Synthesis of a Unique Fatty Acid From *Exocarpus* Seed Oil[1]

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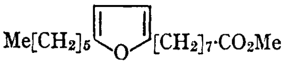
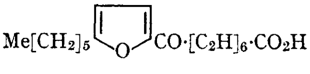
IN a recent communication the isolation of 8-(5-hexyl-2-furyl)octanoic acid from *Exocarpus cupressiformiss* Labill. seed oil was reported.[2] This natural product was characterised as its methyl ester (I), and we now report a six-step synthesis of this ester from 2-furoic acid in high overall yield.



(I)



(II)

Reaction of 2-furoyl chloride with dipentylcadmium afforded 2-hexanoylfuran as a colourless liquid, b.p. 65—67°/0·5 mm., ν_{max} (film) *inter al.*, 1680 (C=O) and 1018 cm.$^{-1}$ (ring breathing). Reduction (Huang-Minlon) gave 2-hexylfuran as a colourless liquid, b.p. 62°/0·5 mm., τ (60 Mc./sec.; CCl_4) 2·65 (1H), 3·70 (1H), 4·02 (1H), 7·35 (2H), and *ca.* 8·1—9·3 (11H).

Acylation of 2-hexylfuran at room temperature with suberic anhydride and boron trifluoride diethyl etherate in benzene gave the keto-acid (II). The n.m.r. spectrum (60 Mc./sec.; CCl_4) of (II) showed a normal AB pattern centred at τ 2·85 (1H) and 3·78 (1H), and in the i.r. spectrum (film) carbonyl stretching absorptions at 1708 (carboxylic acid) and 1675 cm.$^{-1}$ (ketone).

Reduction (Huang-Minlon) of (II) gave the required carboxylic acid which was converted into its methyl ester (I) with ethereal diazomethane. Chromatography of the crude product over Mallinckrodt silicic acid with 10% acetone in pentane as eluent, followed by short-path distillation gave pure (I) as a colourless oil, b.p. 174—176°/0·5 mm. (bath) ν_{max} (CCl_4) *inter al.*, 3105 (ring CH str.), 1743

(C=O str.), 1570 (ring str.) and 1015 cm.$^{-1}$ (ring breathing), λ_{max} (EtOH) 222 mμ (10,200). The n.m.r. spectrum (100 Mc./sec.; CCl_4) exhibited a singlet at τ 4·28 (2H) due to the β-furanoid protons, a singlet at 6·40 (3H) assigned to the methoxy-protons, a triplet centred at 7·44 (4H) due to the methylene protons adjacent to the ring, another triplet centred at 7·75 (2H) due to the methylene protons adjacent to the methoxycarbonyl group, a complex signal at 8·30—8·70 (20H) due to the remaining methylene protons and a triplet centred at 9·05 (3H) due to the methyl group. These properties are in agreement with those reported.[2] The mass spectrum of synthetic (I), obtained by the direct insertion technique using an A.E.I. MS9 instrument was virtually identical to that of the natural material.[2]

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[1] For previous Paper, see: R. Grigg, J. A. Knight, and M. V. Sargent, *J. Chem. Soc.* (*C*), 1966, 976.

[2] L. J. Morris, M. O. Marshall, and W. Kelly, *Tetrahedron Letters*, 1966, 4249.