

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: $\bar{X} = 99.93\%$, $S = 0.4575$, $S_r = 0.00458$, $St = \pm 1.18$, $\bar{X} \pm St = 99.93 \pm 1.18$.

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.

LITERATURE CITED

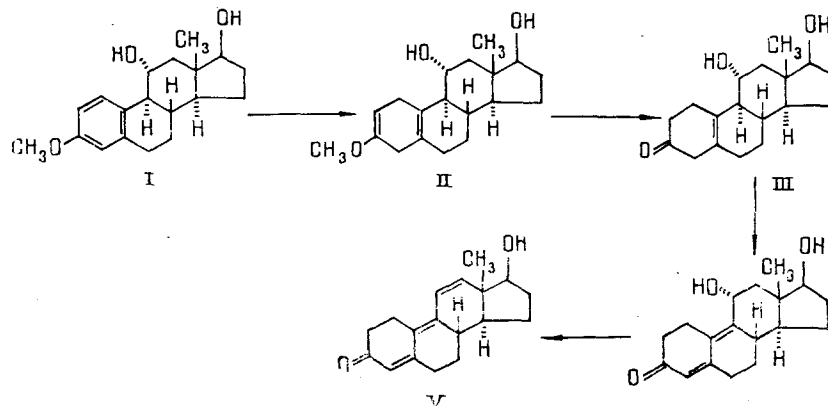
1. M. D. Mashkovskii, Drugs [in Russian], Moscow, Vol. 1 (1985), p. 578.
2. L. Kovalenko, Farmats. Zh., No. 2, 55 (1968).
3. J. A. El-Sebai, A. M. Wahbi, and S. M. Abdel, Pharmazie, 28, No. 3 (1973).
4. J. A. El-Sebai, A. M. Wahbi, and S. M. Abdel, Pharmazie, 28, No. 4 (1973).
5. L. Kovalenko, Farmats. Zh., No. 4, 48 (1986).

NEW APPROACH TO SYNTHESIS OF TRENBOLONE

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In recent years, the anabolic steroid 17 β -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 11 α -hydroxyestradiol [3] under the conditions of the Birch reaction [4].



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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).

17 β -Hydroxyestra-4,9,11-trien-3-one (V): mp $184-186^{\circ}\text{C}$. UV spectrum, λ_{max} : 343 nm (log ϵ 4.41). IR spectrum ($\nu_{\text{max}}^{\text{KBr}}$, 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 ($\text{M}-\text{H}_2\text{O}$, 26).

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

LITERATURE CITED

1. L. Velluz, G. Nomine, K. Bucourt, and J. Mathieu, C. R. Acad. Sci., 257, 569 (1963).
2. A. V. Zakharychev, S. N. Ananchenko, and I. V. Torgov, Steroids, 4, 3 (1964).
3. P. Turnbull, K. Sykora, and J. H. Fried, J. Am. Chem. Soc., 88, 4764 (1966).
4. B. J. Magerlein and J. A. Hogg, J. Am. Chem. Soc., 80, 2220 (1958).

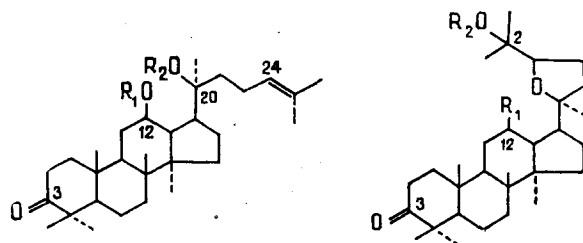
GLYCOSYLATION OF TRITERPENOIDS OF THE DAMMARANE SERIES.

VIII. DAMMARANE HYDROXYKETONE β -D-GLYCOPYRANOSIDES

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



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

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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