A comparison of the spectral characteristics of the isomers of diol (IV) obtained in this way with the described sample of (I) revealed agreement with the less polar isomer. Unfortunately, the absence of information on the configuration of the C-24 center of genin (I) in [1] does not permit a definitive conclusion to be drawn at the present time on the stereochemistry of this center in the hydroxyketones (IV). An x-ray structural study of the individual isomers of (IV) that we are currently conducting will permit an unambiguous answer to this question.

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PHYTOECDYSTEROIDS OF Aerva lanata

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UDC 547.926

Aerva lanata Juss., family Amaranthaceae, is a biennial tropical herbaceous plant. In folk medicine [1, 2], it is used as an anthelminthic, diuretic, and emmolient. It does not grow on the territory of the CIS. Work is being done on introducing it into cultivation [3, 6].

Aiyar et al. [4] have isolated from Aerva lanata α-amyrin, β-sitosterol, and sitosterol palmitate. In a study of the phenolic components of the herb A. lanata, flavonoids [2], β-sitosterol, daucosterol, syringic acid, vanillic acid, feruloyltyramine, and feruloylhomovanillylamine [5] have been found. The carbohydrate complex of the epigeal part of the plant includes water-soluble polysaccharides, an acidic polysaccharide, starch, and hemicellulose.

The first representative of this genus to be investigated for the presence of ecdysteroids was Aerva tomentosa Forssk. [7]. The amount of ecdysteroids in the seeds of this plant was determined by quantitative analysis as 0.03%. By column chromatography, the butanol fraction of the total extractive substances gave ecdysterone in 0.007% yield [7].

We have studied the phytoecdysteroid content of specimens of the herb A. lanata grown in the introduction section of the F. N. Rusanov Botanical Garden of the Uzbek Academy of Sciences.

The air-dry raw material (1030 g) was extracted successively with hexane, chloroform, ethyl acetate, and methanol. Separation of the methanol fraction on a column of silica gel in the chloroform–methanol (9:1) system yielded 3 mg (0.00028%) of a substance which was identified on the basis of physicochemical characteristics and a direct comparison as ecdysterone. When elution of the column with the above solvent system was continued, another two substances were detected the nature of which it has not yet been possible to establish.

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AGLYCONS FROM GLYCOSIDES OF THE STARFISH Lethasterias nanimensis chelifera

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UDC 547.925:593.93

Continuing investigations of steroid metabolites of the Far Eastern starfish Lethasterias nanimensis chelifera, we have isolated the total glycoside fraction from an ethanolic extract of the animals by a known method [1]. Acid hydrolysis of the total glycosides (6.0 g) was performed with 2 N HCl at 100°C for 0.5 h. The aglycons were extracted with chloroform, the extract was evaporated, and the residue was chromatographed twice on a column of silica gel in chloroform–ethanol (18:1) and chloroform–ethyl acetate mixtures with a ratio changing gradually from 30:1 to 30:15. Two main fractions of aglycons were obtained: A (1.200 g) and B (0.420 g).

Fraction A was chromatographed again on a column of silica gel in the chloroform–ethyl acetate system, beginning with a ratio of 30:1 and gradually increasing the proportion of ethyl acetate to 100%. Further purification was achieved by high-performance liquid chromatography (Du Pont chromatograph, refractometer as detector) on a Zorbax ODS (5 μm, 4.6 × 250 mm) column with ethanol–water (63:37) as eluent and a Separon SGX (5 μm, 3 × 150 mm) column with chloroform–ethyl acetate (1:1) as eluent. The individual compounds were identified with known aglycons for starfish by comparison of their PMR and ^{13}C NMR spectra with those given in the literature, and also by the GLC-mass spectrometry of their acetates. We isolated 120 mg of 3β,6α-dihydroxy-5α-pregnen-9(11)-en-20-one (asterone, I) [2], 3 mg of the 17α- isomer of asterone (II) [3], 35 mg of (20E)-3β,6α-dihydroxy-5α-cholesta-9(11),20(22)-diene-23-one (isomarthasterone, III) [2], and 16 mg of (17E)- and (17Z)-3β,6α-dihydroxy-5α-cholesta-9(11),17(20)-dien-23-ones (VI) [2].

Fraction B was crystallized from ethanol–chloroform, to give 200 mg of aglycon (V), with mp 240-242°C, $[\alpha]_{Hg}$ +56.6° (c 0.24; ethanol). This aglycon was identified as 3β,6α,23ξ-trihydroxy-5α-cholest-9(11)-ene on the basis of the following spectral characteristics.

PMR spectrum (C_5D_5N, 250 MHz): 0.73 (s, CH_3-18); 1.02 (d, J = 6.0 Hz, CH_3-27); 1.04 (d, J = 6.0 Hz, CH_3-26); 1.09 (s, CH_3-19); 1.16 (d, J = 6.0 Hz, CH_3-21); 3.08 (dm, J = 10.8 Hz, H-4e); 3.93 (m, H-3, H-6); 4.10 (m, 1/2W = 19.2 Hz, H-23); 5.41 (d, J - 6.0 Hz, H-11). The recording of the nuclear Overhauser effect on irradiation of the multiplet at 4.10 ppm showed an enhancement of the H-21 and H-26 signals, which demonstrated the presence of a hydroxy substituent at C-23.

^{13}C NMR spectrum (C_5D_5N, 62.9 MHz): 36.6 (C-1); 32.5 (C-2); 70.9 (C-3); 34.1 (C-4); 50.9 (C-5); 68.6 (C-6); 43.7 (C-7); 36.2 (C-8); 116.3 (C-9); 38.7 (C-10); 146.7 (C-11); 42.3 (C-12); 41.5 (C-13); 54.0 (C-14); 25.7 (C-15); 28.9 (C-16); 57.7 (C-17); 11.8 (C-18); 19.7 (C-19); 34.3 (C-20); 19.8 (C-21); 46.2 (C-22); 68.0 (C-23); 47.6 (C-24); 25.0 (C-25); 24.1 (C-26); 22.2 (C-27). The assignment of the signals in the ^{13}C NMR spectrum of (V) was made on the basis of an experiment with incomplete decoupling from protons and by comparison with the corresponding spectra of aglycons (I) and (III).

Aglycon (V) has been isolated previously from a hydrolysate of the total asterosaponins of the starfish Asterias amurensis [4]. However, it was not possible to compare the characteristics of the compound that we had isolated with literature values since they were not given in the Japanese authors' paper.

Pacific Ocean Institute of Bioorganic Chemistry, Far Eastern Branch, Russian Academy of Sciences, Vladivostok. Translated from Khimiya Prirodnykh Soedinenii, No. 1, pp. 145-146, January-February, 1992. Original article submitted April 12, 1991.

 0009-3130/92/2801-0124$12.50