Thus, the experimental results suggest that the yield and composition of the products of benzene alkylation by MCHol depend on a series of competing and mutually connected factors, including solubility of B and MCH in H_2SO_4 and, correspondingly, on whether alkylation proceeds in the inorganic phase or in the boundary between the organic and inorganic phases.

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ANODIC FUNCTIONALIZATION OF OLEFINS IN ALCOHOLS IN THE PRESENCE OF HALIDE SALTS

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UDC 541.138.2:547.538.141:
547.33'141

Electrolysis of conjugated, unbranched arylolefins in the presence of alkali metal halides in alcohols affords 1-aryl-2-bromoketals in 60-90% yields. Under these conditions, 2-methyl-1-phenylprop-1-ene is converted into 1-bromo-2-methyl-1-phenylprop-1-ene in 80% yield, and arylolefins with no benzylidene hydrogens give 1-aryl-1-alkoxy-2-bromoalkanes.

α-Bromoketones and α-bromoketals are used as intermediates for the synthesis of acyclic and cyclic compounds [1-3]. Specifically, rearrangement of aryl ethyl ketone α-bromoketals affords 2-arylpropionic acids, which possess antiinflammatory activity [4].

α-Bromoketones can be obtained from olefins by oxidation with six equivalents of $NaBrO_2$ in 60-90% yields [5], or by successive or simultaneous treatment with N-bromosuccinimide and the Jones reagent [6, 7] in 70% yield.

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TABLE 1. Electrochemical Oxidation of Olefins in Methanol in the Presence of Halide Salts*

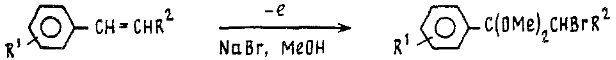
Expt. No.	Olefin	Electrolyte	Conversion of olefin, %	Reaction products, and yields % on olefin reacted
1 †	(Ia)	NaBr	96	(IIa), 48; (Va), 33; (IX), 9
2	(Ia)	NaBr	98	(IIa), 89; (Va), 1; (IX), 7
3	(Ia)	KBr	89	(IIa), 62; (Va), 20; (IX), 6
4	(Ia)	LiBr	99	(IIa), 43; (Va), 32; (IX), 12
5	(Ia)	NH_4Br	100	(Va), 28; (IX), 55
6	(Ib)	NaBr	96	(IIb), 62; 1-(4-methylphenyl)-1-methoxyethane, 7
7	(Ic)	»	97	(IIc), 91
8‡	(Id)	»	78	(IId), 85; 1-bromo-1-phenylprop-1-ene, 9
9‡	(Ie)	»	85	(IIe), 65; 2-bromo-1-methoxy-1-(4-methoxyphenyl)-propane (Ve), 17
10	(IIIa)	»	94	1-Bromo-2-methoxy-2-phenylpropane (VIIa), 94; 2-methoxy-2-phenylpropane, 4
11	(IIIb)	»	78	1-Bromo-2-methyl-1-phenylprop-1-ene, 80; 2-methyl-2-methoxy-1-phenylpropane, 11
12	(IIIc)	NaBr	94	trans-1-Bromo-2-methoxy-2-phenylcyclohexane (VIIc), 62; trans-1-bromo-2-methoxy-1-phenylcyclohexane, 15
13	(IV)	»	100	trans-1-Bromo-2-methoxycyclohexane, 82
14	(Ia)	NaI	6	Not measured
15	(Ia)	NaCl	96	1-Methoxy-1-phenyl-2-chloroethane, 68; 1,2-dimethoxy-1-phenylethane, 30

*Twenty mmoles of olefin and 30 mmoles of electrolyte in 20 ml of methanol, Pt anode, brass cathode, 9.0 F of electricity passed through per mole of olefin, current density 220 mA/cm^2. Temperature (60 ± 2)°C.

†30°C.

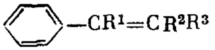
‡18 F of electricity passed through per mole of olefin.

We have previously reported the selective electrochemical bromoalkoxylation of olefins [8]. We have now found that electrolysis of conjugated arylolefins in alcohols in the presence of NaBr gives α-bromoketals directly.*



(Ia-e) (IIa-e)

$R^1 = R^2 = H$ (a); $R^1 = $ 4-Me, $R^2 = H$ (b); $R^1 = $ 3-Cl, $R^2 = H$ (c); $R^1 = H$, $R^2 = Me$ (d); $R^1 = $ 4-OMe, $R^2 = Me$ (e).



$C_6H_5{-}CR^1{=}CR^2R^3$

(IIIa-c)

$R^1 = Me$, $R^2 = R^3 = H$ (a); $R^1 = H$, $R^2 = R^3 = Me$ (b); $R^1 + R^2 = (CH_2)_4$, $R^3 = H$ (c)

Electrolysis of the styrene (Ia), or its derivatives (Ib-e) and (IIIa-c), and of cyclohexene (IV) in the presence of halide salts in solution in methanol, or in ethylene, propylene, or 1,3-butylene glycol, was carried out in a nondiaphragm, constant-flow cell, varying the conditions in such a way as to obtain the maximum yields of bromoketals (II).

