ECDYSTERONE FROM Serratula sogdiana

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The inflorescences of Serratula sogdiana Bge., family Compositae, collected in the Shakhimardan region of the Fergana oblast in June, 1970 were extracted with hot ethanol. According to thin-layer chromatography [silica gel, chloroform–ethanol (4:1) system], the extract contained at least five phytoecdysones, with R_f 0.16, 0.31, 0.40, 0.60, and 0.81.

When the ethanolic extract was chromatographed on a column of silica gel in the chloroform–ethanol (9:1) system, compound (I), $C_{27}H_{44}O_7$, was isolated (with a yield of 0.52%); it had mp 242-244°C (anhydrous acetone), $[\alpha]_D$ + 59.8° (c 1.31; CH_3OH), R_f 0.55–0.60 $\lambda_{max}^{C_2H_5OH}$ 246 nm (log ε 4.40), ν_{max}^{KBr} 3330-3470 (OH), 1650 ($COCH = C <$) cm^{-1}.

The mass spectrum of (I) (170°C, 40 V) lacked a molecular peak with m/e 480, but had peaks with m/e 462, 444, 426, 411, 408, 393, 375, 363, 358, 352, 345, 344, 328, 327, 320, 301, 300 and with m/e 99, 81, which corresponds to the fragmentation of ecdysterone – an insect-molting hormone [1-4].

The nature of the optical rotatory dispersion curve, having a positive Cotton effect with an amplitude of $[\alpha]_{357}$ +91.5° (CH_3OH) corresponds to the cis-A/B linkage of 14α-hydroxy-7-en-6-one compounds of the ecdysone series [5].

Compound (I) was acetylated with acetic anhydride in pyridine (40°C, 4 h). The reaction product was separated on a column of silica gel. Elution with chloroform and with chloroform–ethanol (19:1) gave a tetraacetate of substance (I), $C_{35}H_{52}O_{11}$, mp 204-206°C (acetone–hexane), $[\alpha]_D$ +59.6° (c 0.89; CH_3OH), and a triacetate $C_{33}H_{50}O_{10}$ with mp 196-198°C (ether), $[\alpha]_D$ +57.7° (c 0.75; CH_3OH) [2]. When (I) was treated with anhydrous acetone in the presence of phosphatomolybdic acid (room temperature, 2 h), with subsequent separation in a thin layer of silica gel in the chloroform–ethanol (19:1) system, a diacetonide of (I), $C_{33}H_{52}O_7$, was obtained with mp 232-233.5°C (ether–hexane), $[\alpha]_D$ +40.5 (c 0.88: CH_3OH) [2].

The identity of (I) with ecdysterone was also confirmed by its NMR spectra (C_5D_5N at 100 MHz with HMDS as internal standard, δ scale) – 0.95 (3H at C_{19}, s), 1.08 (3H at C_{18}, s), 1.45 (3H at C_{21}, s), 1.26 (6H at C_{26} and C_{27}, s), 6.07 (H at C_7) – and of its triacetate ($CDCl_3$) – 0.80 (3H at C_{18}, s), 0.98 (3H at C_{19}, s), 1.21 (3H at C_{21}, s), 1.14 (3H at C_{26}, s), 1.17 (3H at C_{27}, s), and 5.80 (H at C_7) [2, 3, 6].

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