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PREPARATION OF ACETATES OF GLUCOSIDES OF STEROID AND TRITERPENE ALCOHOLS VIA THE ISOMERIC ORTHOESTERS

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The conversion of steroid and triterpene 1,2-orthoacetates of α-D-glucose into the isomeric glucosides and substances of nonglucosidic nature has been studied. Isomerization was carried out in nitromethane in the presence of 0.36 mmole of $HgBr_2$ per mmole of orthoester. The yield of cholesterol β-glucoside tetraacetate under these conditions was 65.3%, of β-sitosterol β-glucoside tetraacetate 53.1%, of betulin 28-C-β-glucoside pentaacetate 59.8%, and of betulin β-diglucoside octaacetate 46.3%. It has been established that the initial stage of the conversion of cholesteryl orthoacetate of α-D-glucose in nitromethane in the presence of mercuric bromide is the partial hydrolysis of the orthoester with the formation of the alcohol, which participates in the subsequent transformation of the orthoester.

We have previously reported the formation of byproducts — acetates of the initial alcohols and ethers — in the glycosylation of steroids and triterpenoids by carbohydrate 1,2-orthoacetates [1]. It was assumed that the source of these byproducts was the orthoesters isomeric with the desired glucosides. In actual fact, the additional treatment of 3,4,6-tri-O-acetyl-1,2-O-(1-cholesteryloxyethylidene)-α-D-glucopyranose (I) under the conditions of synthesis led to the formation of a complex mixture of substances the main components of which had been isolated previously in glycosylation. This permitted the opinion to be expressed that the formation of steroid and triterpenoid glucosides under the conditions of the orthoester method is the result of several processes, including the direct glycosylation of the alcohol and the transesterification by the alcohol of the glycosylating orthoester, leading to the appearance in the reaction mixture of an orthoester isomeric with the desired glucoside and undergoing further transformations [3, 4] under the conditions of the reaction.

At the same time, we have shown [5] that isomeric orthoacetates of glycosides of polycyclic alcohols are formed in good yields (60-90%) under azeotropic distillation conditions on the interaction of 3,4,6-tri-O-acetyl-1,2-O-(tert-butoxyethylidene)-α-D-glucopyranose (Ia) or 3,6-di-O-acetyl-1,2-O-(1-methoxyethylidene)-4-O-(2,3,4,6-tetra-O-acetyl-α-D-glucopyranosyl)-α-D-glucopyranose with polycyclic alcohols in nitromethane, chlorobenzene, and toluene in the absence of a specially added catalysts. The ready formation of orthoesters induced us to study in detail the possibility of their conversion into glycosides.

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TABLE 1. Conversion of the Cholesteryl Orthoacetate of α-D-Glucose under the Conditions of a Reaction Catalyzed by Mercuric Bromide (0.5 mmole of the Orthoester (I) in 10 ml of Nitromethane)

Expt. No.	$HgBr_2$, mmole	Temp., °C	Time of reaction	Yield, %					
				I	III	II* and III	IV	V	VI
1	0,18	70	2 h	60,0	2,8	II 1,1 III 1,9	3,2	15,0	14,9
2	0,18	90	1 h 10 min	65,3	3,4	II 1,1 III 1,1	1,8	11,4	12,2
3	0,045	90	2,5 h	55,6	3,3	II 1,4 III 1,9	3,2	18,1	12,8
4	0,18	135—140	40 min	60,0	4,4	II 1,4 III 2,2	3,2	6,2	14,4
5	0,09	135—140	1.5 h	45,0	3,3	II 1,0 III 2,1	3,1	12,7	17,4
6	0,36	135—140	5 min	57,5	6,7	II 1,1 III 1,3	2,3	7,2	15,9
7	0,5	20	3 days	59,7	0,8	II 1,6 III 2,4	3,6	12,5	14,4

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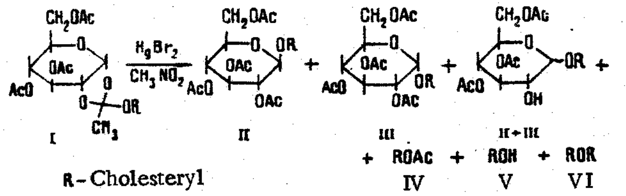
*The yields of compounds (II) and (III) after acetylation of the partially deacetylated glucosides.

