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HYDROGENOLYSIS OF THE ISOMERIC 1,2:4,6-DI-*O*-BENZYLIDENE-α-D-GLUCOPYRANOSE DERIVATIVES WITH THE $LiAlH_4$–$AlCl_3$ REAGENT

ANDRÁS LIPTÁK, JÁNOS IMRE, JÁNOS HARANGI, AND PÁL NÁNÁSI
Institute of Biochemistry, L. Kossuth University, H-4010 Debrecen (Hungary)
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ABSTRACT

Both isomers of 1,2:4,6-di-*O*-benzylidene-α-D-glucopyranose (and their 3-*O*-acetyl and 3-*O*-benzyl derivatives) have been prepared and their ^1H- and ^{13}C-n.m.r. spectra assigned. The mode of hydrogenolysis of the dioxolane ring in these isomers by the $LiAlH_4$–$AlCl_3$ reagent is determined by the configuration at the acetal carbon and is independent of the electronic character of the two oxygen atoms.

INTRODUCTION

The direction of the hydrogenolytic ring-cleavage of dioxolane-type benzylidene derivatives by the $LiAlH_4$–$AlCl_3$ reagent is determined by the configuration of the acetal carbon[1,2]. In the compounds investigated to date, the dioxolane rings were formed from secondary hydroxyl groups and thus the electronegativities of the two oxygen atoms were similar. We now report on the behaviour of the two isomers of 1,2:4,6-di-*O*-benzylidene-α-D-glucopyranose, where the electronegativity of the oxygen atoms of the dioxolane ring is different.

RESULTS AND DISCUSSION

One of the two isomeric 1,2:4,6-di-*O*-benzylidene-α-D-glucopyranoses was prepared by Wood *et al.*[3], and an *endo*-phenyl structure for the 1,2-benzylidene acetal was assigned by Coxon and Hall[4,5]. This assignment was based on the observation that partial catalytic hydrogenolysis of the di-*O*-benzylidene compound gave a 1,2-*O*-benzylidene-α-D-glucopyranose derivative for which an *endo*-phenyl configuration had been inferred by Lemieux and Detert[6].

Recently, a series of 1,2-*O*- and 1,2:4,6-di-*O*-alkylidene-α-D-glucopyranose derivatives was synthesised by Dick *et al.*[7], and four n.m.r. parameters were correlated with the bulk and orientation of the 2′-substituent of the dioxolane ring; three of these n.m.r. features are important, namely, (*a*) the 2′-substituent is deshielded when *endo*, (*b*) H-2 or H-5 is deshielded by bulky *exo* or *endo* 2′-substituents, and

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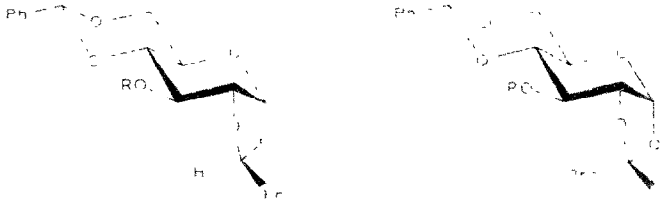
(*c*) the values of $J_{3,4}$ and $J_{4,5}$ are larger when a bulky 2'-substituent is *exo* than when it is *endo*.

In order to gain further insight into the determination of the configuration of this class of compounds, the isomeric 1,2:4,6-di-*O*-benzylidene-α-D-glucopyranoses were investigated.

Benzylidenation of D-glucose by the published procedure[4] gave mainly 4,6-*O*-benzylidene-D-glucopyranose (**1**), but the known 1,2:4,6-di-*O*-benzylidene derivative **2** (10.6%) and the hitherto unknown isomer **3** (3.4%) could be isolated from the mother liquor by crystallisation and column chromatography, respectively. Reaction of **1** with α,α-dimethoxytoluene in *N*,*N*-dimethylformamide in the presence of Amberlite IR-120 (H^+) resin gave **2** and **3** in yields of 17.2% and 16.7%, respectively. Conventional acetylation of **2** and **3** afforded the 3-acetates **4** and **5**, respectively, and benzylation yielded the 3-*O*-benzyl derivatives **6** and **7**. The diastereomeric 3,4,6-tri-*O*-acetyl-1,2-*O*-benzylidene-α-D-glucopyranoses (**8** and **9**) were obtained by the method of Dick *et al.*[7]



