THE ISOLATION OF ECDYSTERONE

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By the extraction of the racemes of Serratula inermis Gilib. with hot methanol and subsequent chromatography on polyamide, we have isolated (about 2% of the air-dry weight of the racemes) a compound $C_{27}H_{44}O_7$ with a double mp, 153–157° C and 236–237° C (from water), $[\alpha]_D^{20} + 55.9°$ (c 0.34; CH_3OH), mp 243–244° C (from dry acetone), IR spectrum (tablets with KBr): 3420, 1655, and 1610 cm^{-1}. UV spectrum (CH_3OH): λ_{max} 242 mμ (ε 15 750). This compound forms a triacetate $C_{33}H_{50}O_{10}$ with mp 192–193.5° C (from ether). UV spectrum: 3446, 1740, 1658 and 1246 cm^{-1}.

When compound I was treated with acetone and anhydrous $CuSO_4$, a diacetonide $C_{33}H_{52}O_7$ with mp 229–231° C (from cyclohexane–ether) was formed. When I was heated with 5% aqueous ethanolic HCl or H_2SO_4 for 10–30 min, a mixture of substances was obtained the UV spectrum of which had two absorption maxima at 248 and 295 mμ. This behavior is characteristic of compounds of the ecdysone series, which are converted under the action of acids into a mixture of the corresponding 8,14- and 7,14-dien-6-ones [1].

The mass spectrum of compound I contained the molecular peak with m/e 480 and also peaks with 468, 444, 411, 408, 393, 375, 363, 356, 352, 346, 344, 328, 327, 310, 300 and with m/e 99 and 81, which corresponds to the fragmentation of the recently-isolated ecdysterone [2–5]. The optical rotatory dispersion curve for I is characterized by a positive Cotton effect with an amplitude $[\alpha]_{340}+55.3°$ (ethanol), which corresponds to the cis-A/B linkage of 14α-hydroxy compounds of the ecdysone series [6]. The nuclear magnetic resonance spectra for compound I (pyridine) (1.21, 1.07, 1.57, 1.38) and its triacetate ($CDCl_3$) (0.88, 1.05, 1.25, 1.23, and 1.20 ppm) also coincide with the NMR spectra of ecdysterone and its triacetate. On this basis, we assume that compound I is identical with ecdysterone [2–5].

The roots of Serratula inermis contain practically no ecdysterone, while in the leaves and stems of the plant collected in the flowering period 0.25 and 0.01% of ecdysterone, respectively, was found.

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