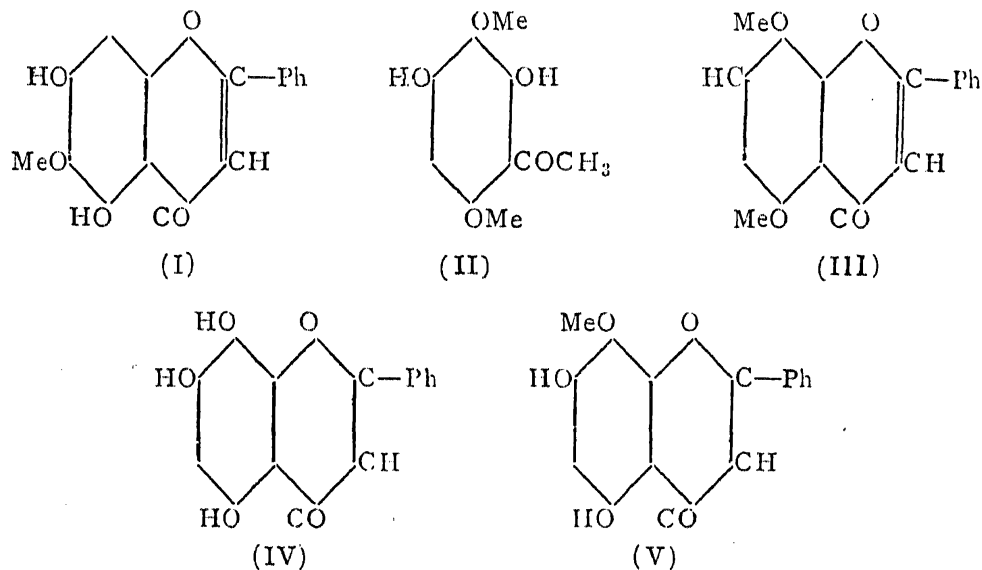


### Synthesis of Wogonin.

THE authors have previously investigated the constitution of Oroxylin-A, a yellow colouring matter isolated from the root bark of *Oroxylum indicum* Vent., and arrived at 5:7-dihydroxy-6-methoxy flavone (I) structure as the most probable formula for Oroxylin-A.<sup>1</sup>

In attempts to synthesise (I) (*cf.* Wessely and Moser),<sup>2</sup> the authors condensed 2:4-dihydroxy-3:6-dimethoxyacetophenone<sup>3</sup> (II) with sodium benzoate and benzoic anhydride, obtaining 7-hydroxy-5:8-dimethoxyflavone (III). Demethylation of (III) with hot hydriodic acid led anomalously to 5:6:7-trihydroxyflavone (baicalein). Demethylation with anhydrous aluminium chloride (1.5 mols) afforded a trihydroxyflavone, m.p. 251–52°, which must be a dimorphic form of the known 5:7:8-trihydroxyflavone<sup>4</sup> (IV) for which the melting point recorded in literature is 227–28°.



Partial demethylation was achieved by the action of anhydrous aluminium chloride (0.75 mol) under milder conditions (*cf.* Gulati and Venkatraman),<sup>5</sup> and gave a dihydroxymethoxyflavone with melting point 200–201°, which is concluded to be Wogonin (5:7-dihydroxy-8-methoxyflavone) (V).

Wogonin which has not been previously synthesised, was first isolated by Takahashi<sup>6</sup> from the root of *Scutellaria baicalensis* Georgi. Its constitution was investigated by Shibata, Iwata and Nakamura,<sup>7</sup> and subsequently more completely by S. Hattori,<sup>4</sup> who assigned to it the formula of 5:7-dihydroxy-8-methoxyflavone. The melting point and properties including colour reactions, solubility, etc., of our synthetic product agree in

all respects with those recorded for Wogonin in literature.

The experimental work described in this note was carried out in the Pharmacological Laboratories of the Seth G. S. Medical College, Parel, Bombay.

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T. S. WHEELER.

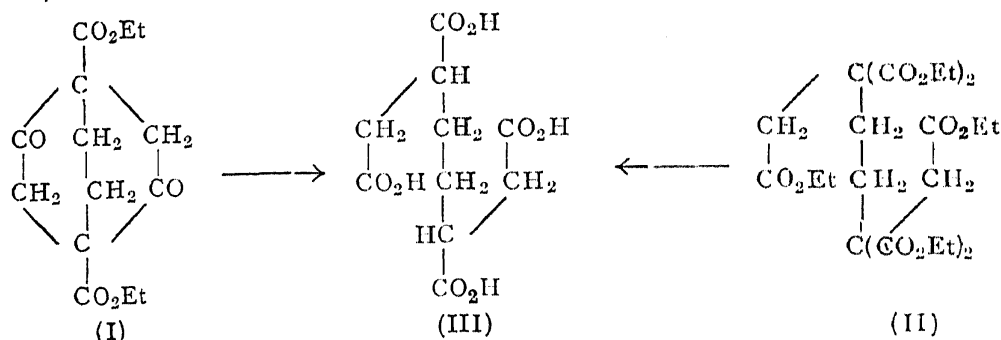
Ismail College, Andheri,  
Seth G. S. Medical College, and  
Royal Institute of Science,  
Bombay,  
April 11, 1938.

