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# SYNTHESIS OF *cis*-10-HYDROXYDEC-2-ENOIC ACID

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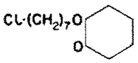
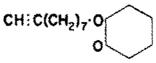
RECENTLY the synthesis of *trans*-10-hydroxydec-2-enoic acid, identical with the acid isolated from the 'royal jelly' of bees by Townsend and Lucas[1] was reported from this Laboratory.[2] We now describe the synthesis of the stereoisomer, *cis*-10-hydroxydec-2-enoic acid[3].

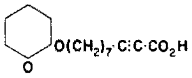
The starting material was pimelic acid, which by esterification, followed by reduction with lithium aluminium hydride, gave heptan-1,7-diol. The diol was converted into the 1,7-chlorohydrin by means of hydrochloric acid and cuprous chloride[4] and the hydroxyl group was then protected by formation of the tetrahydropyranyl ether (I) b.p. 92-95°/0.2 mm. (Found: Cl, 14.9. $C_{12}H_{23}O_2Cl$ requires Cl 15.1%).



I



II



III

$HO(CH_2)_7\cdot CH{:}CH\cdot CO_2H$

IV

Treatment of I with sodium iodide in acetone, followed by sodium acetylide in dimethyl formamide gave 9-2'-tetrahydropyranyloxynon-1-yne (II) b.p. 90-91°/0.3 mm. (Found: C,75.1; H, 10.7. $C_{14}H_{24}O_2$ requires

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[1] G. F. Townsend and C. C. Lucas, *Biochem. J.* **34**, 1155 (1940)

[2] G. I. Fray, R. H. Jaeger and Sir Robert Robinson, *Tetrahedron Letters*, No. 4, 15 (1960).

[3] U.K. Patent Application No. 8576/60.

[4] W. R. Coleman and W. G. Bywater, *J. Amer. Chem. Soc.* **66**, 1821 (1944).

C,75.0; H, 10.8%). Reaction of this acetylene with ethylmagnesium bromide under the special conditions described by Wotiz *et al*[5], followed by treatment with solid carbon dioxide, afforded the tetrahydropyranyloxy-acid (III) which was hydrolysed to 10-hydroxydec-2-ynoic acid, m.p. 72.0-73.0° (Found: C, 65.4; H, 8.7. $C_{10}H_{16}O_3$ requires C, 65.2; H, 8.8%). Its infra-red spectrum showed strong bands at 3.06 (O-H), 4.45 (C≡C), 6.02 (C=O) and 9.50μ (C-OH). Hydrogenation of this acetylenic acid over Lindlar's catalyst[6] gave a virtually quantitative yield of *cis*-10-hydroxydec-2-enoic acid (IV) m.p. 73.5-74.5° (Found: C, 64.4; H, 9.6. $C_{10}H_{18}O_3$ requires C, 64.5; H, 9.7%). The compound had an ultra-violet maximum at 2100Å, ε 12,450 and infra-red absorption bands at 2.90 (O-H), 5.92 (C=O), 6.15 (*cis* C=C), 9.52 (C-OH) and 12.3μ (*cis* CH=CH).

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[5] J. H. Wotiz, C. A. Hollingsworth and R. E. Dessy, *J. Amer. Chem. Soc.* **78**, 1221 (1956)

[6] H. Lindlar, *Helv. Chim. Acta*, **35**, 446 (1952).