

$C_{12}H_{16}O_5$  requires C, 60.0; H, 6.7 and  $-OCH_3$ , 38.8 per cent.). The dried substance melted at 176-77°. The alcoholic mother liquor on dilution with water precipitated a crystalline substance which was identified as 2-hydroxy- $\omega$ :4:6-trimethoxy-acetophenone, m.p. 104-05° C. A mixed melting point with an authentic sample of 2-hydroxy- $\omega$ :4:6-trimethoxy-acetophenone was undepressed.

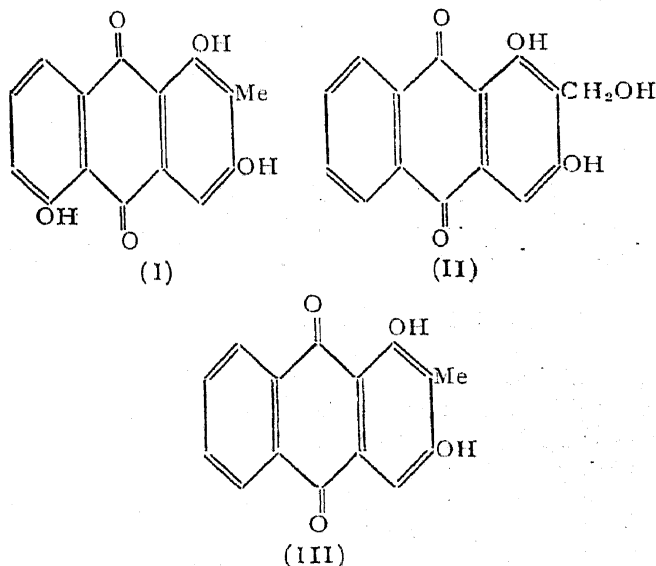
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1. Curd and Robertson, *J. Chem. Soc.*, 1933, 437.

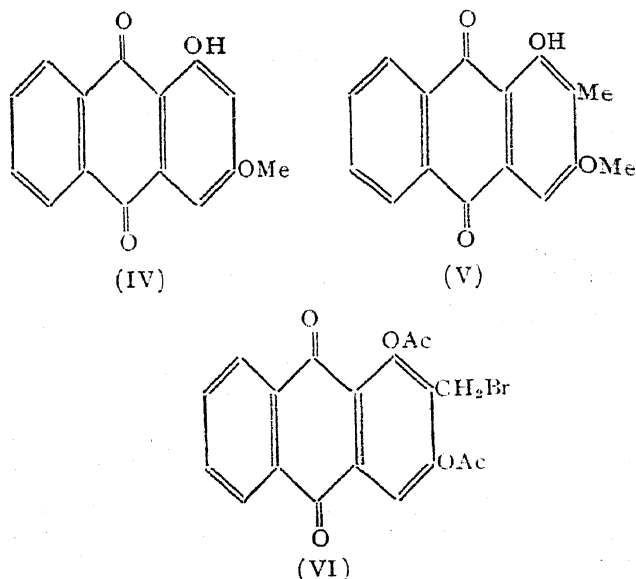
### A SYNTHESIS OF LUCIDIN

FROM the bark of *Coprosma lucida* Briggs and Nicholls<sup>1</sup> isolated a new anthraquinone colouring matter, lucidin, to which they ascribed the structure 1:3:5-trihydroxy-2-methylantraquinone (I). Reconsideration of some of the properties and the infrared spectrum of lucidin led them subsequently to revise the constitution to 1:3-dihydroxy-2-hydroxymethylantraquinone (II).<sup>2</sup> We have confirmed the latter structure (II) by an unambiguous synthesis.



Rubiadin, 1:3-dihydroxy-2-methylantraquinone (III), has been synthesised earlier by several methods, but all are tedious and give poor yields.<sup>3</sup> A greatly improved procedure is an application of the method of Marschalk *et al.*<sup>4</sup> for C-methylation in the anthraquinone series; thus the treatment of the leuco derivative of xanthopurpurin 3-methyl ether (IV)

with formaldehyde gave rubiadin 3-methyl ether (V), which was demethylated to rubiadin (III) by means of boiling hydrobromic and glacial acetic acids. A convenient route to (IV) was the deamination of 1-amino-2:4-dibromoanthraquinone, replacement of bromine by methoxyl by refluxing with sodium methoxide in methanol, and partial demethylation of the resultant 1:3-dimethoxyanthraquinone with hydrobromic acid and acetic acid. Treatment of rubiadin diacetate with N-bromosuccinimide in boiling carbon tetrachloride gave the  $\omega$ -bromo derivative (VI); the position of the bromine



atom was indicated by the ready formation of a pyridinium salt. When (VI) was treated with sodium acetate in boiling alcohol, hydrolysis of both the bromine atom and acetyl groups took place, the product being (II), identical in all its properties with natural lucidin. The triacetate of (II), lucidin triacetate and a mixture of the two had the same melting point, 175-76°.

We are greatly indebted to Dr. L. H. Briggs for samples of natural lucidin and its triacetate.

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1. *J. Chem. Soc.*, 1949, 1241.
2. *Ibid.*, 1953, 3068.
3. Mitter *et al.*, *J. Indian Chem. Soc.*, 1928, 5, 25; 1930, 7, 259; *Ibid.*, 1930, 7, 839; Jones and Robertson, *J. Chem. Soc.*, 1930, 1699; Kusaka, *J. Pharm. Soc. Japan*, 1935, 55, 682.
4. *Bull. Soc. Chim.*, 1936, 3, 1545.