A phytoecdysteroid, 2-deoxyecdysterone 3-acetate, has been isolated from the epigeal organs of *Silene praemixta* M. pop.

We have previously detected in the plant *Silene praemixta* M. Pop (family Caryophyllaceae) ecdysonone, 2-deoxy-α-ecdysone, 2-deoxyecdysterone, silenosterone, and premixisterone [1].

Rechromatography on a column of silica gel of the mother liquors obtained in the isolation of the substances mentioned led to the isolation of viticosterone E (IV) [2, 3], α-ecdysone (V) [4, 5], and a new phytoecdysteroid (II) with the composition C29H46O7. In the IR spectrum of compound (II), in addition to the absorption due to hydroxy groups (3450 cm⁻¹) and an α,β-unsaturated keto grouping (1655 cm⁻¹), there were absorption bands at 1740 and 1255 cm⁻¹ showing the presence of an ester residue. This was also indicated by the presence of a three-proton singlet at 2.00 ppm in the PMR spectrum of the ecdysteroid (II).

A peak with m/z 389 (cleavage of the C-20–C-22 bond) and its derivatives with m/z 371, 329, and 311 permitted the assumption that compound (II) belonged to the 2-deoxyecdysteroid group and had an acetyl residue in the steroid nucleus [6].

The characteristics of the PMR spectra of the ecdysteroid (II) and of 2-deoxyecdysterone (I) were close, with the exception of the chemical shifts of the resonance lines of the protons at C-3 and C-19. In the PMR spectra of compounds (II) and (I) (the corresponding values are given in parentheses), signals at 5.03 (4.14) ppm and 1.02 (1.07) ppm corresponded to

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\begin{aligned}
\text{I. } & R_1 = R_2 = R_4 = R_5 = \text{H; } R_3 = \text{OH} \\
\text{II. } & R_1 = R_2 = R_4 = \text{H; } R_3 = \text{OH; } R_5 = \text{Ac} \\
\text{III. } & R_1 = R_5 = \text{H; } R_3 = \text{OH; } R_2 = R_4 = \text{Ac} \\
\text{IV. } & R_2 = R_3 = \text{H; } R_4 = R_5 = \text{H; } R_1 = \text{OH} \\
\text{V. } & R_2 = R_3 = \text{H; } R_4 = R_5 = \text{H; } R_1 = \text{OH}
\end{aligned}
\]
these protons. Consequently, the signal of the C-3 proton had undergone a downfield shift by 0.89 ppm. This could take place under the influence an acetyl group located on the same carbon atom.

We also investigated the $^{13}$C NMR spectra of 2-deoxyecdysterone (I) and the ecdysteroid (II).

It can be seen from Table 1 that the values of the chemical shifts of the carbon atoms of compounds (I) and (II) are identical, with the exception of the C-1-C-6 group of atoms. On passing from compound (I) to (II), the signal of the C-3 carbon atom undergoes a paramagnetic shift by +4.2 ppm, while the resonance lines of the C-2, C-4, and C-6 atoms shift upfield by –3.7, –3.2, and –1.7 ppm, respectively. The chemical shifts of the C-1 and C-5 carbon atoms, experiencing the $\gamma$-influence of the substituent at C-3, also change. These facts are in harmony with the assumption expressed above that the acetyl group in ecdysteroid (II) is located at C-3 [7-9].

For confirmation, we subjected 2-deoxyecdysterone (I) to selective acetylation with acetic anhydride in pyridine. From the reaction mixture we isolated the known 2-deoxyecdysterone 3,22-diacetate (III) [1] and a substance identified as compound (II).

Thus, ecdysteroid (II) is 2-deoxyecdysterone 3-acetate.

**EXPERIMENTAL**

PMR spectra were obtained on a spectrometer with a working frequency of 300 MHz in $\text{C}_6\text{D}_5\text{N}$ ($\delta$, $0 - \text{TMS}$) and $^{13}$C NMR spectra on a SFT-20 instrument (Varian) in $\text{C}_6\text{D}_5\text{N}$ ($0 - \text{TMS}$). The assignment of the signals of the carbon atoms was made by a comparative study of the spectra recorded under the conditions of complete and partial decoupling from protons, and on the basis of literature information for ecdysterone [7, 8]. For further details, see [1].

**Isolation of the Ecdysteroids.** The mother liquors obtained in the isolation and recrystallization of the ecdysteroids obtained previously that were isolated from 6 kg of Silene praeform [1] were combined and chromatographed on a column of silica gel. Elution was performed with the chloroform-methanol (25:1) system, which gave 120 mg of 2-deoxyecdysterone 3-acetate (II) (0.002% on the weight of the air-dry raw material).

The subsequent washing of the column with the chloroform-ethanol (15:1) system yielded 100 mg (0.0017%) of viticosterone E (IV), $\text{C}_{29}\text{H}_{46}\text{O}_8$, mp 194–196°C (from acetone), $\gamma$-[c 0.52; methanol].

The use of chloroform-ethanol (9:1) for the elution of the column led to 1.5 g (0.025%) of the ecdysteroid (V), $\text{C}_{27}\text{H}_{45}\text{O}_6$, mp 236–238°C (methanol-water), $\gamma$-[c 0.83; methanol], identified as a-ecdysone [5].

2-Deoxyecdysterone 3-acetate (II), $\text{C}_{29}\text{H}_{46}\text{O}_7$, mp 150–152°C (methanol-benzene), $\gamma$-[c 0.70; methanol], $\lambda_{	ext{max}}$ $\text{C}_2\text{H}_5\text{OH}$ 246 nm ($\varepsilon$ 4.01); $\lambda_{	ext{max}}$ $\text{KBr}$ cm$^{-1}$: 3450 (OH), 1655 ($\Delta^7$-keto grouping), 1740, 1255 (ester group); CD (c 0.10; methanol); $\Delta\varepsilon = 1.93$ (253 nm); $\Delta\varepsilon = 1.34$ (327 nm).