The conversion of the orthoester of simple alcohols into glycosides was first shown by Helferich and Weis [6] in the case of the methyl orthobenzoate of α-D-glucose (CH_3NO_2, $HgBr_2$ + HCl). Subsequently, the isomerization of carbohydrate orthoacetates of various degress of complexity into glycoside acetates and other derivatives under the conditions of proton catalysis was reported previously by a number of authors [7, 8]. However, in the case of orthoacetates of polycyclic alcohols, the use of proton catalysts leads to a low yield of trans-glycosides (17-20%). A competing reaction in the proton-catalyzed rearrangements of orthoacetates of polycyclic alcohols is the formation of the acetates of the alcohols, the yields of which amount to 40-60%. A similar conversion of the orthoester (I) into substances of nonglycosidic nature has also been observed by Wulf and Schmidt [9].

Table 1 gives the results of the rearrangement of the orthoester (I) in nitromethane in the presence of various amounts of mercuric bromide. It follows from Table 1 that the most complete conversion of the orthoester (I) into a trans-glucoside was observed at 90°C with 0.36 mmole of mercuric bromide per mmole of orthoester (experiment 2, Table 1). The temperature of the reaction mixture has little effect on the results of the rearrangement when the amount of mercuric bromide is the optimum.



The considerable increase in the yield of the acetate of the trans-glucoside with a simultaneous decrease in the formation of the acetate of the alcohol as compared with proton-catalyzed rearrangements compels the assumption of a reaction mechanism different from that of proton catalysis. A mechanism of a reaction catalyzed by mercuric bromide has been put forward by Bochkov et al. [10]. This mechanism can probably be applied to the orthoacetates of polycyclic alcohols if we assume that the steric hindrance of the aglycone is compensated by an increase in the amount of $HgBr_2$. Furthermore, the experiments performed by these authors on the isomerization of acetylated orthoesters were performed in sealed tubes with the preliminary high-vacuum drying of the solvents and were not accompanied by the formation of byproducts. In our experiments, in view of the formation of a certain amount of byproducts of nonglycoside nature, it is necessary to assume (no special investigations on the mechanism of the reaction were performed) that several processes took place simultaneously, including the partial hydrolysis of the orthoester as the result of proton catalysis caused by the pres

TABLE 2. Conversion of Steroid and Triterpene 1,2-Orthoacetates of α-D-Glucose under the Optimum Conditions

Expt. No.	Initial substances and their amounts, mmole	Solvent, 10 ml	$HgBr_2$, mmole	Temp. °C	Reac. time	Yield, %		
						Acetate of the β-D-glucoside (II)	Acetate of the α-D-glucoside (III)	other substances
8	Orthoester (Ib) 0.55	Nitromethane	0,198	90	40 min	53,1	1,5	β-Sitosterol acetate, 4.0; β-sitosterol, 11.9; di-β-sitosteryl ether, 15.8
9	Orthoester (Ic), 0,50	»	0,18	90	3,5 h	59,8	—	Betulin monoacetate at C-3, 14.0; betulin diacetate; 10.6
10	Orthoester (Id), 0,30	»	0,11	90	3 h	46,3 (diglucoside)	—	Acetate of betulin 3-O-β-glucoside after acetylation of partially deacetylated product, 20.8%
11	Orthoester (Ia), 1,00 Cholesterol 1,00	Toluene, nitromethane	0,36	135, 90	1,5 h 40 min	56,3	3,9	Dicholesteryl ether, 15.9; cholesterol acetate, 3.7
12	Orthoester (Ia), 2.00 28-O-Acetylbetulin, 0,86	Toluene, nitromethane	0,31	135, 90	3 h 20 min	57,3	2,8	Betulin 28-O-monoacetate, 23.0; betulin diacetate, 3.7; 28-O-acetylbetulin-2-ene, 7.4
13	Orthoester (Ia), 6,00 Betulin 1,50	Toluene, nitromethane	0,54	135, 90	4 h 20 min	23,0		Acetate of betulin 3-O-β-glucoside, acetate of betulin 28-O-β-glucoside, and partially deacetylated betulin 3-O-β-glucoside, all identified chromatographically

ence of traces of moisture. Proton catalysis also occurs on transesterification in the absence of specially added catalysts [5]. The absence of a catalyst and the use of a nonpolar solvent and azeotropic distillation directed the process almost completely towards the formation of the orthoester. However, the fact that the reaction was catalyzed by protons present in the reaction mixture is confirmed by the circumstance that an orthoester of the polycyclic alcohol is formed (in trace amounts) when solutions of the tert-butyl orthoacetate of α-D-glucose and of cholesterol in toluene are mixed at room temperature (the orthoester (I) was detected by thin-layer chromatography and by the acid hydrolysis test).

