PHYTOECDYSTEROIDS FROM FIVE SPECIES OF THE GENUS Silene

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Herein we communicate results from a study of five species of the genus *Silene* that have been introduced to the gray—oasis soil of the experimental plot of the ICPS AS RU.

Several principal ecdysteroids were found in the plants using TLC with vanillin— H_2SO_4 and column chromatography. These included ecdysterone (1), polipodin B (2), 2-deoxyecdysteron-22-*O*-benzoate (3), integristerone A (4), ecdysteron-22-*O*-benzoate (5), tomentesterone A (6), tomentesterone B (7), and 2-deoxyecdysterone (8), which were found in *S. tomentella* (6-8), *S. viridiflora* (1, 2, 4, 5), *S. nutans* (1-5), *S. tatarica* (1, 2, 4), and *S. wallichiana* (1, 4, 5, 8) [1-5]. Plant seeds were obtained from the Botanical Garden of the Republic of Uzbekistan.



1: $R_1 = R_2 = R_3 = R_4 = H$; **2**: $R_1 = R_3 = R_4 = H$, $R_2 = OH$; **3**: $R_1 = OH$, $R_2 = COC_6H_5$, $R_3 = H$ **4**: $R_1 = OH$, $R_2 = R_3 = R_4 = H$; **5**: $R_1 = R_2 = R_4 = H$, $R_3 = COC_6H_5$; **6**: $R_1 = H$, $R_2 = Ac$, $R_3 = COC_6H_5$ **7**: $R_1 = R_2 = H$, $R_3 = COC_6H_5$; **8**: $R_1 = OH$, $R_2 = R_3 = H$

The study of the aerial part of *S. nutans* isolated both **1** and **3**. Absorptions in the IR spectrum at 1705 and 1285 cm⁻¹ in combination with bands typical of a benzene ring (1610, 1587, 730) suggested that **3** was an aromatic ester. This was also consistent with five aromatic protons at 7.53 ppm (3H) and 8.28 (2H) in the PMR spectrum in addition to strong peaks in the mass spectrum for ions with m/z 122 (C₇H₆O₂), 105 (C₇H₅O), and 77 (C₆H₅), which are characteristic of benzoic acid.

The neutral fraction from base saponification of **3** contained 2-deoxyecdysterone (**8**); the acidic fraction, benzoic acid. Compared with the PMR spectrum of **3**, that of **8** showed a significant strong-field shift for CH_3 -21 (Table 1). This suggested that benzoic acid in **3** was esterified at the C-22 hydroxyl.

Comparison of the proton chemical shifts for 8 and 3 showed that the signal for C-22 was also shifted. This was consistent with bonding of the benzoic acid to the C-22 hydroxyl. An analogous strong-field shift was noted for ecdysteron-22-*O*-benzoate (5) (Table 1).

Thus, **3** is 2-deoxyecdysteron-22-O-benzoate. This compound was isolated earlier from *S. tatarica* [6, 7] but was isolated from *S. nutans* for the first time.

The known compounds tomentesterone A and B were isolated from *S. tomentella*. 2-Deoxyecdysterone was isolated from this plant for the first time.

The isolated pure ecdysteroids were identified using IR, PMR, and mass spectroscopy and by comparison with standard samples.

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TABLE 1. Proton Chemical Shifts of Ecdysterone (1), 2-Deoxyecdysteron-22-*O*-benzoate (3), Ecdysteron-22-*O*-benzoate (5), and 2-Deoxyecdysterone (8) (C_5D_5N , δ , ppm, 0 = HMDS)

C atom	Compound			
	1	3	5	8
H-2	1.00	-	1.00	-
H-3	4.30	1.11	1.30	1.11
H-7	6.21	6.25	6.22	6.22
H-9	3.57	3.38	3.60	3.52
H-22	3.81	3.77	3.76	3.85
CH ₃ -18	1.19	1.23	1.21	1.22
CH ₃ -19	1.06	1.07	1.10	1.05
CH ₃ -21	1.55	1.81	1.78	1.58
CH ₃ -26/27	1.34	1.32	1.32	1.38
Aromatic protons	-	7.53 (3H),	7.10 (3H),	-
-		8.28 (2H)	8.27 (2H)	

Signals for methyl protons appear as singlets; H-7 in 1, as a doublet with J = 1 Hz, in the others, as a broad singlet; H-2, H-3, and H-9, as multiplets.

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