advantage in analyses of samples with a low fural content, such as is the case with various food materials.

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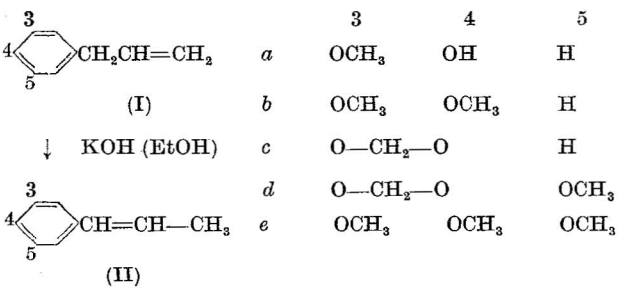
[1] Dunlop, A. P., and Peters, N. H., *The Furans* (Reinhold, New York, 1953).
[2] Stillings, R. A., and Browning, B. L., *Indust. Eng. Chem. Anal. Ed.*, **12**, 499 (1940).
[3] Jolles, A., *Z. Anal. Chem.*, **45**, 196 (1906).
[4] Maljugina, N. J., and Koršinov, J. A., *Zurnal. anal. Chimii*, **2**, 341 (1947).
[5] Davídek, J., *Nature*, **190**, 1003 (1961).
[6] Davídek, J., *Z. anal. Chem.*, **179**, 165 (1961).

Composition of the Myristicin Fraction from Oil of Nutmeg

THE aromatic ether fraction of oil of nutmeg has been previously shown[1] to consist of eugenol (I*a*), isoeugenol (II*a*), safrole (I*c*) and myristicin (I*d*). Vacuum distillation yields a fraction (b.p. 109°–112°/1 mm mercury; 60 g from 1 kg of 'W.I.' oil of nutmeg (George Lueders and Co.)) which consisted of a substance heretofore accepted both chemically[1,2] and pharmacologically[3] as the single compound, myristicin (I*d*).



The isomerization of this fraction with alcoholic potassium hydroxide yielded (*trans*) isomyristicin (II*d*), isolated by crystallization of the distilled reaction mixture. The mother liquors of this isolation, on analysis by vapour phase chromatography, provided the first indication of the complexity of the above 'myristicin' fraction. Of the four peaks observed (Fig. 1), No. 1 was easily identified as methyl isoeugenol (II*b*) by its infra-red spectrum and direct comparison to a commercial sample. Peak No. 3 was to a large extent *trans*-isomyristicin not removed by crystallization. The remaining two peaks were isolated using a Beckman Megachrome preparative V.P.C. instrument using a substrate of silicone 710, on firebrick at 220°. The presence of a methylenedioxy group (by nuclear magnetic resonance) and the absence of absorption in the 963–967 cm^{-1} region of the infra-red spectrum of peak 2 strongly suggested that the isomer might be *cis*-isomyristicin. That *trans*-propenyl aromatic ethers possess a characteristic absorption band in the above region (which is transparent for the *cis*-isomer) has been shown for isosafrole[4], anethol[5], isoeugenol, methylisoeugenol[6] and asarone[7]. Further, it has been shown recently[7,8] that the *cis*-isomer of the stereo-isomeric pairs invariably precedes the *trans*-isomer during vapour chromatography. This peak was verified as *cis*-isomyristicin by its conversion to *trans*-isomyristicin (II*d*) and its synthesis from myristicin, both with alcoholic potassium hydroxide.

The fourth peak was also isolated by preparative chromatography and showed the absence of a methylenedioxy group, but the presence of a strong band at 957 cm^{-1}. Isoelemicin (II*e*) was synthesized by the potassium hydroxide isomerization of elemicin[9] and was found to possess an identical infra-red spectrum.

