SHORT COMMUNICATION

ALKALOIDS OF SOPHORA MACROCARPA

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DURING a current project dealing with Chilean flora^{1,2} now under way in the laboratories at Concepción we have made a study of Sophora macrocarpa SM., a Leguminoseae indigenous to Chile. The defatted plant material, upon treatment with ethanol containing 2 per cent v/v acetic acid, afforded an extract which yielded α -matrine (I),³ methylcytisine⁴ and baptifoline (II).⁵ The alkaloids were characterized by their physical and spectroscopic properties. Methylcytisine and α -matrine were also detected, by paper chromatography, in the light petroleum and benzene-soluble extracts of the plant.

The mass spectrum of baptifoline was of interest. It showed a strong parent ion at m/e 260 (65 per cent) and the base peak at m/e 114, corresponding to $C_6H_{12}NO^+$. A possible fragmentation leading to the base peak is as follows:

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¹ M. SILVA, M. BALOCCHI and P. G. SAMMES, Phytochemistry, in press.

² M. SILVA, R. STUCK and P. CHAVEZ, Arch. Pharm. 295, 7, 515 (1962).

³ F. BOHLMANN, W. WEISE, D. RAHTZ and C. ARNDT, Chem. Ber. 91, 2177 (1958).

⁴ L. H. Briggs and J. Ricketts, J. Chem. Soc. 1795 (1937).

⁵ F. BOHLMANN, C. RAHTZ and C. ARNDT, Chem. Ber. 91, 2189 (1958).

$$(II) \longrightarrow (II) \longrightarrow$$

EXPERIMENTAL

All m.p.s are uncorrected. Microanalyses were performed by Dr. A. Bernhardt, Mulheim, Germany. U.v. spectra were recorded in absolute ethanol solution using a Beckman DU spectrophotometer. NMR spectra were determined on a Varian HA 100 instrument using deuterochloroform as solvent and tetramethylsilane as an internal reference. The mass spectra were performed with an A.E.I. MS9, double-focusing mass spectrometer. The alumina used was Merck neutral grade active aluminium oxide. Paper chromatography was on Whatman No. 1 paper using n-butanol-acetic acid-water, 4:1:5, as solvent. Light petroleum refers to the fraction of b.p. 65-75°.

Extraction of the Alkaloid Fraction

Seeds of Sophora macrocarpa, collected during the summer from near Rere, Yumbel, Chile, were dried and crushed. The dried powder (4558 g) was extracted in a Soxhlet apparatus, first with light petroleum and then with benzene, to exhaustion. The defatted plant material was dried and then the constituents soluble in ethanol containing 2 per cent v/v of acetic acid were extracted. The dark solution was concentrated before diluting with water and the remaining ethanol removed by steam distillation. The solution was brought up to pH 9 and the precipitate thus formed was discarded. The filtrate was continuously extracted with CHCl₃. The CHCl₃ solution was dried (Na₂SO₄), filtered and then concentrated to dryness under reduced pressure. The gummy residue (40 g) was treated with boiling light petroleum to give a petroleum ether soluble fraction.

α-Matrine

The light petroleum soluble fraction (2 g) was chromatographed over Whatman cellulose powder (standard grade). Exhaustive elutions with light petroleum containing increasing amounts of ether afforded α -matrine (500 mg) and a mixture of two other, more polar, alkaloids. The α -matrine, after recrystallization from petroleum ether, had m.p. 75-77°, $\nu_{\max}^{\rm KBF}$ 2900, 2850, 2725, 2660, 1630, 1620, 1470, 1434 and 1413 cm⁻¹. (Found: C, 72·43; H, 9·64; N, 11·28. Calc. for C₁₅H₂₄N₂O: C, 72·54; H, 9·74; N, 11·28 per cent.) The material was identical to a reference sample in its physical properties.

Careful chromatography of the light petroleum insoluble fraction (33 g), on alumina (Grade II, 200 g) with benzene as solvent containing increasing amounts of CHCl₃, afforded, respectively, the alkaloids baptifoline and methylcytisine, characterized as described below.

Baptifoline

Recrystallization from benzene gave crystals (100 mg), m.p. 200°, $[\alpha]_D^{26} - 161^\circ$ (c 1·4, chloroform), $[\alpha]_D^{26} - 154^\circ$ (c 0·9, ethanol), λ_{\max} 235 (ϵ 4400), and 310 nm (ϵ 4840), $\nu_{\max}^{CHL^2}$ 3380, 2920 and 1650 cm⁻¹. The NMR spectrum showed, in the vinyl region, an AMX pattern, at τ 2·78 (1 proton, H_A), 3·66 (1 proton, H_M), 4·08 (1 proton H_X), $J_{AM} = 8\cdot7$ c/s, $J_{AX} = 7\cdot0$ c/s, $J_{MX} = 1\cdot3$ c/s. Its mass spectrum showed peaks at m/e 260 (M^+ , 65 per cent), 243 (8), 188 (4), 160 (12), 146 (21), 114 (100), 96 (11), 70 (18). (Found: N, 11·08. Calc. for $C_{15}H_{20}N_{2}O_{2}$; N, 10·76 per cent.) No melting point depression was observed upon admixture with authentic baptifoline. Paper chromatography confirmed the identity.

Methylcytisine

Recrystallization from light petroleum yielded needles (270 mg), m.p. and mixed m.p. 133–138°, λ_{max} 233 (ϵ 6850) and 308 nm (ϵ 7900), $\nu_{\text{max}}^{\text{KBr}}$ 2937, 2833, 2779, 1660, 1645, 1575, 1550, 1470 and 1425 cm⁻¹. (Found: C, 70-40; H, 8-00; N, 13-86. Calc. for C₁₂H₆N₂O: C, 70-56; H, 7-90; N, 13-72 per cent.) Further identity was established by paper chromatography.

Baptifoline and methylcytisine were also detected in the light petroleum soluble fraction.

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