The initial stage of the reaction catalyzed by mercuric bromide is apparently the partial hydrolysis of the orthoester with the formation of the alcohol. This was confirmed by a control experiment (stoppage of the isomerization reaction after 10 min) in which 70.7% of the initial orthoester, 23.3% of cholesterol, and 3.3% of β-glucose acetate were isolated. The amount of cholesterol in the final reaction mixtures is, as can be seen from Table 1, less than that formed at the beginning of the reaction, which indicates the participation of the alcohol in the further transformation of the orthoester.

The almost identical quantitative ratio of cholesterol acetate and of partially deacetylated cis- and trans-glycosides indicates, as shown by a number of authors [7, 11, 9], a common pathway for the formation of these substances.

The formation of dicholesteryl ether apparently takes place only partly through the dehydration of the alcohol, since a control experiment on the conversion of cholesterol in the presence of mercuric bromide under the conditions of experiment 7 (Table 1) gave only trace amounts of dicholesteryl ether, while in experiment 7 its yield was 14.4%. Apparently, the bulk of the dicholesteryl ether obtained in the reaction is connected with the decomposition of the orthoester (I) as the result of a proton-catalyzed side reaction, as we have reported previously [2].

Table 2 gives information on the rearrangement of 3,4,6-tri-O-acetyl-1,2-O(1-β-sitosteryloxyethylidene)-α-D-glucopyranose (Ib), of 3,4,6-tri-O-acetyl-1,2-O-[1-(3-O-acetylbetulin-28-yloxy)ethylidene]-α-D-glucopyranose (Ic), and of 3,28-bis-O-[3,4,6-tri-O-acetyl-1,2-O-(1-ethylidene)-α-D-glucosyl]betulin (Id), and also information on the rearrangement of steroid and triterpene 1,2-orthoacetates of α-D-glucose without preliminary isolation taking place as the result of transesterification of the orthoester. The yields of trans-glucosides in experiments 8-12 (Table 2) were comparable with the results of the experiments of Table 1. We must mention a comparatively high yield of the acetate of betulin di-β-glucoside (experiment 10, Table 2) in comparison with that from direct glycosylation [1]. On one-stage glycosylation under the given conditions, betulin gave a complex mixture of substances from which 23% of betulin β-diglucoside acetate was isolated.

EXPERIMENTAL

The catalysts and solvents were prepared as described by Kochetkov et al. [12]. Column chromatography was performed on KSK silica gel, 65-100 mesh, treated by the method of Wulf and Schmidt [9]. For thin-layer chromatography (in a fixed layer of silica gel) we used the following solvent systems: petroleum ether—diethyl ether (93:5), petroleum ether—acetone (4:1), (3:1), and (2:1). For column chromatography we used the systems petroleum ether, petroleum ether—diethyl ether (200:1), and petroleum ether—acetone (45:1)→(5:1). The reaction products were detected with a mixture of concentrated sulfuric acid and methanol (1:10) at 100-200°C. Melting points were determined on a Kofler block. The steroid and triterpene orthoacetates of α-D-glucose were synthesized as described previously [5], acid hydrolysis tests being positive. The substances obtained gave no depression of the melting points in mixtures with authentic samples. The yields are given for the chromatographically homogeneous substances.

Experiments 1-7 (for conditions and yields, see Table 1). 1. A reaction flask was charged with 0.358 g of compound (I), 10 ml of nitromethane, and 0.0648 g of mercuric bromide. The reaction was performed at 70°C for two hours. After this time, a few drops of pyridine were added, and the mixture was evaporated and chromatographed on silica gel. This gave 0.22 g of (II), 0.01 g of (III), 0.007 g of (IV), 0.029 g of cholesterol (V), and 0.028 g of dicholesteryl ether (VI). The partially deacetylated glucosides isolated from the reaction mixture were acetylated with a mixture of acetic anhydride and pyridine and rechromatographed. This gave 0.007 g of (III) and 0.004 g of (II).

