TROPINE 3,4,5-TRIMETHOXYCINNAMATE, A NEW ALKALOID FROM *ERYTHROXYLUM ELLIPTICUM* (ERYTHROXYLACEAE)

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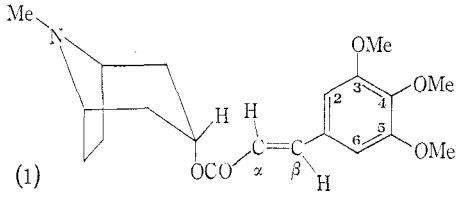
[Manuscript received October 2, 1969]

Erythroxylum ellipticum R.Br (family Erythroxylaceae) is a Queensland tree growing to a height of 35 ft. Extraction of stem bark, collected from a tree growing in *Eucalyptus* forest near Laura, has afforded 0·32% of crude alkaloids. The major component of the alkaloids is tropine 3,4,5-trimethoxycinnamate (1), the identification of which has been confirmed by comparison with synthetic (1) prepared from tropine and 3,4,5-trimethoxycinnamoyl chloride. N.m.r. spectra of the total crude alkaloids, and of fractions obtained by chromatography on alumina, indicate that a second component, tropine benzoate, is also present, and its identification has been confirmed by comparison of the retention time of authentic tropine benzoate by g.l.c. and by the isolation of a small sample by preparative g.l.c. for spectroscopic comparison.



(1)

Experimental

Extraction of milled, dried bark (100 g) by continuous percolation with ethanol at 40°, and work-up by the method previously described for the isolation of total non-quaternary alkaloids,[1] yielded 320 mg of crude alkaloids. Chromatography on a column of neutral alumina gave the major alkaloid as a series of crystalline fractions which were eluted by benzene–chloroform (1 : 1). Crystallization from acetone gave *tropine 3,4,5-trimethoxycinnamate* as colourless prisms, m.p. 165–166°, ν_{max} 1635, 1702 cm^{-1} in CCl_4 (Found: C, 66·7; H, 7·4; N, 4·0. $C_{20}H_{27}NO_5$ requires C, 66·5; H, 7·5; N, 3·9%). The n.m.r. spectrum ($CDCl_3$ solution, tetramethylsilane δ 0·00) showed sharp signals at δ 3·84 (6H, s, $2OCH_3$), δ 3·64 (3H, s, OCH_3), δ 6·74 (2H, s, C 2–H and C 6–H), δ 6·28 (1H, d, *J* 16 Hz, C α–H), δ 7·55 (1H, d, *J* 16 Hz, C β–H) and δ 2·30 (3H, s, NMe), and a series of multiplets ascribed to the protons of the tropine residue.

Mixtures of tropine 3,4,5-trimethoxycinnamate with the second alkaloid were eluted from the alumina column in front of the main fractions consisting of alkaloid (1), and showed, in addition to the n.m.r. signals described above, an extended multiplet typical of the aromatic protons of a benzoate ester. Analysis by gas chromatography showed the presence of a component having the

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[1] Johns, S. R., Lamberton, J. A., and Sioumis, A. A., *Aust. J. Chem.*, 1966, **19**, 2331.

same retention time as tropine benzoate on a column of silanized Chromosorb W (60–80 mesh) coated with 3% SE30. A small sample of the alkaloid was collected by preparative g.l.c., and its i.r. spectrum was identical with that of authentic tropine benzoate.

Synthetic tropine 3,4,5-trimethoxycinnamate was obtained by adding a solution of freshly prepared 3,4,5-trimethoxycinnamoyl chloride (200 mg) in dry chloroform to a solution of tropine (200 mg) in chloroform. The crude basic product was chromatographed on neutral alumina and a major fraction (100 mg) eluted by benzene–chloroform (1 : 1) consisted of tropine 3,4,5-trimethoxycinnamate. Crystallization from acetone gave the synthetic ester as colourless prisms, m.p. 165–166°, identical with the alkaloid from *E. ellipticum* (mixed m.p., comparison of i.r. and n.m.r. spectra).

Acknowledgment

The authors are indebted to Mr. W. T. Jones for the collection of *E. ellipticum*.