SHORT COMMUNICATIONS

THE CONSTITUENTS OF LEGNEPHORA MOOREI F. MUELL.*

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Legnephora moorei F. Muell., family Menispermaceae, is a robust vine with stems and roots up to 9 in. in diameter, which occurs infrequently in the rainforest and "dry" rain-forest areas of eastern Australia. A Prollius extract of the root-bark gave faintly positive alkaloid tests, but strong tests were obtained on a 2 per cent. hydrochloric acid extract. These preliminary tests indicated that a water-soluble quaternary alkaloid was probably present and this was confirmed by a large-scale extraction which yielded isocorydine methiodide. It is interesting to note that the first natural occurrence of this alkaloid was recently recorded in the Australian species Zanthoxylum brachyacanthum F. Muell. (Cannon et al. 1953). The cyclitol, d-quercitol (named (+)-protoquercitol by Angyal and Macdonald 1952) was also isolated in small yield.

Experimental

Analyses are by Miss J. Fildes, University of Sydney.

(a) Extraction.—The dried milled root-bark (9 kg.) was exhausted with methanol in a continuous extractor and the extract concentrated to 1500 cc. After standing for several weeks in a refrigerator the brown liquid had deposited a mixture of crystals and amorphous material. By swirling with a little methanol the lighter amorphous material was separated from the heavier crystals, which were subsequently identified as d-quercitol (0.025% yield).

The mother liquors were concentrated as far as possible under reduced pressure and the black viscous residue extracted with several portions of hot 1% hydrochloric acid until alkaloid tests were negative. The extract was kept for 2 days in the refrigerator, then saturated with potassium iodide, filtered, and exhausted with chloroform. Evaporation of the chloroform left the crude alkaloid as colourless needles (0.1% yield).

(b) Identification of d-Quercitol.—Crystallization from 70% aqueous methanol gave colourless prisms, m.p. 234 °C., $[\alpha]_D^{20^\circ}+22^\circ$ (c, 1·14% in water) which gave an iodoform test (Found: C, 43·7; H, 7·7%. Calc. for $C_6H_{12}O_5$: C, 43·9; H, 7·4%). According to van der Haar (1922) d-quercitol gives an iodoform test and has m.p. 233–234 °C., $[\alpha]_D^{25^\circ}+24^\circ$.

Benzoylation in pyridine with benzoyl chloride gave the pentabenzoyl derivative which was recrystallized from ethanol as colourless needles, m.p. 155 °C., $[\alpha]_D^{15^\circ} + 59 \cdot 5^\circ$ (c, 1·16% in chloroform). Bauer and Moll (1942) record m.p. 155 °C., $[\alpha]_D^{23^\circ} + 61 \cdot 4^\circ$.

- (c) Identification of isoCorydine Methiodide.—The crude alkaloid was recrystallized first from dilute aqueous potassium iodide and then from absolute ethanol giving colourless needles, m.p. 229 °C. (decomp.), $[\alpha]_{0}^{20} + 132^{\circ}$ (c, 1·71% in water) (Found: C, 52·0; H, 5·4; N, 2·5; I, 26·5%. Calc. for $C_{21}H_{26}O_{4}NI$: C, 52·3; H, 5·5; N, 2·9; I, 26·2).
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The values recorded for isocorydine methiodide are m.p. 224–225 °C. (Barger and Sargent 1939) and $[\alpha]_D^{20^\circ}+143\cdot3^\circ$ (c, $1\cdot02\%$ in 50% ethanol) (Gadamer 1911). The ultraviolet absorption spectrum and the X-ray powder photograph were respectively identical with those of an authentic specimen.

Methylation of the alkaloid by the method of Cannon *et al.* (loc. cit.) gave colourless needles, m.p. 258 °C. (decomp.), $[\alpha]_{\rm D}^{24^{\circ}} + 180^{\circ}$ (c, 0·48% in water). An authentic sample had m.p. 258 °C. (decomp.), $[\alpha]_{\rm D}^{25^{\circ}} + 180^{\circ}$ (c, 0·48% in water).

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