- <sup>1</sup> *J.C.S.*, 1936, 591.
- <sup>2</sup> *Monatsch.*, 1930, 56, 97.
- <sup>3</sup> Baker, Nodzu and Robinson, *J.C.S.*, 1929, 74.
- <sup>4</sup> Hattori, *Acta Phytochim.*, 1930, 5, 99.
- <sup>5</sup> *J.C.S.*, 1936, 267.
- <sup>6</sup> *Chem. Zentr.*, 1889, 2, 100.
- <sup>7</sup> *Acta Phytochim.*, 1923, 1, 105.

### Synthesis of $\beta\beta'$ -dicarboxy Suberic Acid.

IN a previous communication by one of us<sup>1</sup> it was shown that bicyclo-(2:2:2)-octanedione dicarboxylic ester (I) gave on treatment with 10 per cent. alcoholic potash a compound which appeared from combustion analysis and equivalent determination to be  $\beta\beta'$ -dicarboxy suberic acid. As this acid is not known in literature, it was considered desirable to synthesise a compound of this structure and then prove its identity with the acid obtained from (I) by alkali treatment.

Ethyl  $\beta\beta\beta'\beta'$ -tetracarboxy suberate (II) has been obtained (i) by the action of ethylene bromide upon carbethoxy succinate



and (ii) by the action of ethyl bromacetate upon ethyl butanetetraacetate. The hexa ester (II) on being boiled with hydrochloric acid (1:1) during 18 hours gets hydrolysed and decarboxylated to give rise to  $\beta\beta'$ -dicarboxy-suberic acid (III). (Found: C, 45.04; H, 4.52; Equiv., 66.34; Calc., C, 45.78; H, 5.38 per cent.; Equiv., 65.5). The acid (III) melts at 177–78° when it is first crystallised from acetic acid and then from water. The acid (III) obtained from (I) on being crystallised from acetic acid and then from water also melts at 177–78°; the mixed melting point with the synthetic variety (m.p. 177–78°) remaining undepressed. The ethyl ester of the acid (III) boils at 195–205°/2 mm. (Found: C, 57.91; Calc., C, 57.75 per cent.).

With a view to effecting a double Dieckmann condensation with the tetra ester, it has been subjected to the action of molecular sodium under varying experimental conditions, the results of which will shortly be published elsewhere.

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<sup>1</sup> Guha, *Curr. Sci.*, 1936, 5, 19.

### The Colouring Matter of Deccan Hemp (*Hibiscus cannabinus*) Flowers— Cannabiscitrin and Cannabiscetin.

FROM the pale yellow flower petals a yellow crystalline glucoside having the formula  $\text{C}_{21}\text{H}_{20}\text{O}_{13}$  has been obtained. It yields a colourless non-acetyl derivative on acetylation. On hydrolysis with dilute mineral acids, it produces a molecule of glucose and a flavonol having the composition  $\text{C}_{15}\text{H}_{10}\text{O}_8$ . The flavonol which occurs free also to some

extent forms a hexa-acetate and a hexa-methylether and displays bright colours in dilute alkaline solutions in the presence of air. It is a penta-hydroxy flavonol which is not identical with gossypetin, quercetagetin or myricetin, but is isomeric. This new member of the flavonol series is named Cannabiscetin and the glucoside therefore becomes Cannabiscitrin. From the flowers of *Hibiscus sabdariffa*, Perkin<sup>1</sup> isolated besides gossypetin a pigment of unknown constitution which he named Hibiscetin. Hence the new names given by us indicate origin from *Cannabinus*.

Cannabiscetin resembles gossypetin in giving the gossypetone reaction though not so readily and probably therefore contains two hydroxyl groups in the positions 5 and 8. Further work is in progress.

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March 14, 1938.

<sup>1</sup> A. G. Perkin, *J.C.S.*, 1909, 1855.

### Influence of Added Chemicals on the Destructive Distillation of Coconut Shells.

SUDBOROUGH, Watson and co-workers<sup>1, 2</sup> in an exhaustive study of distillation of different species of wood and wood wastes, found among the latter class, that coconut shells yielded the highest percentage of acetic acid in the pyroligneous liquor. They also found that the yield of the settled tar from the same source was sufficiently great to warrant a closer study with a view to obtain creosote therefrom. The distillation of the coconut shells has been carried out by A. H. Wells,<sup>3</sup> Georgi and Buckley<sup>4</sup> and by Kidavu and Nambiyar.<sup>5</sup>