

Synthesis of Thujane.

THE synthesis of thujane, the parent hydrocarbon of the naturally occurring bicyclic terpenes of this group, was undertaken simultaneously in this laboratory by two different methods: *viz.*, (i) starting from a *cyclohexane* derivative having two bromine atoms in positions 2 and 4, a methyl group in position 1, and an isopropyl group in position 4, and (ii) from a *cyclopentene* derivative possessing a methyl group in position 1, a double bond between the carbon atoms in positions 2 and 3, and an isopropyl

group in position 3. Our first scheme resulted in the synthesis of thujane starting from menthol.¹

In the present communication, we are going to describe the synthesis of thujane according to our second scheme starting from ethyl 1-methyl-3-isopropylcyclopentane-2-one-1-carboxylate (I). This compound was prepared according to the method of Kotz and Schuler² starting from diethyl adipate. Compound (I), b.p. 133°/12 mm., was reduced with sodium amalgam to the corresponding secondary alcohol (II), b.p. 153–56°/11 mm. (phenyl urethane derivative, m.p. 144–45°) which in its turn was converted into the cyclopentene derivative (III), (b.p. 114–15°/11 mm.) by the action of phosphorus pentoxide in benzene solution. The unsaturated compound (III) reacts with diazomethane on being allowed to stand in ethereal solution at 0° during two weeks to yield the bicyclo-0:1:3-hexane derivative (IV, b.p. 130–32°/12 mm.) giving on hydrolysis with 5 per cent. KOH the corresponding croboxylic acid (V), m.p. 93–94°; Eq. Wt. found 180.9; required 182. The acid on being distilled with soda lime under reduced pressure gives thujane (VI), b.p. 155–56°;

n_D , 1.4400; d_{20}^{20} , 0.8143, the corresponding values of thujane as given by Semmler and Feldstein³ being b.p. 156–57°; d_{21}^{22} , 0.8158; n_D , 1.44121.

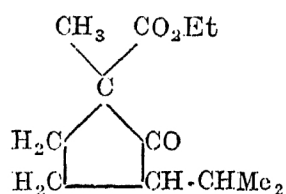
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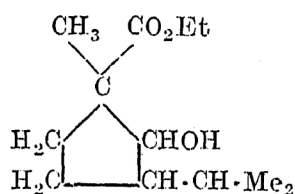
¹ *Ber.*, 1937, 70, 931.

² *Annalen*, 1906, 350, 226.

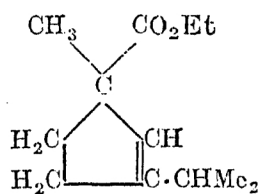
³ *Ber.*, 1914, 47, 387.



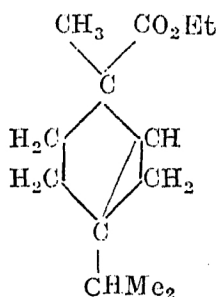
(I)



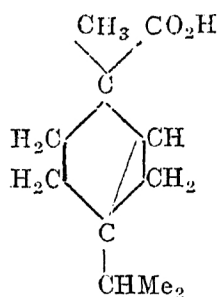
(II)



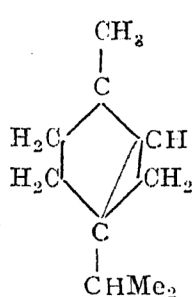
(III)



(IV)



(V)



(VI)