

ISOLATION OF ISOVALLESIIACHOTAMINE FROM LEGUMES OF
RHAZYA STRICTA

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Rhazya stricta Decaisne (Apocynaceae) (1), locally known as "Sehar" (2), is a small glabrous shrub widely used in indigenous medicine for treatment of various diseases (3-5); it has been shown to possess antitumor properties, as well (6,7). As a result of our investigations on the alcoholic extracts of the dried legumes (without seeds) of *R. stricta*, we have isolated an alkaloid that has not been reported previously from this plant.

EXPERIMENTAL¹

The crude alkaloids obtained from the alcoholic extract of the legumes were subjected to selective extractions with organic solvents according to their differential basicities. Alkaloid **1** was separated from the fraction of alkaloids extracted at pH 8.4 and was purified by preparative tlc on silica gel (GF-254) plates in ether-light petroleum (40°-60°) (9:1). The substance was obtained as a crystalline compound, mp 244°, unstable to air and light.

The uv spectrum (MeOH) of the compound showed λ max 223, 291 nm and λ min 256 nm, characteristic of a 2,3-disubstituted indole chromophore. An unusually high extinction coefficient at 291 nm suggested the presence of a second chromophore absorbing in this region.

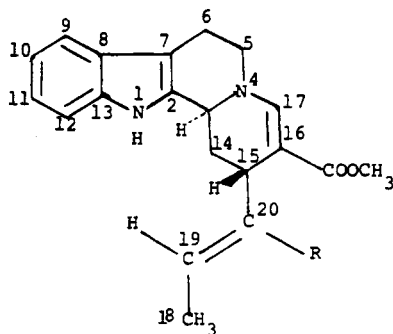
The ir spectrum (CHCl₃) exhibited bands at 3460 (NH), 3310 (C-H), 1665 (α,β -unsaturated C=O), and 1610 cm⁻¹ (C=C). The molecular formula was established by high resolution ms to be C₂₁H₂₂N₂O₃ (calcd 350.1630; obsd 350.1650).

The major peaks were found to occur at m/z 350 (M⁺, 69%), 335 (13%), 322 (39%), 291 (58%), 281 (11%), 279 (100%), 263 (90%), 221 (62%), 170 (25%), and 156 (24%). The pmr spectrum (CDCl₃) afforded resonances identical to those recently reported for isovallesiachotamine (8).

Reduction of the alkaloid with NaBH₄ afforded a new, slower running material, which afforded the molecular ion at m/z = 352 (46%) and other major peaks at 335 (24%), 321 (24%), 293 (13%), 281 (27%), 279 (100%), 221 (18%), 156 (17%), and 144 (17%). The pmr (CDCl₃) of the reduced product afforded resonances at δ 1.74 (d, 3H, J = 6.9 Hz, C-18H), δ 3.67 (s, 3H, COOCH₃), δ 5.40 (q, J = 6.9 Hz, C-19H), and δ 8.30 (s, 1H, indole NH). No aldehyde proton was visible. It was clear from these data that reduction of the aldehyde to the corresponding alcohol **2** had occurred.

On the basis of these spectroscopic and chemical evidences, the alkaloid isolated has been identified as isovallesiachotamine (**1**). It is plausible that isovallesiachotamine may be formed in the plant directly from strictosidine.

Full details of isolation and identification are available on request to the senior author.



- 1:** R = CHO
2: R = CH₂OH

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¹Spectra were recorded on Jasco-IRA-1 ir spectrophotometer, Shimadzu uv-240 uv spectrophotometer, Finnigan MAT312 mass spectrometer, and Bruker WP-100 SY nmr spectrometer.

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ISOLATION OF RHAZIMOL FROM THE LEAVES OF *CATHARANTHUS ROSEUS*

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We have previously reported the isolation and structure of 16-epi-19-*S*-vindolinine, 16-epi-19-*S*-vindolinine-*N*-oxide, fluorocarpamine-*N*-oxide, vindolinine-*N*-oxide, fluorocarpamine, and pleiocarpamine from the leaves of *Catharanthus roseus* (L.) G. Don. (Apocynaceae) (1,2). We now report the isolation of r hazimol, not previously reported from this plant but isolated from *Rhazya stricta* (3) and *Picralima nitida* (4).

EXPERIMENTAL¹

PLANT MATERIAL.—Leaves of *C. roseus* were collected from a field on the University Campus in April 1982. The plant was identified by Dr. Irtifaq Ali, Professor of Botany, University of Karachi.

EXTRACTION AND FRACTIONATION.—The crude alkaloids (120 g) obtained from the alcoholic extract of the air-dried leaves (20 kg) of the plant were dissolved in CHCl_3 (400 ml) and extracted with pH-3 phosphate buffer (1 liter). The CHCl_3 layer was dried (anhydrous Na_2SO_4), concentrated to one-third of its original volume and petroleum ether (300 ml) added to the CHCl_3 solution, which caused some of the alkaloids to precipitate out. The precipitates were filtered, and the filtrate was again concentrated to a gum (36 g). The gum was dissolved in EtOAc (200 ml) and extracted with pH-2 phosphate buffer (1 liter). The aqueous layer was separated, washed with CHCl_3 , basified with NH_3 to pH-10, and again extracted with CHCl_3 (1 liter) to afford fraction (F_1) (20 g). This fraction was chromatographed on an alumina column (200 g), elution being carried out with EtOAc (5 liters). The eluates were concentrated and again loaded (10 g) on another column of tlc grade silica (30 g). The column was eluted with increasing polarities of petroleum ether (1 liter), EtOAc-MeOH (2 liters) and MeOH (1 liter).

ISOLATION OF RHAZIMOL.—Preparative tlc of the MeOH fraction (1.0) in 20% Me_2CO -80% petroleum ether yielded r hazimol (5 mg).

Comparison of ir, uv, pmr, and ms spectral data with those reported in the literature for r hazimol, as well as comparison with an authentic sample of r hazimol isolated by us from *Rhazya stricta* leaves, unambiguously established the identity of the material as r hazimol (3). This alkaloid has not previously been reported from *C. roseus*.

Full details of isolation and identification are available on request to the senior author.

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¹Spectra were recorded on Jasco-IRA-1 ir spectrophotometer, Shimadzu uv-240 uv spectrophotometer, Finnigan MAT 312 mass spectrometer, and Brüker WP-100 SY nmr spectrometer.