2 R = H
4 R = Ac
6 R = Bz

3 R = H
5 R = Ac
7 R = Bz

In the ^{1}H-n.m.r. spectra of **2**, **4**, **6**, and **8**, the dioxolane acetal proton resonated at a δ value higher than that of the corresponding proton of **3**, **5**, **7**, and **9**. On the basis of the rule proposed by Baggett *et al.*[8], these two sets of compounds have *exo*-phenyl and *endo*-phenyl structures, respectively. This finding accords with the configurations proposed for **8** and **9** by Dick *et al.*[7] and Rees *et al.*[9], but not with those proposed by Lemieux and Defert[6] for **8** and **9** or with those reported by Coxon[5] for **2**.

Our configurational assignments were verified by the chemical shifts of H-5 for the *endo*-phenyl isomers and by those of H-2 for the *exo*-phenyl isomers (see Table I). Similar regularities were also found for the coupling constants, as observed by Dick *et al.*[7], thus, $J_{3,4}$ and $J_{4,5}$ had larger values for the *exo*-phenyl isomers. The ^3J values for the bridgehead hydrogens are also characteristic[10] of the configuration of the acetal carbon. For the compounds studied here, $J_{1,2}$ values were greater for the *endo*-isomers, whereas the $J_{5,6}$ values were greater for the *exo*-isomers (see Table II). Thus, the phenyl group in the 1,2-benzylidene group of **2**, **4**, **6**, and **8** is established as *exo*, and that of **3**, **5**, **7**, and **9** as *endo*.

The ^{13}C-n.m.r. rules[11] for the acetal carbon atoms of dioxolane derivatives formed from secondary hydroxyl groups failed for the compounds described above.

TABLE I

^{1}H-N M R DATA FOR 1,2-*O*- AND 1,2:4,6-DI-*O*-BENZYLIDENE-α-D-GLUCOPYRANOSE DERIVATIVES

Compound	*Chemical shift, δ*												
	H-1	*H-2*	*H-3*	*H-4*	*H-5*	*H-6a*	*H-6e*	*Dioxolane (1,2-O-)*	*Dioxane (4,6-O-)*	*OH*	*Ac*	*Ph*	*PhCH$_2$*
2	5.65d	4.35dd	4.11dd	3.55dd	3.90m	3 71dd	4.39dd	6.18s	5.56s	2.95s		7.60–7.30m	
3	5.60d	4.07dd	3.95m	3.50dd	3.95m	3.65dd	4.35dd	5.86s	5.47s	3.16s		7.60–7.20m	
4	5.69d	4.35dd	5.53dd	3.67dd	4.03m	3 70dd	4.40dd	6.33s	5.52s		2.10s	7.80–7.20m	
5	5.69d	4.17dd	5 33dd	3.76dd	4.06m	3.68dd	4.37dd	5.83s	5.50s		2.10s	7.70–7.20m	
6	5.66d			4.60 ← multiplet →3.50				6.07s	5.57s			7.80–6.80m	4.86s
7	5.64d			4.50 ← multiplet →3.50				5.83s	5.55s			8.00–6.80m	4.78s
8	5.73d	4.40–4.08m	5.36dd	4.98dd	4.40–4.08m	4.40–4.08m	4.40–4.08m	6.43s			2.10s 2.08s 2.06s	7.50–7.20m	
9	5 74d	4.20–4.00m	5.29dd	4 93dd	4.20-4.00m	4 20–4.00m	4.20–4.00m	5.86s			2.14s 2.10s 2.09s	7.60–7.10m	

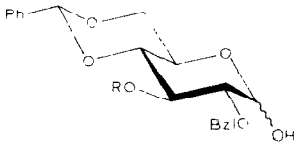
TABLE II

^{1}H-N.M.R. DATA FOR 1,2-*O*- AND 1,2:4,6-DI-*O*-BENZYLIDENE-α-D-GLUCOPYRANOSE DERIVATIVES

Compound	*Coupling constants, J (Hz)*						
	$J_{1,2}$	$J_{2,3}$	$J_{3,4}$	$J_{4,5}$	$J_{5,6a}$	$J_{5,6e}$	$J_{6a,6e}$
2	4.9	6.4	9.35	9.2	10.0	4.9	10.25
3	5.1	5.1	9.1	9.5	10.2	5.1	10.35
4	4.8	5.7	9.3	9.4	9.4	4.6	10.0
5	5.2	3.3	8.1	9.1	9.4	4.6	9.9
8	4.9	4.5	4.8	8.7			
9	5.2	3.0	3.0	9.1			