2. The products were 0.234 g of (II), 0.012 g of (III), 0.004 g of (IV), 0.022 g of (V), and 0.023 g of (VI). The yields of (II) and (III) after the acetylation of the partially deacetylated glucosides were 0.004 g for each.

3. The products were 0.199 g (II), 0.012 g (III), 0.007 g (IV), 0.035 g (V), and 0.024 g (VI). The yields of (II) and (III) after acetylation were 0.007 g and 0.005 g, respectively

4. The reaction was performed under the conditions of azeotropic distillation. It gave 0.215 g of (II), 0.016 g of (III), 0.007 g of (IV), 0.012 g of (V), and 0.027 g of (VI). The yields of (II) and (III) after acetylation were 0.005 g and 0.008 g, respectively.

5. The reaction was performed in a similar manner to experiment 4. It led to 0.216 g of (II), 0.016 g of (III), 0.009 g of (IV), 0.033 g of (V), and 0.044 g of (VI). The yields of (II) and (III) after acetylation were 0.005 g and 0.010 g, respectively.

6. The reaction was performed in a similar manner to experiment 4. It gave 0.206 g of (II), 0.024 g of (III), 0.005 g of (IV), 0.014 g of (V), and 0.030 g of (VI). The yields of (II) and (III) after acetylation were 0.004 g and 0.005 g, respectively.

7. A mixture of 0.358 g (0.5 mmole) of compound (I) and 0.0648 g (0.18 mmole) of $HgBr_2$ in 10 ml of nitromethane was left at room temperature. Monitoring by means of the hydrolytic test and TLC showed the absence of (I) after three days. The reaction mixture was worked up as in the preceding experiments, giving 0.222 g of (II), 0.003 g of (III), 0.008 g of (IV), 0.025 g of (V), and 0.028 g of (VI). The yields of (II) and (III) after acetylation were 0.006 g and 0.009 g, respectively.

Experiments 8-13 (for conditions and yields, see Table 2). 8. A mixture of the orthoester (Ib) (0.407 g) and $HgBr_2$ (0.07128 g) was heated as in experiment 2. This gave 0.216 g of β-sitosterol β-glucoside tetracetate, 0.010 g of β-sitosterol acetate, 0.035 g of di-β-sitosteryl ether, and 0.027 g of β-sitosterol.

9. A mixture of the orthoester (Ic) (0.408 g) and $HgBr_2$ (0.0648 g) was heated under the conditions of experiment 2. This gave 0.244 g of betulin 28-O-β-D-glucoside pentaacetate, 0.034 g of betulin 3-O-monoacetate, and 0.028 g of betulin diacetate.

10. A mixture of the orthoester (Id) (0.348 g) and $HgBr_2$ (0.0396 g) was heated under the conditions of experiment 2, giving 0.161 g of betulin β-diglucoside octaacetate and 0.053 g of a partially deacetylated product which, after acetylation and chromatography, was identified as betulin 3-O-β-glucoside pentaacetate.

11. A mixture of cholesterol (0.386 g) and toluene (10 ml) was charged into a reaction flask, and 3 ml was distilled off. Then 0.205 g of the tert-butyl orthoacetate of α-D-glucose was added and the reaction was carried out with azeotrophic distillation of the solvent for 40 min. Another 0.205 g of the tert-butyl orthoacetate was added and the reaction was continued for 1 h to 10 min. The toluene was distilled off from the reaction mixture, the flask was cooled to room temperature, and 10 ml of nitromethane and 0.36 mmole of $HgBr_2$ was added to it. This reaction mixture was kept at 90°C for 40 min. After 40 min, no orthoester remained in the reaction mixture, which was then worked up in the usual way. This yielded: 0.403 g of cholesterol β-D-glucoside tetraacetate, 0.028 mg of cholesterol α-D-glucoside tetraacetate, 0.060 g of dicholesteryl ether, and 0.016 g of cholesterol acetate.

12. The reaction was performed similarly to experiment 4. It yielded 0.4 g of betulin 3-O-β-glucoside pentaacetate, 0.20 g of betulin 3-O-α-glucoside pentaacetate, 0.096 g of betulin 28-O-monoacetate, 0.017 g of betulin diacetate, and 0.030 g of 28-O-acetylbetulin-2-ene, which was identical with an authentic sample in relation to mass spectrometry and ^{13}C NMR spectroscopy [13].

