ALKALOIDS OF Verbascum nobile

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Verbascum nobile Vel., family Scrophulariaceae is an endemic plant growing along the banks of the river Strum in southwestern Bulgaria [1-4].

The comminuted raw material collected on September 20, 1970, was moistened with 10% ammonia solution and extracted with chloroform. The chloroform extract was concentrated and extracted with 10% sulfuric acid, the acid solution of alklaoid salts was made alkaline with 25% ammonia and again extracted with ether and then with chloroform. After distillation of the extracts, 0.5% of the ethereal and 0.6% of the chloroform fractions of the combined alkaloids, based on the weight of the dry plant, were obtained.

The ethereal fraction of the mixed bases was separated into ten fractions according to basicity. Fractions 8-10 were chromatographed on a column of silica gel. Three individual bases were isolated: (I), (II), and (III), with mp 100° C, R_f 0.54 on TLC in silica gel in the benzene–ethanol (4:1) system. Base (I) was called verbasine and (II) verbaskine.

Verbasine has the composition $C_{25}H_{42}N_4O_4$, mp 74-75° C, mol. wt. 462 (mass spectrometrically), R_f 0.10. Its IR spectrum (in ethanol) has maxima at 218, 225, and 284 nm (log ε 4.11, 3.96, 4.16).

The IR spectrum of (I) has bands at 3300 cm^{-1} (⟩NH), 2940 and 2870 cm^{-1} (–CH_2–), 1650 cm^{-1} (–NH–C(=O)–), 1605 cm^{-1} (aromatic ring), and 775 and 715 cm^{-1}.

The mass spectrum of (I) has peaks of ions with m/e 462, 419, 371, 331, 289, 161, 160, 147, 146, 145, 131, 117, 105, 91, 85, 84 and 77.

Verbaskine, $C_{27}H_{44}N_4O_4$, was obtained with mp 125-126° C (ethanol), mol. wt. 488 (mass spectrometrically), R_f 0.61. UV spectrum of (II): λ_{max} (ethanol), 219, 225, 284 nm (log ε 4.16, 4.05, 4.20). Its IR spectrum shows absorption bands at 3440 cm^{-1} (OH), 3300 cm^{-1} (⟩NH), 3070, 2940, and 2870 cm^{-1} (–CH_2–), 1650 cm^{-1} (–NH–C(=O)–), 1610 cm^{-1} (aromatic ring), and 775 and 715 cm^{-1}. In the mass spectrum of (II) there are peaks of the molecular ion with m/e 488 and of other fragments (with m/e 357, 210, 155, 154, 147, 146, 131, 121, 105, 103, 91, 84 and 77).

From the chloroform fraction of the combined alkaloids a base (IV) was isolated with mp 133-135° C, R_f 0.24, a mixture of which with the pediculinine from Pedicularis olgae [5] gave no depression of the melting point.

The mass spectra were taken on a MKh-1303 mass spectrometer at an energy of the ionizing electrons of 40 eV and at a temperature of 145° C for (I) and 140° C for (II).

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