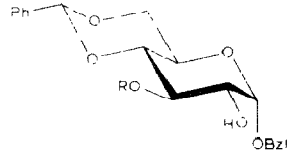
Similar chemical shifts were also found[12] for the 1,2-acetal carbons of the 1,2:3,4-di-*O*-benzylidene-β-L-arabinopyranoses. The ^{13}C-n.m.r. data are listed in Table III.

Hydrogenolysis of the *exo*-isomer **2** with the $LiAlH_4$–$AlCl_3$ reagent for 10 min at 0° gave a 95:5 mixture of two products, the major component (**10**) of which was isolated crystalline. Compound **10** reduced Fehling's solution and could be detected by the aniline hydrogenphthalate reagent on paper chromatograms, suggesting that HO-1 was unsubstituted. This assumption was supported by ^{1}H-n.m.r. data; the signals for the anomeric protons could be assigned as an AB-quartet, signals for a benzyl group were present, and the signal at δ 5.50 indicated the presence of a 2-phenyl-1,3-dioxane ring. These data indicated **10** to be 2-*O*-benzyl-4,6-*O*-benzylidene-D-glucopyranose, and this was confirmed by a synthesis involving conventional benzylidenation of 2-*O*-benzyl-D-glucose.



10 R = H

12 R = Bzl



11 R = H

13 R = Bzl

Ring cleavage of the *endo*-isomer **3** required 30 min at 0° and gave only one product, namely, benzyl 4,6-*O*-benzylidene-α-D-glucopyranoside (**11**). Compound **11** was the minor product (5%) of the hydrogenolysis of **2**.

Likewise, ring cleavage of the 3-*O*-benzyl derivative **6** gave 2,3-di-*O*-benzyl-4,6-*O*-benzylidene-D-glucopyranose (**12**), whereas **7** afforded benzyl 3-*O*-benzyl-4,6-*O*-benzylidene-α-D-glucopyranoside (**13**). The structure of **12** was verified by a synthesis involving benzylidenation of 2,3-di-*O*-benzyl-D-glucose.

These reactions clearly demonstrate that the electronegativities of the oxygens of the dioxolane ring do not play a decisive role in the determination of the direction of ring cleavage. However, ring cleavage must be determined by the conformation of the dioxolane ring, which, in turn, is strongly influenced by the orien-

TABLE III

^{13}C-N M R CHEMICAL SHIFTS (p.p.m.) FOR COMPOUNDS **2–9**

Carbon atom	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
C-1	98.4	98.7	98 3	97.9	98.4	98 4	97.3	96.6
					181.5			
C-2	79.0	78.1	77.3	77.2	79.4	79.3	74.5	74 8
C-3	71.3	73.2	71.3	73.3	78.7	78.7	70.9	70.8
C-4	79.3	79.9	77.2	78.2	77.3	77.1	68.3[a]	67.3[a]
C-5	63.4	63 4	63.6	62.6	63.3	63.2	68.8[a]	68.7[a]
C-6	68 8	68.8	68.8	68.9	68.8	68.8	62.9	62.5
Dioxolane Ph*C*H	102.8	101.1	102.8	101.6	102.8	102.9	103.9	103.0
q[b]	137 8[a]	137.1[a]	137.6[a]	136.4[a]	137.3	137.3	136.4	138.1
Dioxane Ph*C*H	102.0	101.8	101.6	101.6	101.3	101.3		
q	137.2[a]	137.2[a]	137.2[a]	137.2[a]	137.5[a]	137.4[a]		
3-O-*C*O-CH$_3$			169.5	169.6			170.4[a]	170.6[a]
3-O-CO-*C*H$_3$			20.7	20.8			20.6	20.6
4-O-*C*O-CH$_3$							169.6[a]	169.7[a]
4-O-CO-*C*H$_3$							20.6	20.6
6-O-*C*O-CH$_3$							169.4[a]	169.3[a]
6-O-CO-*C*H$_3$							20.6	20.6
3-O-*C*H$_2$-Ph					73.1	73.1		
q					137 9	137 9		

[a]Assignments may be reversed. [b]Quaternary carbon of Ph.

tation of the C-2′ substituent. The results reported here confirm the earlier observations that hydrogenolysis is a highly chemoselective reaction and that, under the appropriate conditions, the reagent can distinguish between the dioxane and dioxolane rings.

