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STACHYSTERONE D, VITICOSTERONE E AND α-ECDYSONE FROM Lychnis chalcedonica

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UDC 547.926

We have previously reported the isolation from the epigeal part of Maltese cross campion *Lychnis chalcedonica* L., family Caryophyllaceae, of ecdysterone, polypodin B, 24(28)-dihydromakisterone, and integristerone A [1].

On rechromatography of the fractions of weakly polar compounds [SiO_2, chloroform–methanol: 1) (15:1); 2) (9:1)] three ecdysteroids were isolated. Their yields, calculated on the air-dry raw material were: for (I) – 0.0004; (II) – 0.0001; (III) – 0.0005%.

Substance (I), $C_{27}H_{42}O_6$, mp 255-257°C (ethyl acetate–methanol).

PMR spectrum (C_5D_5N, 0 – HMDS, δ, ppm): 0.95 (CH_3-18/19, s), 1.10 (CH_3-26/27, s), 1.28 (CH_3-21, s); 3.92-4.10 (H-2, 3 and H-22, m), 3.42 (H-9, t), 6.06 (H-7, br.s).

Mass spectrum, m/z: 462 (M^+), 444, 426, 406, 363, 345, 327, 309, 301, 279, 161, 125, 99, 81, 55.

Compound (I) was identified from its spectral characteristics as stachysterone D [2-4].

Substance (II), $C_{29}H_{46}O_8$, mp 196-198°C (acetone), $[\alpha]_D^{20}$ +58.9 ± 2° (c 0.72; methanol), ν_{max}^{KBr}, cm^{-1}: 3430 (OH), 1670 (Δ^7-6-keto grouping), 1730, 1275 (ester grouping).

PMR spectrum (C_5D_5N, 0 – HMDS, δ, ppm): 0.94 (CH_3-19, s), 1.08 (CH_3-18, s), 1.30, 1.36 (CH_3-26/27, s), 1.48 (CH_3-21, s), 1.80 (OAc), 3.46 (H-9, m), 3.68 (H-22, m), 4.08 (H-2, 3, m), 6.06 (H-7, br.s).

Mass spectrum, m/z: 522 (M^+), 462, 444, 426, 408, 411, 393, 375, 363, 301, 99, 81.

Compound (II) was identified from its spectral indices and by a direct TLC comparison (system 2) with an authentic sample as viticosterone E [5, 6].

Substance (III), $C_{27}H_{44}O_6$, mp 236-238°C (ethyl acetate–methanol), $[\alpha]_D^{20}$ +63.0 ± 2° (c 0.65; methanol).

PMR spectrum (C_5D_5N, 0 – HMDS, δ, ppm): 0.59 (CH_3-18, s), 0.94 (CH_3-19, s), 1.14 (CH_3-21, d, ^3J = 6 Hz), 1.24 (CH_3-26/27, s), 3.36 (H-9, m), 3.82 (H-2, 22, m), 4.06 (H-3, m), 6.02 (H-7, br.s).

Mass spectrum, m/z: 464 (M^+), 446, 431, 428, 413, 410, 395, 377, 348, 330, 315, 300, 126, 117, 109, 99, 81.

From its spectral characteristics and also by a direct comparison with an authentic sample, compound (III) was identified as α-ecdysone [7, 8].

Siberian Botanical Garden, Tomsk State University. Institute of the Chemistry of Plant Substances, Academy of Sciences of the Uzbek SSR, Tashkent. Translated from Khimiya Prirodnykh Soedinenii, No. 4, pp. 585-586, July-August, 1991. Original article submitted February 5, 1991.

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STEROID GLYCOSIDES FROM Solanum tuberosum SEEDS

TUBEROSIDES C AND D

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UDC 547.918+547.917

No investigations of the presence of steroid glycosides in the seeds of Solanum tuberosum L. have previously been made. In the present communication we give information on the isolation and determination of the structures of steroid glycosides from potato seeds, which we have called tuberosides C and D.

After the elimination of alkaloids by precipitation with ammonia, a methanolic extract was subjected to repeated chromatography on a column of silica gel, as a result of which individual glycosides were obtained. The separation was monitored by thin-layer chromatography in the chloroform—methanol—water (65:35:10, lower layer) system.

Tuberoside C (I), with mp 236-238°C, $[\alpha]_D^{20}$ −63° (c 1.7; Py) and tuberoside D (II), with mp 266°C, $[\alpha]_D^{20}$ −70°, gave positive reactions with the Sannié reagent and negative reactions with the Ehrlich reagent [2], which permitted them to be assigned to the spirostanol glycosides. Their IR spectra contained absorption bands at 845, 890 < 920, and 980 cm^{-1}, which are characteristic for spiroketal chains of the (25S)-series.

On complete acid hydrolysis a single aglycon was isolated, which was identified as yamogenin, the identification being confirmed by physicochemical methods (mp 201°C; $[\alpha]_D^{20}$ (c 1.0; $CHCl_3$), $[M^+]$ 414).

With the aid of gas—liquid chromatography of the acetates of the aldononitrile derivatives of the sugars [3] the presence of the following monosaccharides in the given ratios was established: for tuberoside C — rhamnose and galactose (1:1); and for tuberoside D — rhamnose and glucose (2:1).

By means of partial hydrolysis a number of progenins were obtained, and these were investigated by physicochemical methods. For tuberoside C — a progenin with mp 230-233°C, $[\alpha]_D^{20}$ −91° (c 1.0; CH_3OH); for tuberoside D — progenins with mp 264°C, $[\alpha]_D^{20}$ −97° (c 1.0; CH_3OH) — yamogenin glucopyranoside — and with mp 241-243°C, $[\alpha]_D^{20}$ −57° (Py) — yamogenin rhamnoglucopyranoside [2].

The positions of attachment of the carbohydrate units to one another were revealed by the methanolysis of permethylates of tuberosides C and D, obtained by Hakomori's method [4]. By GLC in the presence of markers, for glycoside C we identified methyl 2,3,4-tri-O-methyl-

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Institute of Ecological Genetics of the Moldavian SSR, Kishinev. Translated from Khimiya Prirodnykh Soedinenii, No. 4, pp. 586-587, July-August, 1991. Original article submitted June 11, 1990; revision submitted January 10, 1991.

0009-3130/91/2704-0515$12.50