

X-RAY STRUCTURE ANALYSIS OF A FURTHER ALKALOID FROM *PODOPETALUM ORMONDII*

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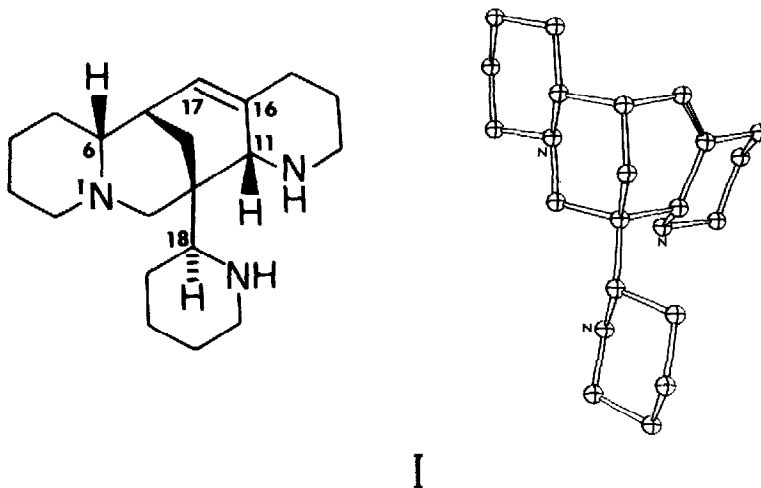
Examination by gas chromatography-mass spectrometry of the total alkaloid extract¹ of *Podopetalum ormondii* F. Muell. has shown the presence of a number of alkaloids with m/e 315 or 317. The most abundant of the alkaloids is podopetaline, the structure and configuration of which was established previously by X-ray crystal structure analysis of its hydrobromide. The alkaloid next in abundance has been assigned the laboratory designation R-6; the pure alkaloid, m.p. 118-119°, $C_{20}H_{33}N_3$ (M^+ 315.2674; satisfactory combustion analysis) was isolated from the extract by a sequence of partitions between aqueous buffers and organic solvents followed by recrystallization from acetone.

Crystals of the free base obtained by slow evaporation from acetone were always twinned. One such twin was cut so that, according to the X-ray data, the ratio of twin volumes was about 5:1.

Crystal data: Triclinic: $a = 9.00$, $b = 10.49$, $c = 19.82 \text{ \AA}$, $\alpha = 97.0$, $\beta = 89.3$, $\gamma = 92.1^\circ$; space group $P1$ or $P\bar{1}$; former confirmed. $Z =$ four molecules per cell.

Only reflections from the major twin component were collected on a diffractometer using $CuK\alpha$ radiation, and, where there was overlapping, intensity contributions from the minor component were subtracted. Of the resulting 6156 reflections measured, 3582 were considered significant. The structure was solved for the four crystallographically independent molecules by direct methods. Current least-squares anisotropic refinement has a conventional R of 0.092.

The four independent molecules are closely similar in dimensions and one of them is shown in I with the same chirality as podopetaline.³ This alkaloid is epimeric with podopetaline at C6; in the crystal, the ring fusion incorporating this centre is trans (lone pair on N trans to C6-H), while in podopetaline (hydrobromide) it is cis. Nevertheless, as in podopetaline, all of the six-membered rings have chair conformations except for the one incorporating the C16-C17 double bond, which has the 'sofa' conformation.



Structure I has already been assigned on chemical grounds to *amazonine*, a constituent of *Ormosia amazonica*, by Valenta.⁴ Their material was isolated in small amounts as a derivative and unfortunately we have not been able to establish unequivocally the identity or nonidentity of their material with ours, hence we are currently refraining from assigning the alkaloid a new name.

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References and Notes

1. We are deeply indebted to Dr. J.A. Lambertson who provided us with a very generous supply of the total extract.
2. N.K. Hart, S.R. Johns, J.A. Lambertson, M.F. Mackay, A. McL. Mathieson and L. Satzke, *Tetrahedron Lett.*, 5333 (1972). M.F. Mackay, L. Satzke and A. McL. Mathieson, *Tetrahedron*, 31, 1295 (1975).
3. This chirality is arbitrarily chosen for convenience of illustration. The optical activity of R-6 is vanishingly small (c.d. from 400 to 220) and the questions that this raises are being investigated currently.
4. Personal communication from Dr. Z. Valenta.