EXPERIMENTAL

General methods. — Melting points were determined on a Kofler hot-stage apparatus and are uncorrected. Optical rotations were measured with a Perkin–Elmer 241 polarimeter. N.m.r. spectra (for solutions in $CDCl_3$ with internal Me_4Si) were recorded at room temperature and at frequencies of 100.1 MHz (^1H) and 25.16 or 50.3 MHz (^{13}C) with Jeol MH-100, Varian XL-100 FT-15, or Bruker WP-200 SY spectrometers. T.l.c. and column chromatography were carried out on Kieselgel G (Merck). G.l.c. was performed at 275° with a Hewlett-Packard 5840 A instrument, and a column (1.2 m × 2 mm) of 10% of UCW 982 on Gas Chrom-Q (80–100 mesh) with N_2 as the carrier gas at 20 mL/min.

The exo- (**2**) *and* endo-*isomers* (**3**) *of 1,2:4,6-di-O-benzylidene-α-*D-*glucopyranose.* — (*a*) A mixture of powdered, anhydrous D-glucose (20 g), freshly fused and powdered zinc chloride (20 g), and benzaldehyde (400 mL) was shaken for 24 h, cooled to 0°, and diluted with ice–water (200 mL). After 1 h, the aqueous phase was decanted, and light petroleum (b.p. 40–60°) was added to the organic layer. The product was collected, and washed with water (2 × 50 mL) and light petroleum (2 × 50 mL), to give **1** (4.62 g, 15.6%), m.p. 143–154°, $[\alpha]_D$ +26°, (*c* 0.6, methanol); lit.[3] m.p. 140–150°.

The filtrate was extracted with chloroform (250 mL), the extract was washed with water (3 × 50 mL), and excess of benzaldehyde was removed by steam distillation in the presence of $NaHCO_3$. A solution of the residue in chloroform (250 mL) was washed with water (3 × 100 mL), dried (Na_2SO_4), and concentrated. T.l.c. (19:1 chloroform–acetone) of the partially crystalline residue revealed components with R_F 0.63 and 0.50 in the ratio 72:28 (determined by g.l.c.). Crystallisation of this mixture from ethanol (20 mL) gave the major component (**2**, 3.92 g). Concentration of the mother liquor and column chromatography of the residue gave, first, the component with higher mobility (**2**, 0.98 g), which was combined with the foregoing product and recrystallised from ethanol (15 mL) to give **2** (4.22 g, 10.6%), m.p. 164–165°, $[\alpha]_D$ +96° (*c* 1, chloroform). R_F 0.63 (19:1 chloroform–acetone); lit.[3] m.p. 161–162°, $[\alpha]_D$ +107° (*c* 1.3, chloroform).

Eluted second was **3** (1.89 g) which, after two recrystallisations from ether (5 + 5 mL), gave material (1.34 g, 3.38%) having m.p. 133–134°, $[\alpha]_D$ +93° (*c* 0.7, chloroform), R_F 0.50.

Anal. Calc. for $C_{20}H_{20}O_6$: C, 67.41; H, 5.66. Found: C, 67.34; H, 5.64.

(*b*) A mixture of **1** (4.5 g), *N*,*N*-dimethylformamide (20 mL), Amberlite IR-120 (H^+) resin (1 g), and α,α-dimethoxytoluene (10 g) was stirred for 1 h *in vacuo* at 70°. T.l.c. then revealed the presence of **2** and **3**. The mixture was filtered and

concentrated, and a solution of the residue in dichloromethane was washed several times with water, dried, and concentrated. Column chromatography of the residue gave **2** (1.03 g, 17.22%), m.p. 163–164°, $[\alpha]_D$ +98° (*c* 0.7, chloroform); and **3** (0.996 g, 16.66%), m.p. 133–135°, $[\alpha]_D$ +91° (*c* 0.8, chloroform).

