

We have previously reported^{1,2} on the main alkaloids of *Romneya coulteri* var. *trichocalyx* (Eastwood) Jepson and now wish to report that this plant also contains sanguinarine (isolated as the 14-methoxydihydrosanguinarine derivative) and dihydrosanguinarine. Experimental details of isolation and identification are given below.

EXPERIMENTAL

Dried, powdered root (200 g) of *Romneya coulteri* var. *trichocalyx* (Eastwood) Jepson¹ was macerated with 100 ml of 10% Na₂CO₃ solution, and 300 ml of 1:1 *n*-BuOH–benzene was added, and the mixture allowed to stand overnight. It was filtered and the filtrate was extracted 3 × with 100 ml of 1M H₂SO₄. The acidic layers were combined, made basic to pH 9, and extracted with 3 vol. CHCl₃. The CHCl₃ solutions were combined, dried (Na₂SO₄) and evaporated to yield 1.3 g of crude gummy alkaloids. This residue was dissolved in CHCl₃ and chromatographed through a column of Florisil (55 g, 60–100 mesh, Fisher Scientific Co.) prepared in CHCl₃. Elution with CHCl₃ yielded first, after evaporation, 120 mg of crude syrup which contained mainly two alkaloids by TLC (*R_f* = 0.52 and 0.65 silica gel with EtOAc). Visualization was with Dragendorff's reagent and UV light. The previously isolated alkaloids (coulteropine, romneine, protopine, and reticuline) were eluted in later fractions.

The 120 mg of crude alkaloids was separated by preparative TLC into two bands which were removed from the silica gel with MeOH and CHCl₃. The *R_f* = 0.65 band material (35 mg) was recrystallized from benzene–EtOH to yield dihydrosanguinarine, m.p. 188–190°, identical with a sample prepared by reduction of authentic sanguinarine. The *R_f* = 0.52 band (62 mg) was recrystallized from CHCl₃–MeOH to yield light tan crystals, m.p. 216–219°, assigned the 14-methoxydihydrosanguinarine structure by comparison with an authentic sample prepared as follows. Commercial 'sanguinarine nitrate' (Aldrich Chemical Co.; a mixture of sanguinarine, chelerythrine, protopine, and other alkaloids) was purified by a chromatographic scheme like that described above for the isolation work to yield pure sanguinarine nitrate. This was dissolved in basic solution, extracted with CHCl₃, and the residue crystallized from CHCl₃–MeOH to yield light tan crystals of m.p. 216–219°, identical with the isolated compound. *Anal.*: Calc. for C₂₁H₁₈NO₅: C, 69.22; H, 4.97; N, 3.84. Found: C, 69.29; H, 4.65; N, 3.54. MS: *m/e* 364 (M⁺); 332 (base peak = M⁺ – OCH₃).

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¹ F. R. STERMITZ, L. CHEN and J. I. WHITE, *Tetrahedron* **22**, 1095 (1966).

² F. R. STERMITZ and L. C. TENG, *Tetrahedron Letters* 1601 (1967).

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PIPERACEAE

CONSTITUENTS FROM *PIPER BANKSII*

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Key Word Index—*Piper banksii*; Piperaceae; elemicin; dillapiole; *N*-isobutyl-*trans*-2, *trans* 4-octadienamide.

Plant. *Piper banksii* Miq.—Piperaceae. *Source*. Clump Point, near Tully, N. Queensland. Herbarium No. SN 8276. *Previous work*. Related species, *P. novae-hollandiae*.¹

Leaf and stem. Constituents extracted and separated as before.¹ Steam volatile oil contained elemicin and dillapiole. The only amide detected was *N*-isobutyl-*trans*-2, *trans*-4-octadienamide, but the small plant sample precluded examination of minor components.

¹ J. W. LODER, A. MOORHOUSE and G. B. RUSSELL, *Austral. J. Chem.* **22**, 1531 (1969).

EXPERIMENTAL

Elemicin identified from PMR, IR, UV and MS; oxidized (KMnO_4) to 3,4,5-trimethoxybenzoic acid (m.p., m.m.p., PMR). *Dillapiole* identified by comparison with authentic sample (PMR, MS and co-GLC (two stationary phases)). *N-isobutyl-trans-2,trans-4-octadienamide*, m.p. 88–90° (Recorded² m.p. 94°) was identified by comparison with an authentic sample (MS, co-GLC on two stationary phases: SE-30 and OV-17).

² M. JACOBSON, *J. Am. Chem. Soc.* **78**, 5084 (1965).

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CONSTITUENTS OF *PIPER NEPALENSE*

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Key Word Index—*Piper nepalense*; Piperaceae; caryophyllene oxide; triacontanol; sitosterol; *N-isobutyl-deca-trans-2-trans-4-dienamide*; piperine; piperlonguminine.

Plant. *Piper nepalense* Miq. *Source.* Himalayas. *Uses.* In Ayurvedic medicine. *Previous work.* None in the literature. *Date.* Collected in August 1971.

Stems. Powdered stems (0.5 kg) extracted with light petroleum (60–80°). The individual constituents were isolated by chromatography (neutral alumina, Brockmann) and by preparative TLC. The compounds were identified by m.p., m.m.p., TLC, UV, IR, NMR and MS.

Constituents. Caryophyllene oxide, m.p. 59–60°, $\text{C}_{15}\text{H}_{24}\text{O}$; triacontanol, m.p. 87–88°, $\text{C}_{30}\text{H}_{62}\text{O}$, sitosterol, m.p. 136–137°, $\text{C}_{29}\text{H}_{50}\text{O}$, *N-isobutyl-deca-trans-2-trans-4-dienamide*,¹ m.p. 82–83°, $\text{C}_{14}\text{H}_{25}\text{NO}$; piperine, m.p. 129–130°, $\text{C}_{17}\text{H}_{19}\text{NO}_3$; and piperlonguminine,² m.p. 166–168°, $\text{C}_{16}\text{H}_{19}\text{NO}_3$.

¹ K. L. DHAR and C. K. ATAL, *Indian J. Chem.* **5**, 588 (1967).

² A. CHATTERJEE and C. P. DUTTA, *Tetrahedron Letters* **16**, 1797 (1966).