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STEROID COMPOUNDS FROM OPHIUROIDS.

III. SULFATED STEROIDS FROM Gorgonocephalus caryi

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Continuing an investigation of physiologically active compounds from ophiuroids [1, 2], we have studied the composition of the polar steroids from an ethanolic extract of Gorgonocephalus caryi ("Gorgon's head") collected in the summer of 1988 on the Kashevarov bank in the Sea of Okhotsk from a depth of 160-170 m. By extraction of the dry residue with ethanol, column chromatography on silica gel in the chloroform–methanol–water (3:1:0.05) system with the addition of ammonia to pH 7-8, and HPLC [Ultrasphere-Si, 10 × 250 mm, 1 ml/min, methanol–1.6% aqueous solution of sodium dihydrogen phosphate (25:1)], followed by column chromatography on Sephadex LH-20 in methanol, we isolated compound (I). A positive qualitative Liebermann-Burchard reaction confirmed that it belonged to the steroid series. Solvolytic desulfation on heating in a mixture pyridine and dioxane and the IR spectrum (KBr, 1235, 1064 cm^{-1}) showed the presence of sulfate groups in its molecule. The ^1H and ^{13}C NMR spectra of (I) coincided with those for the cholest-5-ene-3α,4β,21-triol 3α,21-di(sodium sulfate) isolated previously from Ophiura sarsi [2]. Atomic absorption analysis showed the presence of sodium ions in (I) as the counter-ions to the sulfate groups.

In addition, by a method described previously [3], we isolated the sulfated steroid (II), the R_f value of which on TLC and the chemical shifts of the signals of the protons in its ^1H NMR spectrum coincided with the corresponding characteristics for cholesterol sulfate. The desulfation of (II) by heating in the pyridine–dioxane system gave cholesterol.

Thus two compounds known previously have been isolated from the far-eastern ophiuroid Gorgonocephalus caryi: cholest-5-ene-3α,4β,21-triol 3α,21-di(sodium sulfate) and cholesterol sulfate.

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