3-O-Acetyl-exo- (**4**) *and* endo-*1,2:4,6-di-O-benzylidene-α-*D-*glucopyranose* (**5**). — Conventional treatment of **2** (0.2 g) with pyridine (1 mL) and acetic anhydride (1 mL), with two recrystallisations of the product from ethanol, gave **4** (0.2 g, 89.4%), m.p. 188–189°, $[\alpha]_D$ +66° (*c* 0.9, chloroform), R_F 0.48 (9:1 benzene–acetone); lit.[3] m.p. 178°, $[\alpha]_D$ +81° (*c* 1, chloroform).

Likewise, acetylation of **3** (0.2 g) gave **5** (0.22 g, 78.7%), m.p. 104–105°, $[\alpha]_D$ +97° (*c* 1, chloroform), R_F 0.40 (9:1 benzene–acetone).

Anal. Calc. for $C_{22}H_{22}O_7$: C, 66.32; H, 5.57. Found: C, 66.41; H, 5.50.

3-O-Benzyl-exo- (**6**) *and* endo-*1,2:4,6-di-O-benzylidene-α-*D-*glucopyranose* (**7**). — A mixture of **2** (1.5 g), potassium hydroxide (3 g), and benzyl chloride (10 mL) was stirred at 105° for 12 h, cooled, diluted with chloroform (150 mL), and washed with water (5 × 50 mL). The organic layer was steam distilled from $NaHCO_3$. The aqueous solution was extracted with chloroform, and the extract was washed with water (5 × 50 mL), dried, and concentrated. The residue was recrystallised from ethanol, to yield **6** (1.75 g, 93.1%), m.p. 133–134°, $[\alpha]_D$ +77° (*c* 0.6, chloroform), R_F 0.40 (9:1 light petroleum–ethyl acetate).

Anal. Calc. for $C_{27}H_{26}O_6$: C, 72.63; H, 5.87. Found: C, 72.55; H, 5.90.

Likewise, benzylation of **3** (0.275 g) and crystallisation of the product from ethanol (5 mL) afforded **7** (0.302 g, 87.6%), m.p. 75–76°, $[\alpha]_D$ +83° (*c* 0.6, chloroform), R_F 0.30.

Anal. Found: C, 72.69; H, 5.84.

*2-O-Benzyl-4,6-*O*-benzylidene-*D*-glucopyranose* (**10**). — (*a*) To a solution of **2** (1 g) in 1:1 ether–dichloromethane (100 mL) at 0° were added, with stirring, $LiAlH_4$ (0.32 g) and a solution of $AlCl_3$ (1.3 g) in ether (5 mL). After 10 min, t.l.c. revealed products with R_F 0.50 and 0.45 (19:1 chloroform–methanol) in the ratio 95:5; a few percent of **2** was also detected. The minor product corresponded to the hydrogenolysis product of the *endo*-isomer. To minimise the formation of by-products, the reaction was terminated before all of **2** had reacted.

The excess of reagent was decomposed with ethyl acetate (3 mL) and water (5 mL), and the mixture was diluted with ether (100 mL), filtered, washed with water (5 × 25 mL), dried, and concentrated. The major product was isolated by column chromatography and then recrystallised from ethanol (3 mL), to give **10** (0.73 g, 72.1%), m.p. 180–181°, $[\alpha]_D$ −6° (*c* 0.5, chloroform), R_F 0.50 (19:1 chloroform–methanol). ^{1}H-N.m.r. data [$CDCl_3$–$(CD_3)_2SO$, 1:1]: δ 7.60–7.10 (m, 10 H, 2 Ph), 6.98 (b, 1 H, OH), 6.43 (b, 1 H, OH), 5.50 (s, 1 H, PhC*H*), 5.18 (d, <1 H, $J_{1,2}$ 4.0 Hz, H-1α), 5.00–4.60 (m, <3 H, H-1β and PhCH_2), and 4.30–3.08 (m, 6 H, skeleton protons).

Anal. Calc. for $C_{20}H_{22}O_6$: C, 67.03; H, 6.19. Found: C, 67.22; H, 6.14.

