ALKALOIDS OF *PACHYGONE PUBESCENS* (MENISPERMACEAE)

By N. K. HART,* S. R. JOHNS,* J. A. LAMBERTON,* and H. SUARES*

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Pachygone pubescens Benth., a woody climber belonging to the family Menispermaceae, was collected at Bamaga in northern Queensland. The crude alkaloids isolated by extraction of roots and tops were obtained as a dark black to purple-coloured, largely intractable mixture, most of which was sparingly soluble in chloroform. Chromatography of the benzene-soluble portion on neutral alumina gave the bisbenzylisoquinoline alkaloid isotrilobine, which had previously been obtained from *Cocculus trilobus* D.C.[1] and *Cocculus sarmentosus* D.C.[2] (family Menispermaceae). From the chloroform-insoluble portion the chlorine-containing alkaloids acutumine and acutumidine were obtained. These alkaloids were previously isolated from *Sinomenium acutum* Rehd. & Wills and *Menispermum dauricum* D.C.,[3] and a biosynthetic scheme for their derivation from hasubanonine has been suggested.[4]

Experimental

The crude alkaloids were separated in approximately 0·3% yield from combined roots and tops of *P. pubescens* (herbarium voucher specimen SN 7816) by the method previously described.[5] The crude alkaloids (12·0 g) were extracted in turn with hot benzene and chloroform and the insoluble residue (5·0 g) was removed by filtration. The benzene-soluble portion was added to a column of alumina (Spence Type H, neutralized with ethyl acetate) and the fractions eluted by benzene–ethyl acetate (9 : 1) consisted largely of one compound. After repeated crystallization from acetone, isotrilobine (570 mg) was obtained as colourless needles, m.p. 217–218°, $[\alpha]_D$ +325° (*c*, 0·41 in $CHCl_3$) (lit. m.p. 215°, $[\alpha]_D$ +343° in $CHCl_3$). The n.m.r. spectrum ($CDCl_3$ solution) confirmed the presence of two methoxyl groups (δ 3·78, 3·93) and two *N*-methyl groups (δ 2·50, 2·31), and the i.r. and mass spectra corresponded with those reported in the literature.[6,7]

When the crude alkaloids were extracted with hot benzene and chloroform, and the extracts were allowed to stand, a brown powdery material (500 mg) gradually separated out. This material was separated by filtration; it was re-dissolved in chloroform–methanol and the solution

* Division of Applied Chemistry, CSIRO, P.O. Box 4331, Melbourne, Vic. 3001.

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decolorized with charcoal. Repeated crystallization from chloroform–methanol and from acetone–methanol gave colourless crystals. This product was a mixture, and comparison of its n.m.r. and mass spectra with those of acutumine and acutumidine[3] indicated that it was an approximately 1 : 1 mixture of these two compounds. These alkaloids are difficult to separate, and fractional crystallization from acetonitrile as recommended by Tomita *et al.*[3] eventually gave pure acutumidine, m.p. 238–240 (dec.), $[\alpha]_D$ −185° (*c*, 0·1 in pyridine), and acutumine, m.p. 239–241°, $[\alpha]_D$ −175° (*c*, 0·7 in pyridine), contaminated with only a trace of acutumidine.

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