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COMPONENTS OF THE ROOTS OF Haplophyllum perforatum

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The roots of the plant <u>Haplophyllum perforatum</u> (MB.) Kar. et Kir., growing in the Kizylkum region of Chimkent province, Kazakh SSR, have not been studied previously.

The dry comminuted raw material (1 kg, collected in the flowering period) was extracted with methanol. The concentrated and dried methanolic extract was separated by the usual method into basic and neutral fractions. The latter were separated chromatographically on columns of silica gel. From the basic fraction (3 g) were isolated 0.5 g of a substance with mp 117-118°C, 0.02 g of dictamnine (benzene eluates), 0.26 g skimmianine, and 0.15 g of evoxine (benzene-acetone eluates), and from the neutral fraction (16 g) 1.0 g of the substance with mp 117-118°C (benzene eluates) and 0.3 g of diphyllin (benzene-acetone eluates). All the compounds with the exception of the substance with mp 117-118°C were identified by direct comparison with authentic samples obtained previously from this plant species [1, 2].

The substance with mp 117-118°C, which was quantitatively the main component of the roots (0.15% of the weight of the dry roots), dissolved readily in chloroform and carbon tetrachloride and crystallized from methanol, and was insoluble in dilute acid while in concentrated acid it turned yellow. Its IR spectrum showed absorption maxima at (cm⁻¹) 2860-2970, 1730, 1635, 1600, 1470, 1445, 1375, 1295, 1265, 1245, 1225, 1200, 1160, 1120, 1075, 840, and 740, and its UV spectrum at (nm) 218, 284, 294, and 330. The PMR spectrum (CDCl₃) of this substance contained three pairs of doublets from six ortho-protons (5.62, 6.12, 6.61, 6.75, 7.11, and 7.38 ppm) and a sharp singlet from the protons of a gem-dimethyl group (1.40 ppm); in the mass spectrum there were the peaks of ions with m/z (%): 228 (M⁺, 25), 213 (100), and 185 (20). The spectral characteristics given for the substance isolated agree with the corresponding characteristics of the spectra of the known compound seselin [3, 4], which was first isolated from Skimmia repens Nakai [5] and S. japonica Thunb. [6]. This is the first time that pyranocoumarins (dimethylchromenes) have been detected in plants of the Haplophyllum genus.

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14-ACETYLNUDICAULIDINE AND 18-DEOXYLYCOCTONINE - NEW DITERPENE ALKALOIDS FROM Delphinium confusum

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We have investigated the epigeal part of <u>Delphinium confusum</u> gathered in the flowering stage in the Kusavlisai gorge, Shakhristan Pass (Tadzhik SSR). Chloroform extraction in a Soxhlet apparatus gave 0.54% of total alkaloids, from which we isolated delphatine [1], base (I) with the composition $C_{26}H_{41}NO_{7}$, mp 206-208°C (hexane-ether), base (II) with the composition $C_{25}H_{41}NO_{6}$, mp 73-75°C (hexane-ether), and base (III) with the composition $C_{27}-H_{43}NO_{8}$, mp 115-117°C (hexane-ether).

The IR spectrum (KBr) of (I) contained absorption bands at 1100, 1740, and 3400-3550 cm⁻¹. Its PMR spectrum (CDCl₃, δ -scale) showed the signals of a tertiary methyl group (0.85 ppm, 3H, s), of an aminoethyl group (1.00 ppm, 3H, t, J = 7 Hz), of an acetoxy group (2.1 ppm, 3H, s), of three methoxy groups (3.18, 3.26, 3.36 ppm, 3H each, singlets), and of a β -H at C-14 (4.7 ppm, 1H, t, J = 4.5 Hz).

The PMR and mass spectra of (K) were characteristic for such C_{19} -diterpene alkaloids. In the mass spectrum, the maximum peak was due to the M^+ - OCH₃ ion, which agrees with the presence of a methoxy group at C-1 [2].

The molecular peak had an intensity of $^{\sim}1\%$, and the peak of the M⁺ - CH₃ ion one of $^{\sim}30\%$. This ratio of the intensities of these peaks in the mass spectrum of (I) is characteristic for lycoctonine alkaloids having a diol system at C-7 and C-8 and methoxy groups at C-1 and C-6 [3].

The acetoxy group in (I) was present at C-14, as was confirmed by the position of H-14 β signal at 4.7 ppm [4]. The PMR spectrum also contained two one-proton broadened singlets at 3.87 and 3.81 ppm (W_{1/2} \approx 6 Hz), one of which was probably due to an α -proton geminal to a β -methoxy group at C-6 [5]. On the basis of the facts given above, structure (I) may be proposed for the alkaloid, from which it follows that the base (I) was the 14-acetyl derivative of nudicaulidine [6].

The IR spectrum of base (II) had absorption bands at 1100 and $3400-3600 \text{ cm}^{-1}$. Its PMR spectrum exhibited the signals of a tertiary methyl group (0.94 ppm, 3H, s), of an aminoethyl group (1.00 ppm, 3H, t, J=7 Hz), and of four methoxy groups (3.34 and 3.36 ppm, 3H each, singlets; 3.45, 6H, s).

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