

Temp.° C.	E. M. F. millivolts.
100	5.1
200	10.8
300	17.2
400	23.7
500	30.6

Thermocouples with Nickel generally (in combination with Nichrome, Iron and Copper) give irregular thermo-e.m.f. curves over the temperature range 250°–400° C. owing to some molecular transformation taking place in Nickel⁵; but we have observed that this irregularity is overcome when Constantan is coupled with it.

We have recorded the neutral points (*i.e.*, the mean difference in temperature of the cold and hot junctions of the couple when the thermo-e.m.f. curve crosses the zero e.m.f. line—*vide* Tech. Data on Fuel, 1928, p. 27) at 205° and 275° C. for Iron-Nichrome and Copper-Iron couples respectively.

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¹ *Landolt Bornstein Tabellen*, 1923, p. 1029.

² Eichelberger, *J. Amer. Chem. Soc.*, 1934, **56**, 799.

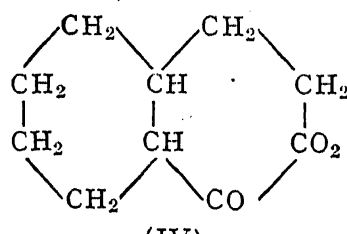
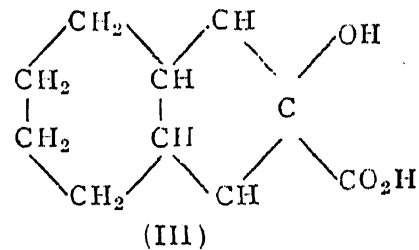
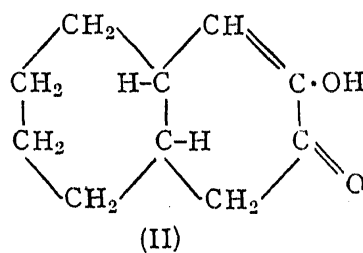
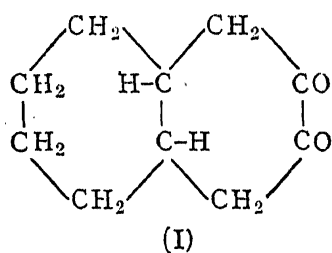
³ Webb and Lindsley, *ibid.*, 1934, **56**, 874.

⁴ Quiggle, Tongsberg and Fenske, *Ind. Eng. Chem., Anal. Ed.*, 1934, **6**, 466.

⁵ *Dictionary of Applied Physics*, 1922, **1**, 901.

2 : 3-Diketotransdecalin.

ON treating the dibromo compound from (*cis*)- β -decalone with aqueous potash, Wallach and Wissenborn¹ obtained (*cis*)-2 : 3-diketodecalin (m.p. 88–89°), along with



a small quantity of a diketonic compound (m.p. 99–100°) not investigated. Recently, Rao and Kuppusamy² obtained by oxidising *trans*- β -decalone with selenium dioxide in boiling xylene (24 hours) two fractions: (i) b.p. 106–08°/6 mm. (yield, about 25%), and (ii) b.p. 126–30°/6 mm. (yield, about 35%), of which the former was considered by them to be the diketone (2 : 3 or 1 : 2) of *trans*decalin and latter was not investigated. The diketone prepared in connection with some other work is now found to be different from that of Rao and Kuppusamy.

Trans- β -decalone was readily oxidised by selenium dioxide in boiling ethyl alcohol (3 hours) to yield a crystalline product, m.p. 99–100° (yield, 60–65%) along with a small quantity of a liquid, b.p. 96–130/5 mm. (5–10%), which on fractionation yielded some decalone and the solid. The compound, m.p. 99–100° (dioxime, m.p. 229°, yielding a characteristic red nickel salt; disemiacarbazone, m.p. 264–65°; quinoxalin derivative, m.p. 177–78°), has now been shown to be *trans*-2 : 3-diketodecalin (I), exhibiting properties of the diosphenol form (II), (i) by oxidation with hydrogen peroxide in quantitative yield to *trans*cyclohexane-1 : 2-diacetic acid, (ii) by reduction with sodium amalgam to 2 : 3-dioxy*trans*decalin, m.p. 141°³ and (iii) by converting to *trans*hexahydrohydrindenol-carboxylic acid (III), m.p. 134°⁴ by boiling with N-sodium hydroxide.

The product m.p. 99–100°, of Wallach and Wissenborn (*loc. cit.*) is evidently 2 : 3-diketotransdecalin, probably formed by the contamination of their *cis*- β -decalone with a little of the *trans*-form; the isomerisation of the diketone (m.p. 88–89°) from the *cis*- to the *trans*-form (I) is not probable since the keto groups in the com-

pound are in the β -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*- β -decalone is found to be reactive.⁵ *Trans*- β -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*- β -decalone with selenium dioxide.

Full details will be published elsewhere.

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¹ *Annalen*, 1924, **437**, 163.

² *J. Annamalai Univ.*, 1937, **7**, 23.

³ Lehmann and Kratschell, *Ber.*, 1934, **67**, 1867.

⁴ —, *ibid.*, 1935, **68**, 360.

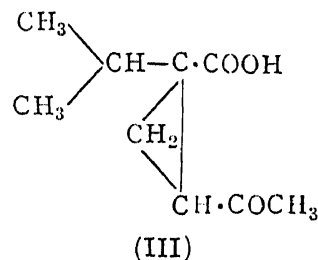
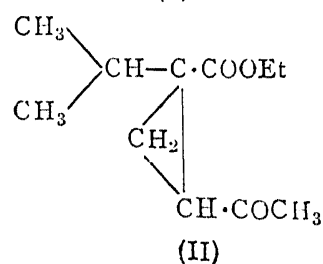
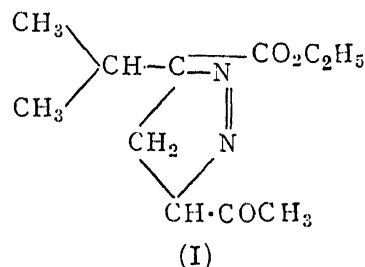
⁵ Feu, Mc Quillon and Robinson, *J.C.S.*, 1937, **53** ;
cf. Cook and Lawrence, *J.C.S.*, 1937, 877.

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. ; II, 9.23 per cent. ; calc. : C, 66.67 ; II, 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 Tutin¹ 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,² 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.³

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¹ *J.C.S.*, 1904, **83**, 645.

² Ranganathan, *J. Ind. Chem. Soc.*, 1936, **13**, 419.

³ *Cf.* Owen and Simonsen, *J.C.S.*, 1933, 1223.