13. The reaction was performed similarly to experiment 4. This gave 0.380 g of betulin β-diglucoside octaacetate.

SUMMARY

1. The optimum conditions for the rearrangement of steroid and triterpene 1,2-orthoacetates of α-D-glucose have been found.

2. It has been established that an increase in the amount of mercuric bromide as catalyst leads to an increase in the yield of the acetates of β-glucosides of steroid and triterpene alcohols in the isomerization of the appropriate orthoesters.

3. It has been shown that the rearrangement of orthoacetates of polycyclic alcohols under the usual conditions is accompanied by the initial partial hydrolysis of the orthoester with the formation of an alcohol, which participates in the subsequent transformation of the orthoester.

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PREPARATION OF 21-ALDEHYDES OF $\Delta^{17(20)}$-STEROIDS

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A method is described for the synthesis of 17(20)-unsaturated 21-aldehydes by the oxidation of 17β-hydroxy-17α-vinylsteroids with pyridine chlorochromate. The following compounds were obtained in the course of the investigation: 3-hydroxy-trans-pregna-5,17-dien-21-al acetate, mp 177-178°C (MeOH); trans-pregna-5,17(20)-diene-3β,21-diol 3-acetate (I), mp 150-153°C (MeOH); the 21-acetate of (I), with mp 130-133°C (ether—hexane); 3-oxo-trans-pregna-4,17(20)-dien-21-al, with mp 130-132°C (MeOH); and 3-oxo-trans-pregna-4,17(20)-dien-21-cic acid, $C_{21}H_{28}O_3$, mp 261-263°C. The IR and NMR spectra of the substances obtained are given.

In the use of some steroids (such as cholesterol and β-sitosterol) for the synthesis of hormone preparations, the question arises of the passage from compounds of the androstane series to the pregnane series, especially to corticosteroids. In this connection, interest is presented by 17(20)-unsaturated aldehydes, which are convenient intermediates for the construction of a dihydroxyacetone chain in position 17 of the molecule.

Among methods for the synthesis of $\Delta^{17(20)}$-21-aldehydes recently published in the literature we must mention the condensation of 17α-ethynyl-17β-hydroxysteroids with dimethylformamide acetal [1]. This reaction, giving a high yield (90%), has been described for the case of the methyl ether of ethynylestradiol. However, the greatest interest is presented by compounds containing the Δ^4-3-keto grouping that is a structural element of the articosteroids. But in this case, the condensation described above takes place with a low yield even when ethylene ketal protection is used for the 3-keto group as, for example, for 17α-ethynyl-17β-hydroxyandrost-4-en-3-one.

In the present paper we describe a method for the synthesis of 17(20)-unsaturated aldehydes by the oxidation of vinyl-substituted steroid alcohols with pyridine chlorochromate. As the initial compounds we selected 17α-vinylandrost-5-ene-3β,17β-diol 3-acetate (I) and 17β-hydroxy-17α-vinylandrost-4-en-3-one (V). Oxidation with pyridine chlorochromate with the aim of obtaining aldehydes has been proposed for vinyl-substituted acyclic alcohols. For steroid alcohols it has been described in the case of 3α-vinylcholestan-3β-ol, which contains a sterically unhindered equatorial hydroxy group in a six-membered ring. The product of oxidation was a mixture (1:1) of isomeric aldehydes with a yield of 70% [2].

We have studied the oxidation of vinyl-substituted alcohols with the alcohol group in the five membered ring of androstane derivatives. In our case, the reaction had to be performed at a sterically hindered group. Oxidation was carried out in methylene chlorine at room temperature. The reaction was monitored by the TLC method and was continued until the initial compound had disappeared (about 120 h). The reaction took place with 17α-vinylandrost-5-ene-3β,17β-diol 3-acetate with a satisfactory result (yield 50%).

The oxidation of 17β-hydroxy-17α-vinylandrost-4-en-3-one (V) formed a mixture of substances, from which it was possible to isolate the aldehyde (VI) only by chromatography on silica gel followed by preparative TLC of the corresponding fractions. The acid (VII), the structure of which agrees with the results of spectral analysis, was isolated at the same time. On the basis of literature analogy and the results of TLC, the formation of the oxide

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