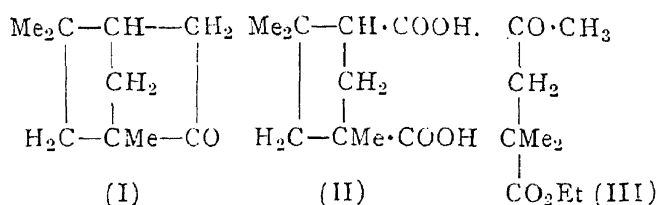
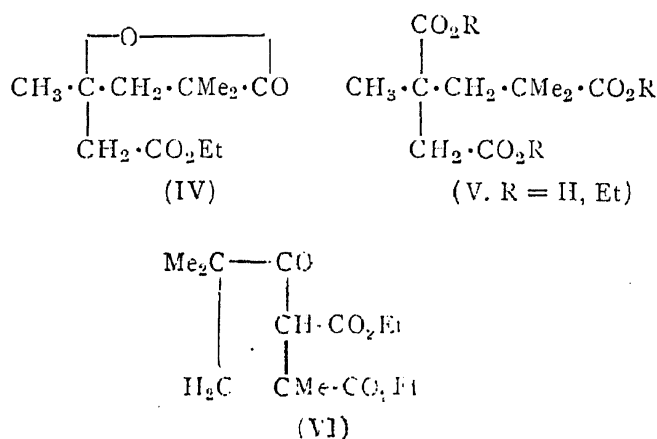


Experiments towards the Synthesis of Isopenchone and Its Degradation Products.

THE classical investigations of Wallach, Aschan and their collaborators¹ have shown the correctness of Semmler's formula for isopenchone (I). Direct evidence by synthesis, however, has been wanting for the constitution of either isopenchone or any of its products of degradation, e.g., isopenchocamphoric acid (II). The present investigation has been undertaken with a view to filling such a gap, and a preliminary report is now made of the results obtained so far.



Ethylmesitonate² (III) (ethyl $\alpha\alpha$ -dimethyl lævulate), b.p. 108–110°/25 mm. (Semicarbazone, m.p. 154°; 2 : 4-dinitrophenylhydrazone, m.p. 98°) has been condensed with zinc and ethyl bromoacetate to yield the lactone of ethyl β -hydroxy- $\beta\delta\delta$ -trimethyl adipate (IV), b.p. 137–38°/6 mm. The lactonic ester adds KCN at 220° and the intermediate cyanoester yields on hydrolysis with concentrated HCl $\beta\delta$ -dimethylpentane- $\beta\delta\epsilon$ -tricarboxylic acid (V, R = H), m.p. 172°. The corresponding ester (V, R = Et) prepared by alcohol vapour method with



concentrated H₂SO₄, boils at 125–128°/1–2 mm. The constitution of the compound (VI) obtained from the ester (V) by cyclisation is being confirmed. Further work on the synthesis of isopenchone from (VI) is in progress.

My thanks are due to Prof. P. C. Guha for his interest in the work.

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¹ Wallach, *Annalen*, 1908, **362**, 191; **363**, 5.

Wallach and Homberger, *Ibid.*, 1909, **369**, 97.

Aschan, *Annalen*, 1912, **387**, 1.

Sandelin, *Ibid.*, 1913, **396**, 285.

² Pinner, *Ber.*, 1882, **15**, 529; cf. Anschutz and Gillet, *Annalen*, 1888, **247**, 99.

A Volumetric Method for the Estimation of Moisture.

OF the two main methods, oven drying and distillation with an entrainer, available for the determination of moisture in substances such as starch and cotton, the former is not always applicable on account of the possibility of decomposition. Thus, except for precautions in the weighing of the highly hygroscopic bone-dry cotton, the moisture content of cotton may normally be estimated by drying in an oven to constant weight, but the method is unsuitable in the case of cotton which contains certain kinds of extraneous matter or has undergone considerable degradation.

For these and other reasons the Marcusson procedure¹ of distilling the substance with a liquid such as toluene or xylene and reading the volume of the water directly has become increasingly popular. This method, however, has not been free from criticism with regard to the time involved, the choice of the entrainer and the entanglement of condensed water. According to Tate and Warren,² who have devised a new apparatus, the comparatively satisfactory types of apparatus due to Friedrichs,³ and to Bidwell and Sterling⁴ both lead to inaccurate results. A new moisture tube has also been recently described by Alexander.⁵ The extensive literature on moisture estimation apparatus would appear to point to the practical difficulties of the method, notably the removal of water drops from the inside of the condenser; as a result an "additive apparatus correction constant"⁵ seems unavoidable. A variation of the process⁶ involving the dehydration of the