The percentage of DOCSA was determined from the formula

$$C = \frac{D \cdot 1250 \cdot C_0}{D_0 \cdot p \cdot l \cdot 1},$$

where D is the optical density of the solution being analyzed at 445 nm;

D_0 is the optical density of the solution of the standard sample of DOCSA;

C_0 is the concentration of the standard sample (0.000024 g/ml);

p is the weight of the sample, g; and

ℓ is the layer thickness, cm.

The results of the determination at p = 0.95 and n = 6 are given in the form of the following metrological characteristics: $\bar{X}$ = 99.93%, S = 0.4575, S_r = 0.00458, St = ±1.18, $\bar{X}$ ± St = 99.93 ± 1.18.

In comparison with known methods, the method developed is characterized by high sensitivity and simplicity of performance. The time of an analysis is 15-20 min.

A method has been developed on the basis of this procedure for the quantitative determination of DOCSA in 0.5% solution for injection.

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NEW APPROACH TO SYNTHESIS OF TRENBOLONE

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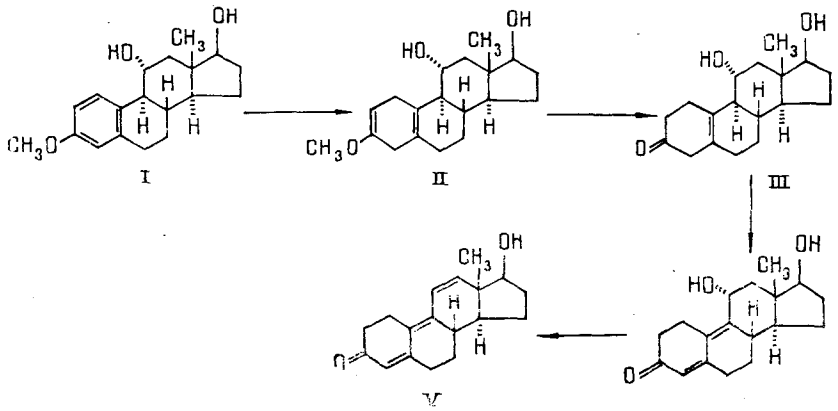
In recent years, the anabolic steroid 17β-hydroxyestra-4,9,11-trien-3-one (trenbolone), obtained by the scheme for the total synthesis of steroids proposed by Velluz [1] has been widely used in veterinary medicine. In the present paper we describe a new variant of the synthesis of trenbolone which is an extension of the Amanchenko-Torgov scheme for the total synthesis of esterone [2]. The key stage of the synthesis of trenbolone is the reduction of the known methyl ether of 11α-hydroxyestradiol [3] under the conditions of the Birch reaction [4].



I II III IV V

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For this purpose, the methyl ether (I) was treated with 44 equivalents of lithium in the presence of methanol in liquid ammonia at − 70°C, and the methoxydiene (II) was obtained (yield 68%). The hydrolysis of (II) with acetic acid in methanol led to the ketone (III) (yield 70%). The latter, on bromination-dehydrobromination with one equivalent of pyridine bromide-perbromide in pyridine formed the diene (IV), the dehydration of which with HCl in chloroform led to trenbolone (V).

17β-Hydroxyestra-4.9.11-trien-3-one (V): mp 184-186°C. UV spectrum, λ_{max}: 343 nm (log ε 4.41). IR spectrum (ν_{max}^{KBr}, cm^{-1}): 3350 (OH), 1640 (C=O): 1570, 1560, 1540 (C=C). PMR spectrum ($CDCl_3$, δ, ppm 0 − HMDS): 0.91 (s, 3H, CH_3), 3.90, (t, 1H, H-17), 5.78 (s, 1H, H-4), 6.42 and 6.47 (d, 1 H each, 1 H, J = 10 Hz, H-11 and H-12). Mass spectrum (m/z, %): 270 (M^+, 100), 258 (M−H_2O, 26).

The intermediate compounds (III) and (IV) exhibit anabolic effects.

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GLYCOSYLATION OF TRITERPENOIDS OF THE DAMMARANE SERIES.

VIII. DAMMARANE HYDROXYKETONE β-D-GLYCOPYRANOSIDES

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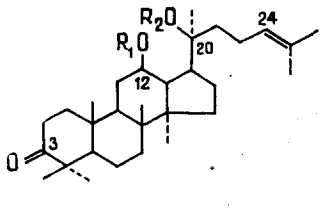
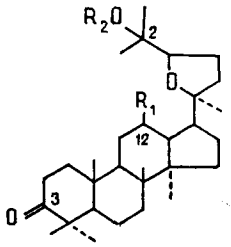
UDC 547.917+547.918+547.597

To study structure-activity relationships, we have obtained glucosides from one of the components of the triterpene fraction from birch leaves − 12β,20(s)-dihydroxydammar-24-en-3-one (I) [1] − and also of the 3-ketodammarane alcohols (II) and (III). Glycosylation was





I. R_1=R_2=H
II. R_1=OH; R_2=H
III. R_1=R_2=H
IV. R_1=Glc(OAc)$_4$; R_2=H
V. R_1=H; R_2=Glc (OAc)$_4$
VI. R_1=Ac; R_2=Glc (OAc)$_4$
VII. R_1=OGlc (OAc)$_4$; R_2=H
VIII. R_1=OGlc (OAc)$_4$; R_2=Glc (OAc)$_4$
IX. R_1=H; R_2=Glc (OAc)$_4$

effected with α-acetobromoglucose in the presence of silver oxide by a method described previously [2]. The results are given below (α-ABG − α-acetobromoglucose):

Initial substances, mmole			Reaction products	Recovery of the initial substances, %
hydroxyketone	α-ABG	Ag_2O		
I, (I)	8	3	39.1% (IV): (V) = 3:1	31.5
II, (I)	3	3	59.9% (VII); 9.9% (VIII)	25.0
III, (I)	3	3	13.9% (IX)	75.2

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