The results (Table 1) show that increasing the temperature favors the oxidation of (Ia) to (IIa), the best alkali metal bromide with NaBr, and in the presence of NH_4Br no (IIa) was formed from (Ia). Nor were α-bromoketals obtained on oxidation of (IIIa-c) or (IV).

The kinetics of formation of oxidation product (Ia) (Fig. 1) show that the immediate precursors of (IIa) are 2-bromo-1-methoxy-1-phenylethane (Va) and α-methoxystyrene (VIa).

These results, taken in conjugation with those previously reported [8], suggest the following reaction mechanism:

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*For previous communication, see [9].

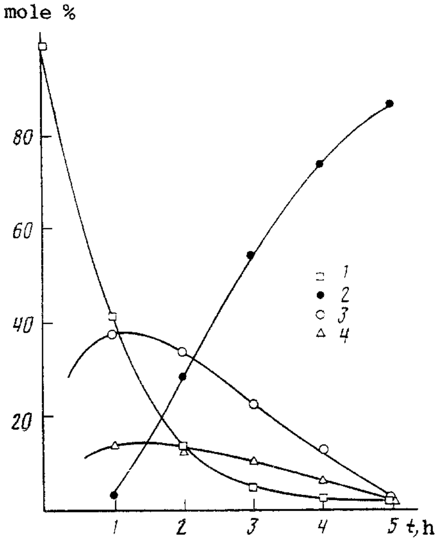
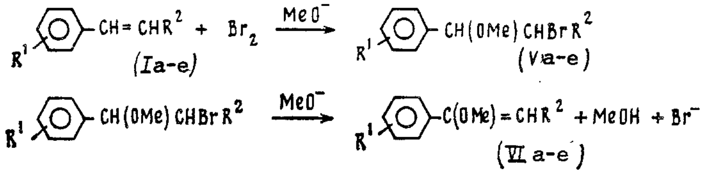


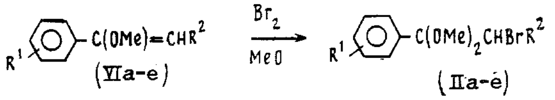
Fig. 1. Kinetics of formation of reaction products, and conversion of styrene in experiment 2, Table 1 (I = 1 A, constant current flow): 1) styrene; 2) bromoketal; 3) bromoether; 4) α-methoxystyrene.

at the anode : $2Br^- - 2e \rightarrow Br_2$
at the cathode: $2M^+ + 2e \rightarrow 2M$ (M = Li, Na, K)
$2M + 2MeOH \rightarrow 2M^+ + 2MeO^- + H_2$
or $2MeOH + 2e \rightarrow 2MeO^- + H_2$

in solution:

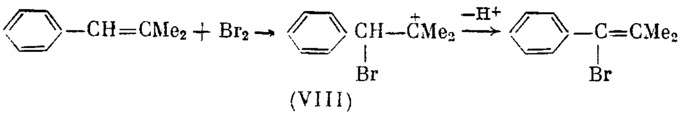


It is noteworthy that MBr is a mediator up to the stage of formation of (VI). Subsequent bromomethoxylation of (VI) under the electrolysis conditions affords (II).



The absence of a benzyl hydrogen atom in the bromomethoxylation products (IIIa, c) excludes the possibility of formation of unsaturated compounds such as (VI) in these two instances, so that the reaction is identical with the previously-examined bromomethoxylation of arylolefins [8], giving predominantly $PhCR^1(OMe)CR^2R^3Br$ (VIIa, c), respectively.

Electrooxidation of 2-methyl-1-phenylprop-1-ene (IIIb) resulted in a different mode of addition of bromine, and the preferred stabilization of the intermediate cation (VIII) was by ejection of a proton.



With cyclohexene (IV), the reaction stopped, as in the case of (IIIa, c), at the bromomethoxylation stage, subsequent dehydrohalogenation did not take place as a result of the absence of a sufficiently labile proton.

The decreased yield of (IIa) when LiBr was used was probably due to the reduced basicity of MeOLi as compared with MeONa. Raising the temperature (60°C instead of 30°C), as would be expected from the proposed mechanism, had a favorable effect on the elimination of HBr to give (VIa). When NaI was used in place of NaBr, the conversion of the (Ia) was only 6%.

