

cent silica gel sheets; acetone as developing solvent) to contain a further alkaloid having R_f 0.57 (R_f alstonisine 0.49; R_f alstonisine 0.66). The new component was purified by preparative TLC (E. Merck silica gel plates) and crystallization from methanol to give macralstonine (IV),⁷ m.p. 276–278°, of identical u.v., i.r., NMR, and mass spectra, and optical rotation to those of authentic material.

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ALKALOIDS OF *HAZUNTA MODESTA*

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Plant. Hazunta modesta (Bak.). Pichon (Syn. *Tabernaemontana modesta* Bak.).

Source. Madagascar.

Previous work. From branches and stem bark of sister species *H. velutina* were isolated vobasine, tabernaemontanine, dregamine, voacarpine, hazuntine and hazuntinine.¹

Examined part. Roots, extracted with EtOH until exhaustion. The alkaloids were dissolved from dry extract in 5% citric acid, the bases set free with aqueous ammonia, extr. with CHCl_3 and chromatographed on a neutral Al_2O_3 column. Ibogamine, tabernaemontanine (eluted with C_6H_6) and dregamine [eluted with C_6H_6 -ether (7:3)] were identified. Total alkaloids constituted 2.5 per cent of weight of the dry roots.

Ibogamine. $\text{C}_{19}\text{H}_{24}\text{N}_2$ —Found: m.p. 155–7° (MeOH); $[\alpha]_D - 36^\circ$ ($\text{C} = 1$, CHCl_3). Required; m.p. 162–3°;² $[\alpha]_D - 36.4^\circ$ (CHCl_3);² i.r.,³ u.v.⁴ and m.s.⁵ were in full accordance with those reported for authentic ibogamine. NMR spectrum (in CDCl_3) provided further confirmation of proposed identification. It showed peaks between τ 2.5 and 3 (5 indole

* M.ps are uncorrected.

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protons), between $\tau 6.5$ and 7.3 (8 protons: >CH-N and Ar-CH-), between $\tau 8$ and 8.7 (8 methylenic and methynic protons); and at $\tau 9.1$ (3 methylic protons).

Tabernaemontanine. $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_3$. Found: m.p. $206-8^\circ$ (MeOH); $[\alpha]_D - 52^\circ$ ($\text{C} = 1$, CHCl_3). Required: m.p. $215-6^\circ$; $[\alpha]_D - 57.5$ ($\text{C} = 1$, CHCl_3)⁷ i.r.,⁸ m.s.⁸ and NMR⁹ spectra were in full accordance with those reported for authentic tabernaemontanine.

Dregamine. $\text{C}_{21}\text{H}_{26}\text{N}_2\text{O}_3$. Found: m.p. $106-8^\circ$ (MeOH); $[\alpha]_D - 90^\circ$ ($\text{C} = 1$, CHCl_3); HCl-ide, m.p. $238-40^\circ$ (EtOH); $\text{CH}_3\text{J-ide}$, m.p. $211-14^\circ$ (EtOH) (Required: m.p. $106-9^\circ$; $[\alpha]_D - 93^\circ$ (CHCl_3);⁹ HCl-ide,⁹ m.p. $249-50^\circ$; $\text{CH}_3\text{J-ide}$, m.p. $215-7^\circ$).⁶ i.r. and u.v. spectra were in full accordance with those reported for authentic dregamine.^{4,9} M.s. and NMR spectra provided further identity confirmation. M.s. showed ion signals at m/e 322, 196, 182, 122, which can be interpreted in terms of cleavages as already reported for tabernaemontanine.⁸ NMR (in CDCl_3) showed peaks at $\tau 0.5$ (>N-H), between $\tau 2.2$ and 3.4 (aromatic

protons), $\tau 6$ (-C-N), whilst 13 protons appeared between $\tau 6.7$ and 7.7 ($\text{CH}_3\text{O-}$, shifted
 $\begin{array}{c} | \\ \text{H} \end{array}$

from its normal $\tau 6.3$ value by the chromatic field which it is pointing toward; $\text{CH}_3\text{N-}$; $\text{-CH}_2\text{-N-}$; $\text{-CH}_2\text{CO-}$; >CH-COOR ; $\text{Ar-CH}_2\text{-}$) and 7 protons between $\tau 8.7$ and 9 ($\text{CH}_3\text{-CH}_2\text{-}$; >CH- ; >CH-), in accordance with the proposed structure.

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