SHORT COMMUNICATIONS

THE CONSTITUENTS OF \textit{LEGENEPHORA MOOREI} F. MUELL.*

By G. K. Hughes,† F. P. Kaiser,† N. Matheson,† and E. Ritchie†

\textit{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 rain-forest 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 \textit{Zanthoxylum brachyacanthum} F. Muell. (Cannon et al. 1953). The cyclitol, \textit{d}-quercitol (named (+)-protoquercitol by Angyal and Macdonald 1952) was also isolated in small yield.

\textit{Experimental}

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

\textit{(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 \textit{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).

\textit{(b) Identification of \textit{d}-Quercitol.}—Crystallization from 70% aqueous methanol gave colourless prisms, m.p. 234 °C, [\(\alpha\)]\textsubscript{D}\textsuperscript{20} +22° (c, 1.14% in water) which gave an iodoform test (Found: C, 43.7; H, 7.7%. Calc. for \textit{C}_{6}\textit{H}_{12}\textit{O}_{5}: C, 43.9; H, 7.4%). According to van der Haar (1922) \textit{d}-quercitol gives an iodoform test and has m.p. 233–234 °C, [\(\alpha\)]\textsubscript{D}\textsuperscript{20} +24°.

Benzoylation in pyridine with benzoyl chloride gave the pentabenzoyl derivative which was recrystallized from ethanol as colourless needles, m.p. 155 °C, [\(\alpha\)]\textsubscript{D}\textsuperscript{19} +59.5° (c, 1.16% in chloroform). Bauer and Moll (1942) record m.p. 155 °C, [\(\alpha\)]\textsubscript{D}\textsuperscript{19} +61.4°.

\textit{(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.), [\(\alpha\)]\textsubscript{D}\textsuperscript{20} +132° (c, 1.71% in water) (Found: C, 52.0; H, 5.4; N, 2.5; I, 26.5%. Calc. for \textit{C}_{12}\textit{H}_{20}\textit{O}_{4}\textit{NI}: C, 52.3; H, 5.5; N, 2.9; I, 26.2).  

* Manuscript received October 10, 1952.
† Department of Organic Chemistry, University of Sydney.
The values recorded for isoerycine methiodide are m.p. 224–225 °C. (Barger and Sargent 1939) and \([\alpha]_D^{25°} +143.3° (c, 1.02\% \text{ in 50}\% \text{ ethanol})\) (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.), \([\alpha]_D^{25°} +180° (c, 0.48\% \text{ in water})\). An authentic sample had m.p. 258 °C. (decomp.), \([\alpha]_D^{25°} +180° (c, 0.48\% \text{ in water})\).

The authors are grateful to Mr. L. J. Webb, C.S.I.R.O., for the plant material and to Mr. C. S. Adams, University of Sydney, for the X-ray powder photographs.

References