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PULCHELLIN C FROM Inula britannica

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The sequiterpene lactone britanin has been isolated previously [1] from Inula britannica L., and from the variety chinensis of the species the sesquiterpene lactones tomentosin, ivalin, 4-epi-isoinuviscolide, gaillardin, and inuchinenolides A, B, and C [2].

We have investigated the sesquiterpene lactones of the epigeal part of this plant collected in June, 1984, close to the village of Astrakhanovka, Shemakhinskii raion. The air-dry epigeal part (700 g) of Inula britannica was extracted with acetone three times. The total extractive substances obtained (5 g) were dissolved in 15 ml of chloroform and chromatographed on a column (3 × 130 cm) of alumina. The column was eluted with hexane, hexane—chloroform, chloroform, chloroform—ethyl acetate, and ethyl acetate. The volume of each fraction was 200 ml. Fraction 35, eluted by ethyl acetate, was rechromatographed on a column (0.8 × 20 cm) of silica gel (L 40/100 μm, Chemapol) with chloroform. On standing, the chloroform eluates deposited crystals which were recrystallized from aqueous ethanol. This gave 15 mg of a white crystalline substance with the composition $C_{15}H_{20}O_4$ (M^+ 264), mp 200-201°C.

The IR spectrum of the substance had absorption bands of OH groups (3600, 3490 cm^{-1}), of the CO group of an α-methylene-γ-lactone ring (1760, 1670 cm^{-1}) and of a double bond (1660 cm^{-1}). The presence in the mass spectrum of the peaks of the ions M^+, 246 ($M - H_2O$) 231 ($M - H_2O - CH_3$), and 228 ($M - H_2O - H_2O$) permitted the assumption that this substance was a diol.

According to NMR spectra (δ scale) 0 — TMS, Bruker WH-250), both hydroxy groups were of secondary nature (d, 3.85 ppm, J = 10 Hz; sx, 3.55 ppm, $J_1 = J_2 = 10$ Hz, $J_3 = 5$ Hz). One-proton signals at 4.75, 5.26 and 5.64, and 6.18 ppm showed the presence in the molecule of

TABLE 1. Chemical Shifts of the Protons of the Compounds under Investigation and of Pulchellin C

Protons	Compounds under investigation		Pulchellin C in $CDCl_3$
	in DMSO-d_6	in $CDCl_3$	
H-2	—	sx, 3,55, $J_1=J_2=10$ Hz $J=5$ Hz	m, 3,5
H-3	—	d 3,85, $J=10$ Hz	d, 3,78, $J=9$Hz
H-7	m, 3,08	m, 3,00	m, 3,00
H-8	sx, 4,52, $J=5$, $J_2=$ $J_3=2$ Hz	sx, 4,52, $J_1=J_2=5$ Hz $J_3=2$ Hz	br.t, 4,52, $J=5$ Hz
H-13a	s, 5,75	s 5,64	d, 5,60, $J=1$ Hz
H-13b	s 5,96	s, 6,18	d, 6,18, $J=1$ Hz
H-14a	d, 5,16, $J=2$ Hz	s, 5,26	s, 5,26
H-14b	d, 4,70, $J=2$ Hz	s, 4,75	s, 4,71
CH_3-15	c, 0,68	s, 0,87	s, 0,82
—OH	d, 4,75, $J=5$ Hz		
—OH	d, 4,90, $J=5$ Hz		

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the lactone under investigation of two exocyclic methylene groups (at C-4 and C-11, respectively). The paramagnetic shift (4.75 ppm) of the signal of one of the protons of an exocyclic methylene group and a signal at 3.85 ppm of a gem-hydroxylic proton indicated that they were present on neighboring carbon atoms (at C-4 and C-3, respectively) [3]. The nature of the multiplicity and the SSCC values of the gem-hydroxylic protons also showed the presence of OH groups on neighboring carbons (C-3 and C-2).

On comparing the details of the PMR spectra given above and others with those of pulchellin C (Table 1) [4], we identified the lactone under investigation as pulchellin C, and this is the first time that it has been isolated from Inula britannica L.

The IR spectrum was taken on a UR-20 spectrometer in paraffin oil, the mass spectrum on a MS-EI instrument at an ionizing energy of 70 eV, and the NMR spectra on a Bruker H-250 MHz instrument (δ scale, 0 — TMS).

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TRITERPENE GLYCOSIDES OF Astragalus AND THEIR GENINS.

XXVIII. CYCLOARTANES OF Astragalus babatagi

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Continuing investigations of cycloartane methylsteroids and their glycosides of plants of the genus Astragalus, we have analyzed Astragalus babatagi M. Pop. (Leguminosae). The epigeal part of this plant did not contain the substances being sought. The air-dry comminuted roots (170 g) of A. babatagi collected on July 1, 1986, in the environs of the village of Tavabulak (Babatag, Tadzhik SSR) were exhaustively extracted with methanol. This gave 23.6 g (13.9% on the weight of the air-dry raw material) of extractive substances. Their treatment by the method described in [1] gave 14 g of purified material which was chromatographed on a column of type L silica gel (Czechoslovakia). The column was eluted successively with chloroform and with the chloroform—methanol (15:1) and chloform—methanol—water (70:12:1) systems. Five fractions containing individual compounds of glycosidic nature were collected and these compounds were designated in order of increasing polarity as substances (1)-(5).

Substance (1) (10 mg — 0.006%), mp 276-279°C (from methanol), $[\alpha]_D^{24}$ −36 ± 2° (c 0.8; pyridine), was identified as β-sitosterol β-D-glucopyranoside [2].

Fraction 2 was rechromatographed on a column with elution by the ethyl acetate—methanol (15:1) system. This gave 1.177 g (0.7%) of substance (2), mp 229-230°C (from methanol), $[\alpha]_D^{24}$ −21.2 ± 2° (c 1.04; methanol), which was identified as cyclosieversioside A [3, 4].

Fractions 3 and 4 were rechromatographed on a column with elution by the chloroform—methanol—water (140:14:1) system. This led to the isolation of 270 mg (0.16%) of substance (3) and 170 mg [0.1% of substance (4)].

Substance (3), mp 185-188°C (from ethanol), $[\alpha]_D^{24}$ +16.6 ± 2° (c 0.84; methanol) was identical with cyclosieversioside B [4, 5].

Substance (4), mp 253-255°C (from methanol), $[\alpha]_D^{24}$ +20.7 ± 2° (c 1.16; methanol) was identified as cyclosieversioside C [3, 4].

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