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SYNTHESIS OF THE SIDE CHAIN OF A SAPOGENIN FROM THE SOFT CORAL Asterospiculata randalli

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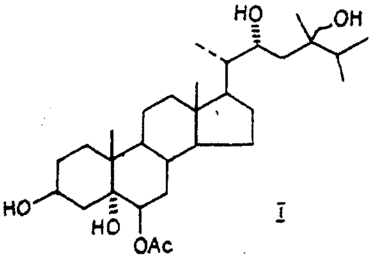
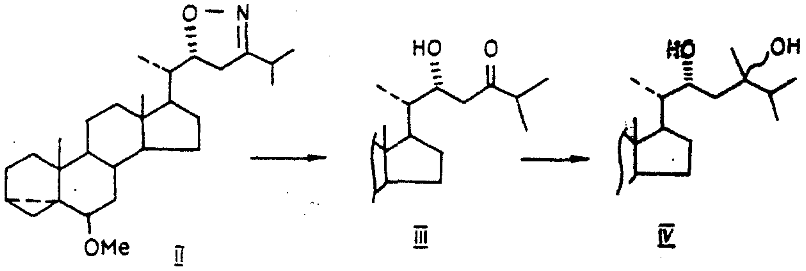
UDC 547.92

In 1984, the isolation from the soft coral *Asterospicularia randalli* of the polyhydroxylated steroid (I) with a set of hydroxy functions at C-22 and C-24 unusual for steroids of marine organisms was reported [1].



In a preceding paper, a method of constructing the side chain that is characteristic for this genin was proposed for the first time. The method is based on the use of a steroid isoxazoline (II) that we had obtained previously [2]. The reductive cleavage of the heterocyclic nucleus of compound (II) over Raney nickel in the presence of acids (H_3BO_3, AcOH, etc.) led to the formation of the 22R-hydroxy-24-oxo derivative (III). The formation of a β-ketone of this structure was shown by the appearance of the absorption bands of a C=O group (1710 cm^{-1}) and of a hydroxy group (3510 cm^{-1}) in the IR spectrum and the disappearance from the PMR spectrum of the signals of the methylene protons at C-23 characteristic for the isoxazoline ring. The interaction of the resulting compound (III) with methyllithium in ether at 0°C gave the desired product – the 22R,24ξ-diol (IV). It must be mentioned that we obtained both diastereomers of compound (IV) and separated them chromatographically. For the less polar isomer: PMR spectrum (200 MHz, δ, ppm): 0.75 s (3H, 18-Me), 0.85 d (3H, J = 7 Hz, 26-Me), 0.95 d (3H, J = 7 Hz, 21-Me), 0.98 d (3H, J = 7 Hz, 27-Me), 1.02 s (3H, 19-Me), 1.09 s (3H, 28-Me), 2.02 m (1H, C$_{25}$-H), 3.33 s (3H, OMe), 4.09 m (1H, C$_{22}$-H). IR spectrum (KBr, cm^{-1}): 3510, 1100. Mass spectrum (m/z): 446 [M]$^+$, 431 [M – Me]$^+$, 414 [M – MeOH]$^+$, 391, 353, 313, 284, 255. For the more polar isomer: PMR spectrum (200 MHz, δ, ppm): 0.75 s (3H, 18-Me), 0.86 d (3H, J = 7 Hz, 26-Me), 0.92 d (3H, J = 7 Hz, 27-Me), 0.95 d (3H, J = 7 Hz, 21-Me), 1.03 s (3H, 19-Me), 1.19 s (3H, 28-Me), 2.78 m (1H, C$_6$-H), 3.33 s (3H, OMe), 4.10 m (1H, C$_{22}$-H). IR spectrum (KBr, cm^{-1}): 3510, 1100. Mass spectrum (m/z): 446 [M]$^+$, 431 [M – Me]$^+$, 414 [MeOH]$^+$, 391, 353, 313, 284, 255.



Institute of Bioorganic Chemistry, Belorussian Academy of Sciences, Minsk. Translated from Khimiya Prirodnykh Soedinenii, No. 1, pp. 143-144, January-February, 1992. Original article submitted May 22, 1991.

0009-3130/92/2801-0122$12.50

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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