbic acid with an accuracy of 0.4%. Gas-liquid chromatography<sup>6-8</sup> and paper chromatography<sup>9</sup> have also been successful in isolating and determining sorbic acid content. However, methods have not been described for the determination of the individual isomers of sorbic acid.

A gas chromatographic method has been developed for the quantitative determination of the sorbic acid isomers. The procedure involves the formation of the volatile trimethylsilyl (TMS) esters of the acids.

### Materials and methods

The gas chromatograph that was used was a Varian Aerograph Model 1860 with thermal conductivity detectors. The recorder was an Aerograph Model 20. The mass spectra were obtained on a Consolidated Electrodynamics Corporation Model 21-104 rapid scanning mass spectrometer. The gas chromatograph-mass spectrometer (GC-MS) coupling was carried out using the metering valve technique<sup>10</sup>.

**Column and conditions.** The chromatographic column was 1/4 in. O.D. by 9 ft. aluminum tubing containing 16% OV-210 on acid-washed, DMCS-treated, 45-60 mesh Chromosorb W. Before use, the column was conditioned overnight at 230°.

The injection port and detector temperatures were 230°. The oven temperature was 150° and was programmed, after 7.5 min, to 230° at a rate of 10°/min. The helium flow rate was 40 ml/min.

**Silylation procedure.** Weigh 100 mg of sample and 15 mg of sorbitol (internal standard) into a reaction vial. Add 0.5 ml of pyridine and heat until complete dissolution. Add 1 ml of hexamethyldisilazane (HMDS) and 0.5 ml of trimethylchlorosi-

lane (TMCS). Allow to react with stirring for 10 min at room temperature. Heat for 10 min on steam bath. Centrifuge and inject 8  $\mu$ l into gas chromatograph.

### Results and discussion

A typical gas chromatogram obtained for a mixture of the TMS derivatives of the isomers of sorbic acid is shown in Fig. 1. The reagents used for the silylation procedure were completely eluted from the gas chromatograph after 3 min. Next followed three peaks which, by GC-MS, NMR and IR, were identified as the *cis* ( $\gamma,\delta$ )-*trans* ( $\alpha,\beta$ ), *trans* ( $\gamma,\delta$ )-*cis* ( $\alpha,\beta$ ) and *trans-trans* isomers of sorbic acid-TMS. The TMS derivative of the internal standard, sorbitol, eluted after 22 min.

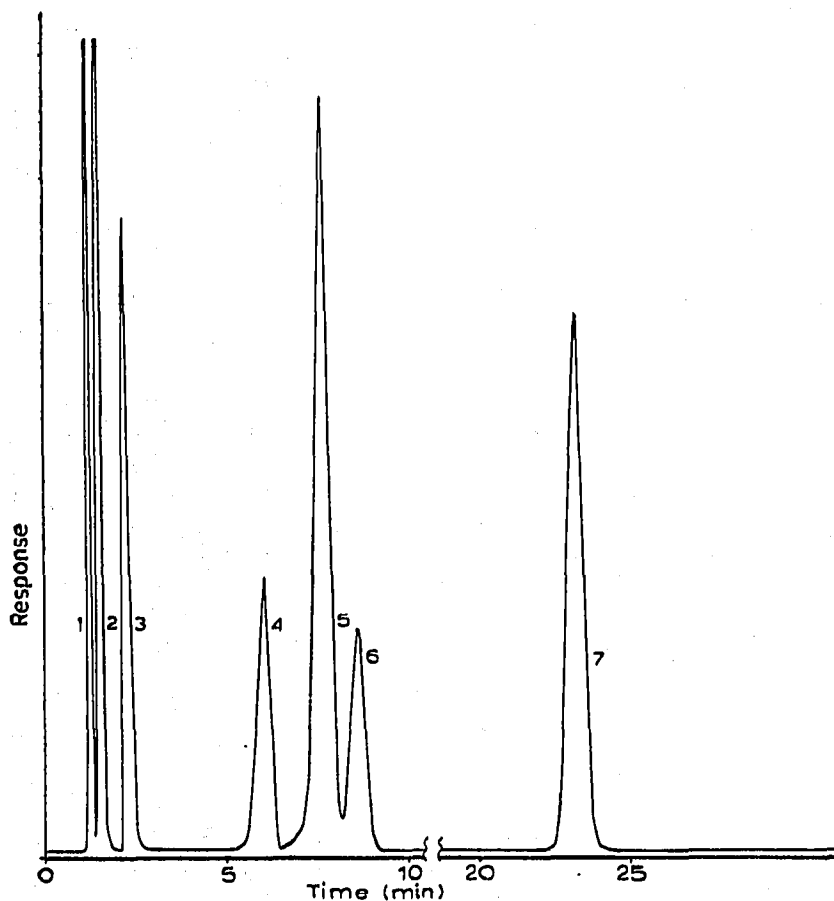


Fig. 1. GLC separation of the TMS derivatives of the isomers of sorbic acid. (1) TMCS, (2) HMDS, (3) pyridine, (4) *trans* ( $\gamma,\delta$ )-*cis* ( $\alpha,\beta$ ) sorbic acid, (5) *cis* ( $\gamma,\delta$ )-*trans* ( $\alpha,\beta$ ) sorbic acid, (6) *trans-trans* sorbic acid, (7) sorbitol.

A response factor\* of 1.13, with a relative standard derivation of 1.8%, was found for a sorbic acid (*trans-trans*) to sorbitol ratio of 1:1. The response and the precision were found to be independent of the concentration ratio. However, it is expected

$$* \text{ Response factor} = \frac{\text{area sorbitol/g sorbitol}}{\text{area sorbic acid (trans-trans)/g sorbic acid}}$$

ted that the precision of the method will be poorer when a sample contains the four sorbic acid isomers. That would be due to the imperfect separation of the isomers.

*Research Department, Chemcell Limited,  
P.O. Box 99, Edmonton, Alberta (Canada)*

J. H. VERMEER  
J. R. DEAN

- 1 D. MELNICK AND F. H. LUCKMANN, *Food Res.*, 20 (1955) 649.
- 2 D. MELNICK AND F. H. LUCKMANN, *Food Res.*, 19 (1954) 20.
- 3 G. ALDERTON AND J. C. LEWIS, *Food Res.*, 23 (1958) 338.
- 4 P. SPANYAR AND A. SANDOR, *Z. Lebensm.-Untersuch.-Forsch.*, 108 (1958) 402.
- 5 K. M. FLOYD, *J. Ass. Offic. Anal. Chem.*, 50 (1967) 1123.
- 6 J. VOGEL AND J. DESHUSSES, *Anal. Abstr.*, 13 (1966) 2669.
- 7 T. NISHIMOTO AND M. UYETA, *Anal. Abstr.*, 13 (1966) 409.
- 8 J. PL. GODDIJN, M. VAN PRAAG AND H. J. HARDON, *Anal. Abstr.*, 12 (1965) 1418.
- 9 T. HOYEM, *J. Ass. Offic. Anal. Chem.*, 45 (1962) 902.
- 10 R. S. GOHLKE, *Anal. Chem.*, 31 (1959) 539.

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