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N-ACETYLCYTISINE FROM *SOPHORA TOMENTOSA*

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Plant. Sophora tomentosa, L., collected in April in the Bonnin Islands, Japan. *Previous work.* Isolation of lupine alkaloids matrine, cytisine and *N*-methylcytisine.[1] *N*-Acetylcytisine has previously been isolated from *Thermopsis alterniflora*.[2]

Present work. Seven alkaloids were isolated; *N*-acetylcytisine, baptifoline, anagyrine and oxymatrine have not previously been isolated from *S. tomentosa*.

EXPERIMENTAL

Extraction and fractionation. Dried aereal parts of *S. tomentosa* (3·25 kg) was extracted with 70% EtOH, and the crude alkaloids (10·42 g) were displaced from the alumina (250 g) column by a modification of previous methods,[3,4] 100 ml fractions being collected: C_6H_6 was used to elute fraction 1–17, C_6H_6–Et_2O (1 : 1) for 18–25, CH_2Cl_2 for 26–40, and MeOH for the remainder. Fractions within each group were combined and further separated by employing preparative TLC or columns on silica gel and alumina: fractions 1–17* gave matrine (0·15%), m.p. 76°; 18–25, *N*-methylcytisine (trace) and anagyrine (trace), after purification by preparative TLC (alumina; C_6H_6–Me_2CO–MeOH, 34 : 3 : 3); 26–30, the combined alkaloids mixture was passed through a silica gel (40 g) column using CH_2Cl_2–MeOH–28% NH_4OH (94 : 5 : 0·3) and collected in 10 ml fraction. *N*-Acetylcytisine (0·001%) appeared in fractions 10–15, cystisine (0·055%) in 40–60. N-*Acetylcytisine*, m.p. 210–213°, $[\alpha]_D^{20}$ −208° (*c*, 0·182 in EtOH), IR ν_{max}^{KBr} 1610–1660 cm^{-1} (broad, C=O), MS *m/e* 232 (M^+, 61%), significant peaks at *m/e* 190(18), 189(16), 160(21), 147(91) and 146(100). It was found to be identical in all respects when compared with synthetic material. Cytisine, m.p. 155–156°, was purified by sublimation under vacuo (10^{-2} mmHg); 31–40, gave cytisine (0·035%) and oxymatrine (0·025%), m.p. 212–213°, after purification on silica gel (30 g) column using CH_2Cl_2–MeOH–28% NH_4OH (94 : 5 : 1); Silica gel (30 g) column using the solvent system CH_2Cl_2–MeOH–28% NH_4OH (95 : 4 : 1) for MeOH fraction for the remainder enabled the recovery and identification of baptifoline (0·022%) and oxymatrine (0·02%). Baptifoline, m.p. 210–213°, after recrystallization from C_6H_6.

All compounds were identified by comparison with authentic samples (MS, m.ps, co-TLC and IR).

Synthesis of N-*acetylcytisine.* A cytisine was refluxed with Ac_2O for 8 hr. After concentration *in vacuo* the crystalline product was recrystallized from CH_2Cl_2–Et_2O to yield *N*-acetylcytisine (40 mg), m.p. 210–213°. Identical with *N*-acetylcytisine obtained from the natural source on MS, m.m.p., co-TLC and IR.

* Sophocarpine was not identified in this fraction by the method[3] of a rearrangement of sophocarpine into 13-ethylsophoramine on a prolonged heating in 10% KOH–EtOH.

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