Methylisoeugenol may be assumed to be in the propenyl form in the original sample of nutmeg distillate. There is an unresolved peak at this identical time in the original

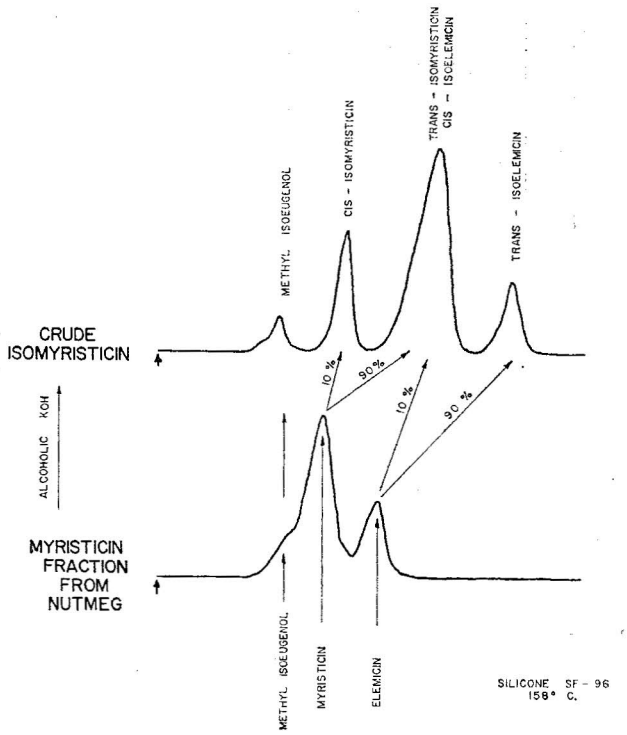


Fig. 1

'myristicin' fraction, and a search for the logical precursor, methyl eugenol, has been unsuccessful[1].

The implied presence of elemicin (I*e*) in the original myristicin fraction was confirmed by the successful separation of it from myristicin by the low-temperature chromatography (158°, silicone '*SF*–96') as shown in the lower part of Fig. 1. Repeated fractional distillation was ineffective in increasing the myristicin content of this constant boiling fraction over 70 per cent. Consequently, in assigning chemical and biological properties to the substance as isolated from nutmeg allowance must be made for this congeneric contaminant.

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[1] Power, F. B., and Salway, A. H., *J. Chem. Soc.*, **91**, 2037 (1907).
[2] Salway, A. H., *J. Chem. Soc.*, 1204 (1909). Trikojus, V. M., and White, D. E., *ibid.*, 436 (1949).
[3] Truitt, jun., E. B., Calloway, III., E., Braude, M. C., and Krantz, jun., J. C., *J. Neuropsychiat.*, **2**, 205 (1961).
[4] Naves, Y. R., and Ardisio, P., *Bull. Soc. Chim. France*, 1053 (1957).
[5] Naves, Y. R., Ardisio, P., and Favre, C., *Bull. Soc. Chim. France*, 566 (1958).
[6] Naves, Y. R., and Grampoloff, A. V., *Bull. Soc. Chim. France*, 1233 (1959).
[7] Baxter, R. M., Fan, M. C., and Kandel, S. I., *Canad. J. Chem.*, **40**, 154 (1962).
[8] Stahl, E., and Trennheuser, L., *Arch. Pharm.*, **293/65**, 826 (1960).
[9] Hahn, G., and Wassmuth, H., *Ber.*, **67**, 696 (1934). Pearl, I. A., *J. Amer. Chem. Soc.*, **70**, 1747 (1948).

Synthesis of some 3-Deoxy-heptulosonic Acids (3-Deoxy-2-keto-heptonic Acids)

A SUITABLE synthesis for 3-deoxy-aldulosonic acids has been published by Kuhn *et al.*[1]. After successfully repeating this work, resulting in the isolation of crystalline 3-deoxy D- and L-*erythro*-hexulosonic acid[2], we investigated the synthesis of some 3-deoxy-heptulosonic acids.

Starting from D-glucose, we followed the procedure as described for the hexulosonic acids (Fig. 1).

By this method the compounds II and III were obtained, both characterized by their spectra. However, we were