SYNTHESIS OF FUCOSTEROL β-D-GLUCOPYRANOSIDE

L. N. Atopkina, O. F. Smetanina,
T. A. Kuznetsova, and N. I. Uvarova

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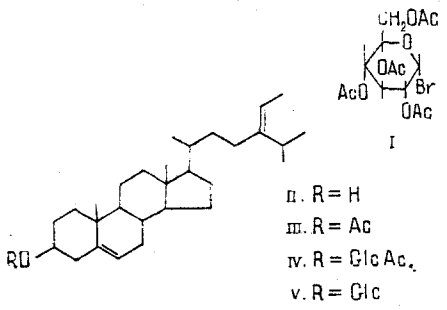
It is known that sterols and their derivatives possess hypocholesterolemic activity [1, 2]. In the search for more effective derivatives, we have performed the synthesis of fucosterol β-D-glucopyranoside (V).



Fucosterol (II) was isolated from the brown alga *Pelvetia wrightii* by the usual method [3, 4], and its constants and spectral characteristics corresponded to those given in the literature [3, 5].

The glycosylation of (II) was performed with 2,3,4,6-tetra-O-acetyl-α-D-glucopyranosyl bromide (I) in the presence of mercuric cyanide in absolute nitromethane at room temperature. The course of the reaction was monitored with the aid of TLC on silica gel in the hexane–acetone (3:1) and benzene–chloroform–methanol (6:4:1) system. By chromatography on a column of KSK silica gel in the hexane–acetone (40:1) system, we isolated 3-O-acetylfucosterol (III) (yield 21.2%), and fucosterol tetra-O-acetyl-β-D-glucopyranoside (IV) (yield 24.4.%). Compound (III) had mp 118°C (ethanol) and the ^{1}H NMR spectrum (δ, ppm): 0.68-1.02 (3 H × 4, s), 1.53 (3 H, d) — protons of methyl groups; 2.03 (3 H, s) — protons of an acetyl group; 4.65 (1 H, m, C_3-H); 5.12 (1 H, C_{28}-H); 5.29 (1 H, C_6-H). According to the literature [3]: mp 119°C.

Compound (IV): $C_{43}H_{66}O_{10}$, mp 148-150°C (ethanol), $[\alpha]_D^{20}$ −24.2° (c 1.0; chloroform). ^{1}H NMR spectrum (δ, ppm): 0.68 (3 H, s), 0.94 (3 H, s), 0.99 (3 H, s), 1.01 (3 H, s), 1.53 (3 H, d) — the protons of methyl groups; 2.00-2.07 (3 H×4, s) — the protons of acetyl groups; 4.58 (1 H, d, $J_{1,2}$ = 7.3 Hz) — anomeric proton (β configuration of the glycosidic bond).

Saponification of the tetraacetate (IV) with a catalytic amount of sodium methanolate in in methanol gave the free glycoside (V) with a yield of 71%. $C_{35}H_{58}O_6$, mp 250-260°C (decomp.) $[\alpha]_D^{20}$ −42.0° (c 1.0; pyridine).

LITERATURE CITED

1. T. Tabata, M. Tanaka, and T. Iio, Yakugaku Zasshi, 100, (5), 546 (1980).
2. F. Gasparini, A. Wolfson. R. Hockberg, and S. Lieberman, J. Biol. Chem., 25, 6650 (1979).
3. I. M. Heilbron, R. F. Phipers, and H. R. Wright, J. Chem. Soc., 1572 (1934).
4. B. A. Knights, Phytochemistry, 9, 903 (1970).
5. C. F. Gibbons, L. J. Goad, and T. W. Goodwin, Phytochemistry, 6, 677 (1967); 7, 983 (1968).

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