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STEROID HEXAOL FROM Crossaster papposus

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From an ethanolic extract of the starfish Crossaster papposus by chromatography on Polikhrom-1, silica gel, and Florisil we have isolated a steroid polyol (I). On the basis of spectral characteristics, the structure of 5α-cholestane-3β,6β,8,15α,16β,26-hexaol has been established for (I).

Substance (I), $C_{27}H_{48}O_6$, mp 253-255°C (from chloroform–ethanol), $[\alpha]_{Hg}^{20}$ +40° (C 0.3; methanol) was obtained with a yield of 0.001% on the weight of a lyophilizate of an ethanolic extract of the raw material. The animals were collected in August, 1983, in the Sea of Okhotsk in the littoral of the island of Onekotan (Kurile Islands) from a depth of 100 m. Mass spectrum (m/z, %): 468 (4; M^+); 450 (100); 432 (60); 414 (43); 399 (16); 396 (19); 331 (11); 321 (51); 303 (51); 285 (38); 225 (95); 107 (41). PMR spectrum (C_5D_5N, 250 MHz, δ, TMS, ppm): 1.09 (d, J = 6.7 Hz, CH_3-27); 1.15 (d, J = 7.0 Hz, CH_3-21); 1.45 (dt, J_1 = 2.0 Hz, J_2 = 2.0 Hz, J_3 = 12.0 Hz, H-5); 1.57 (d, J = 10.5 Hz, H-14); 1.60 (s, CH_3-19), 1.60 (dd, J_1 = 7.2 Hz, J_2 = 10.5 Hz, H-17); 1.77 (s, CH_3-18); 2.00 (dm, H-4e); 2.10 (dd, J_1 = 2.5 Hz, J_2 = 14.5 Hz, H-7a); 2.45 (q, J = 11.5 Hz, H-4a); 3.28 (dd, J_1 = 3.0 Hz, J_2 = 14.5 Hz, H-7e); 3.64 (Add, J_1 = 6.2 Hz, J_2 = 10.2 Hz, H-26); 3.77 (Bdd, J_1 = 5.5 Hz, J_2 = 10.2 Hz, H-26'); 4.02 (m, H-3); 4.24 (q, J = 2.2 Hz, H-6); 4.76 (dd, J_1 = 2.0 Hz, J_2 = 7.2 Hz, H-16); 5.06 (dd, J_1 = 2.0 Hz, J_2 = 10.5 Hz, H-15).

^{13}C NMR spectrum (C_5D_5N, 62.9 MHz, δ, TMS, ppm): 40.9 (C-1); 32.1 (C-2); 71.2 (C-3); 37.0 (C-4); 48.6 (C-5); 73.2 (C-6); 45.5 (C-7); 76.1 (C-8); 56.6 (C-9); 36.2 (C-10); 19.3 (C-11); 42.7 (C-12); 44.8 (C-13); 63.9 (C-14); 80.5 (C-15); 82.4 (C-16); 60.3 (C-17); 17.0 (C-18); 15.9 (C-19); 30.0 (C-20); 18.4 (C-21); 36.7 (C-22); 24.4 (C-23); 34.5 (C-24); 36.7 (C-25); 67.5 (C-26); 17.4 (C-27).

The assignment of the signals in the PMR and ^{13}C NMR spectra of (I) was made on the basis of a comparison with the corresponding spectra of 5α-cholestane-3β,6α,8,15α,16β,26-hexaol (II) and asterosaponin P_1 (III) which we had isolated previously from the starfish Patiria pectinfera [1, 2]. The sequence of protons H-4–H-7 was established on the basis of the nature of the H-3 multiplet (4.02 ppm) by spin-decoupling experiments, and the sequence of proteins H-14, H-16, H-17 on the basis of the H-15 signal (5.06 ppm).

The configurations of the hydroxy groups were determined from the SSCCs of the protons. In the PMR spectrum of (I), the H-6 signal was present in the form of a narrow quartet, in contrast to the broad triplet of doublets for H-6 in the analogous spectrum of the steroid (II) [1]. The H-4a signal (2.45 ppm) was shifted downfield, and the H-4e signal (2.00 ppm) upfield, in comparison with the corresponding signals (1.86 and 3.15 ppm), respectively for compound (III) [2]. These facts indicated the β-configuration of the OH group at C-6.

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A steroid hexaol with a structure similar to that of (I) has recently been isolated from the starfish Sphaerodiscus placenta [3]. A comparison of the physicochemical constants and spectral characteristics of (I) with literature information on this steroid did not appear possible, since only the PMR spectrum (CD_3OD) is given for it.

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N-PHENYL-2-NAPHTHYLAMINE FROM Bupleurum aureum FLOWERS

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On studying the flavonoids of the flowers of Bupleurum aureum Fisch., we turned our attention to the fact that ethanolic extracts of them sometimes contain a substance of unestablished nature the amount of which varies during the flowering process. To free it from flavonoids, an alcoholic extract was subjected to chromatography on polyamide with elution by chloroform. The chloroform eluate, after concentration, was chromatographed twice on a column of silica gel which was also washed with chloroform. The combined fractions containing substance (I), after evaporation, were chromatographed on a column of silica gel using, in this case, petroleum ether–diethyl ether (9:1) as the eluent. After concentration of the fractions containing (I), crystals deposited.

Substance (I) – colorless needles with mp 107.5-108.5°C, composition $C_{16}H_{13}N$, on the basis of elementary analysis and mass spectrometry. The results of elementary analysis and also a comparison of the melting point and spectral characteristics (IR, mass, PMR spectra) with the literature [1-4] permitted substance (I) to be identified as N-phenyl-2-naphthylamine. There is information on the isolation of N-phenyl-2-naphthylamine from a number of plant sources [5, 6], but this is the first time that it has been isolated from plants of the genus Bupleurum.

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