

pound are in the  $\beta$ -position and their experimental conditions are not favourable to such an isomerisation.

During halogenation and condensation reactions only the carbon atom in position 3 of *trans*- $\beta$ -decalone is found to be reactive.<sup>5</sup> *Trans*- $\beta$ -decalone yields on oxidation with nitric acid *cyclohexane*-1:2-diacetic acid along with a small quantity of the 1-carboxy-2-propionic acid, indicating that the carbon atom in position 1 is also affected. Hence, a careful search is being made for the 1:2-diketodecalin (IV) in the products of oxidation of *trans*- $\beta$ -decalone with selenium dioxide.

Full details will be published elsewhere.

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<sup>1</sup> *Annalen*, 1924, **437**, 163.

<sup>2</sup> *J. Annamalai Univ.*, 1937, **7**, 23.

<sup>3</sup> Lehmann and Kratschell, *Ber.*, 1934, **67**, 1867.

<sup>4</sup> — —, *ibid.*, 1935, **68**, 360.

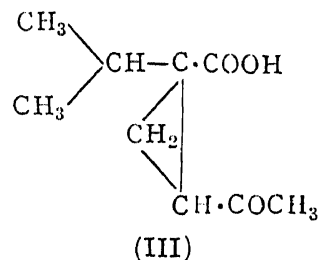
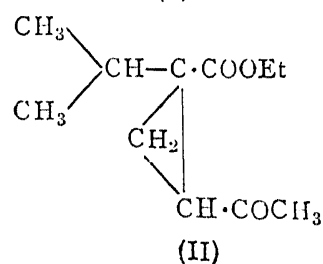
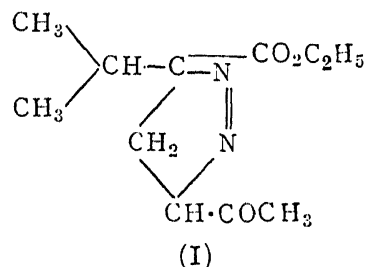
<sup>5</sup> Feu, Mc Quillon and Robinson, *J.C.S.*, 1937, **53** ;  
*cf.* Cook and Lawrence, *J.C.S.*, 1937, 877.

### Synthesis of Umbellulonic Acid.

UMBELLULONIC acid (III) is a direct oxidation product of the naturally occurring bicyclic ketone umbellulone. It has now been synthesised as follows :

Ethyl *isopropyl* acrylate has been condensed with diazoacetone to yield a pyrazoline compound, b.p. 130–35°/3 mm. (Found : N, 11.68 per cent. ; calc., 12.39 per cent.) The pyrazoline compound (I) splits off nitrogen at 180° and the resulting nitrogen-free ester (II) distils at 135–45°/25 mm. 233–35°/685 mm. ; (Found : C, 66.95 per cent. ; H, 9.23 per cent. ; calc. : C, 66.67 ; H, 9.09 per cent.) and has yielded umbellulonic acid (III) on hydrolysis. The acid b.p. 190–92°/50 mm. (the b.p. agreeing with that of umbellulonic acid obtained from umbellulone by oxidation), however, does not crystallise, but forms an oxime, m.p. 145–46° (Found : N, 7.64 per cent. ; equiv., 182) and a semicarbazone, m.p. 170° (Found : N, 18.30 per cent. ; equiv., 228.6.) The combustion values and the equivalents

of the oxime and the semicarbazone are in agreement with those of the corresponding derivatives of umbellulonic acid. It is rather peculiar that to the oxime of umbellulonic acid Tutin<sup>1</sup> has attributed the m.p. 169–70°.



The *cis*-configuration of umbellulonic acid now synthesised has been proved by oxidising it to *cis*-umbellularic acid monohydrate, m.p. 95°, and the anhydrous acid, m. p. 124–25°. The mixed melting points of the monohydrate and the anhydrous acid, when taken with the corresponding samples synthesised in this laboratory,<sup>2</sup> remained undepressed.

The resolution of the synthetic variety of the umbellulonic acid could not be effected by its brucine salt on account of the latter not coming in crystalline form due perhaps to the presence of the keto-group.<sup>3</sup>

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<sup>1</sup> *J.C.S.*, 1904, **83**, 645.

<sup>2</sup> Ranganathan, *J. Ind. Chem. Soc.*, 1936, **13**, 419.

<sup>3</sup> *Cf.* Owen and Simonsen, *J.C.S.*, 1933, 1223.