(*b*) 2-*O*-Benzyl-D-glucose[13] (0.2 g) was treated with benzaldehyde (2 mL) in

the presence of freshly fused $ZnCl_2$ (0.5 g) for 12 h. The excess of benzaldehyde was removed by steam distillation, to give a syrupy residue that crystallised from ethanol (1 mL) to afford pure **10** (0.12 g, 46.7%), m.p. 179–180°, $[\alpha]_D$ −5° (*c* 0.5, chloroform).

*Benzyl 4,6-O-benzylidene-α-*D*-glucopyranoside* (**11**). — To a solution of **3** (0.5 g) in 1:1 dichloromethane–ether (100 mL) were added $LiAlH_4$ (0.16 g) and a solution of $AlCl_3$ (0.56 g) in ether (5 mL). After 30 min, no **3** could be detected by t.l.c., and a single product, identical with the by-product of the hydrogenolysis of **2**, was present. Work-up as described above and recrystallisation of the product from ethanol (2 × 2 mL) gave **11** (0.393 g, 78.3%), m.p. 161–162°, $[\alpha]_D$ +102° (*c* 0.5, chloroform), R_F 0.41 (19:1 chloroform–methanol); lit.[14] m.p. 161–162°, $[\alpha]_D$ +107° (*c* 1, chloroform).

*2,3-Di-O-benzyl-4,6-O-benzylidene-*D*-glucopyranose* (**12**). — (*a*) Compound **6** (0.5 g) was hydrogenolysed in 1:1 ether–dichloromethane (30 mL) with $LiAlH_4$ (0.045 g) and $AlCl_3$ (0.155 g) for 1 h at 20°. The usual work-up, with crystallisation of the product from ethanol (10 mL), gave **12** (0.406 g, 81.5%), m.p. 160–162°, $[\alpha]_D$ −30° (*c* 0.5, chloroform), R_f 0.45 (9:1 benzene–methanol). ^{1}H-N.m.r. data: δ 7.70–7.00 (m, 15 H, 3 Ph), 5.55 (s, 1 H, PhC*H*), 5.24–5.04 (m, 1 H, H-1), 4.92–4.65 (m, 4 H, 2 PhCH_2), 4.44–3.12 (m, 6 H, skeleton protons), and 1.57 (b, 1 H, OH).

Anal. Calc. for $C_{27}H_{28}O_6$: C, 72.30; H, 6.29. Found: C, 72.22; H, 6.33.

(*b*) 2,3-Di-*O*-benzyl-D-glucose[15] (0.2 g) was benzylidenated, as described for **10**, to give **12** (0.138 g, 53.9%), m.p. 162°, $[\alpha]_D$ −33° (*c* 0.4, chloroform).

*Benzyl 3-O-benzyl-4,6-O-benzylidene-α-*D*-glucopyranoside* (**13**). — To a solution of **7** (0.1 g) in 1:1 dichloromethane–ether (10 mL) were added $LiAlH_4$ (20 mg) and $AlCl_3$ (70 mg), and the mixture was stirred at room temperature for 1 h. G.l.c. then revealed two products in the ratio 98:2. T.l.c. showed the minor product (R_f 0.52) to be identical with the hydrogenolysis product of the *exo*-isomer **6**. The major product was isolated by column chromatography and recrystallised from ethanol (5 mL), to give **13** (0.067 g, 66.8%), m.p. 122–123°, $[\alpha]_D$ +87° (*c* 0.6, chloroform), R_F 0.62 (19:1 dichloromethane–acetone). ^{1}H-N.m.r. data: δ 7.52–7.00 (m, 15 H, 3 Ph), 5.56 (s, 1 H, PhC*H*), 5.04 (d, 1 H, $J_{1,2}$ 3.3 Hz, H-1), 4.92–4.43 (m, 4 H, PhCH_2), 4.42 (dd, 1 H, $J_{2,3}$ 8.4 Hz, H-2), 4.00–3.48 (m, 5 H, skeleton protons), and 2.30 (d, 1 H, OH).

Anal. Calc. for $C_{27}H_{28}O_6$: C, 72.30; H, 6.29. Found: C, 72.33; H, 6.24.

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