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Note

Separation of some oxindole alkaloids using high-efficiency liquid chromatography

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During recent years considerable interest has been shown in the phytochemical investigation of the genus *Mitragyna* from which a number of oxindole alkaloids have been isolated. Thin-layer chromatography has been used extensively for purposes of separation¹⁻⁵, but difficulties occur in attempting complete separation of isomitraphylline, uncarine F, isopteropodine, and isorhynchophylline and Phillipson and Hemingway⁶ in their gas chromatographic examination of the oxindole alkaloids report no separation of the isomeric pairs.

These difficulties have been overcome by using high-efficiency liquid chromatography.

EXPERIMENTAL

Instrument: Nester Faust (Perkin-Elmer) Model 1240 analytical liquid chromatograph.

Stationary phase: Corasil C18.

Column size: 0.5 m × 1.7 mm I.D.

Mobile phase: methanol-water (80:20) at 60°.

Column pressure: 100 p.s.i.

Flow-rate: 0.5 ml/min.

Column temperature: 60°.

Detector: UV, 254 nm.

Alkaloids investigated: (a) Closed E-ring alkaloids, speciophylline, uncarine F; pteropodine, isopteropodine; mitraphylline and isomitraphylline; (b) Open E-ring alkaloids, rhynchophylline, isorhynchophylline; rotundifoline, isorotundifoline; rhynchociline and ciliaphylline.

RESULTS AND DISCUSSION

Obtained R_T values (sec): speciophylline, 180, uncarine F, 108; pteropodine, 138, isopteropodine, 48; mitraphylline, 192, isomitraphylline, 372; rhynchophylline, 216, isorhynchophylline, 162; rotundifoline, 102, isorotundifoline, 168; rhynchociline, 378, ciliaphylline, 228.

Each pair of spiro isomers is clearly separated and there are no difficulties in

distinguishing between isomitraphylline, isorhynchophylline, uncarine F, and isopteropodine. It will be observed that C(9)-OH substitution (rotundifoline and isorotundifoline) leads to increased water solubility whereas C(9)-OCH₃ substitution (rhynchocilline, ciliaphylline) leads to reduced water solubility. (Rhynchophylline and isorhynchophylline are the C(9) unsubstituted comparable alkaloids.)

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