TABLE 2. Physicochemical and Spectral Data for Reaction Products

Compound	Bp, °C (p, mm Hg)	PMR spectrum	^{13}C NMR spectrum
		δ, ppm	
(IIa) *	122–123(14)	3.27 s(6H. OCH_3). 3.67 s(2H. CH_2). 7.40 and 7.55 m (5H, C_6H_5)	35.5t (CH_2). 49.3 q (OCH_3). 101.4 s (C), 127.3 d, 127.9 d, 128.3 d, 138.6 s(C_6H_5)
(IIb)*	133–135(14)	2.40 s (3H. CH_3). 3.26 s(6H. OCH_3), 3.67 s (2H. CH_2). 7.30 m (4H, C_6H_4)	21.1 q (CH_3), 35,6 t (CH_2). 49.3 q (OCH_3). 101.4 s (C), 127.8 d, 129.3 d, 135.9 s and 138,0 s (C_6H_4)
(IIc)*	148–149(14)	3.20 s (6H. OCH_3), 3.57 s (2H. CH_2). 7.20. 7.35and 7,51m (4H. C_6H_4)	35.1s (CH_2). 49.5 q (OCH_3). 101.0 s (C). 125.5 d, 127.8 d, 128.7 d, 129.3 d, 134.2 s and 140.9 s (C_6H_4)
(IId)*	125–127(12)	1.53 d (3H. CH_3). 3.23 s (3H. OCH_3), 4.50 q (1H. CH). 7.38. 7.56 m(5H. C_6H_5)	21.3 q (CH_3). 49.0 q and 50.1 q (OCH_3), 50.5d (CH). 103.2 s (C), 127.3 d, 128.4 d, 129.2 d and 135.8 s (C_6H_5)
(IIe) *	162–164(12)	1.45 d (3H. CH_3). 3.13 s (3H. OCH_3). 3.28 s (3H. OCH_3). 3.75 s (3H. *n*-OCH_3). 4.41 q (1H. CH). 6.84 d and 7.39 d(4H, C_6H_4)	21.2 q (CH_3). 45.7 q. 49.9 q. (OCH_3). 50.8 d(CH). 55.2 (*p*-OCH_3). 103.2 s (C). 112.7 d, 130.3 d, 130.6s. 159.6 s (C_6H_4)
2-Bromoethyl-2-phenyl-1,3-dioxolane (Xa)*	150–152(14)	3.68 s (2H. CH_2Br). 3.89 m and 4.18 m (4H, OCH_2). 7.35 m and 7.51 m (5H. C_6H_5)	38.0t (CH_2Br). 64.5 t (OCH_2). 106.9 s (C). 127.8 d, 128.0 d, 128.4d and 139.4 s (C_6H_5)
2-Bromoethyl-2-phenyl-1,3-dioxane (Xb)*	97–99(0.09)	1.25 m and 2.18 m (2H. CH_2). 3,45 s (2H. CH_2Br). 3.75–4.02 m (4H. OCH_2), 7.4 m (5H. C_6H_5)	24.8 t (CH_2). 40.9 t (CH_2Br). 61.4 t (OCH_2). 98.8 s(C). 127.8 d, 128.3 d, 128.7 d and 138.8 s (C_6H_5)
2-Bromoethyl-2-phenyl-4-methyl-1,3-dioxolane (XI)*	113–116(0,01)	1.25 t and 1.28 t(CH_3), 1.25 m and 1.70 m (CH_2), 3.43m and 3.85m (CH_2Br). 3.7–4.1 m (CH_2O and CHO), 7,3–7.6 m (C_6H_5)	21.7 q and 22.0 q (CH_3). 31.8 t and 32.3 t(CH_2). 40.0 and 41.3 t(CH_2Br). 60.2 and 60.7 t (CH_2O), 66.0 d and 37.3 d (CHO), 98.1 s and 99.2 s (C). 127.8. 127.9. 128.3. 128.7. 128.8. 138.7. 138.8 (C_6H_5)
1-(4-Methylphenyl)-1-methoxyethane	82–84(14)	1.43 d (3H. CH_3). 2.59 s (3H. CH_3). 3.22 s (3H. OCH_3), 4.28 q (1H. CH). 7.25 m(4H. C_6H_4)	
1-Bromo-1-phenylprop-1-ene	105–108(12)	1.91 d (3H. CH_3), 5.30 q (1H. CH). 7.35m (5H. C_6H_5)	
2-Bromo-1-methoxy-1-(4-methoxyphenyl)-propane	152–153(12)	1.58 d (3H, CH_3), 3.23 s (3H. OCH_3), 3,74 s (3H. OCH_3), 4.21 m (2H. CH), 6.84 m and 7.20 m (4H. C_6H_4)	20.6 q (CH_3), 52.6 d (CHBr). 55,0 q and 57.2 q (OCH_3), 86,7 d (CHO). 113,5d, 128,5 d, 130.3 s and 159.4 s(C_6H_4)
1-Bromo-2-methoxy-2-phenylpropane*	138–140(26)	1.73 s (3H. CH_3), 3.17 s(3H. OCH_3). 3.59 m (2H, CH_2), 7.37 m (5H, C_6H_5)	21.8 q (CH_3). 42.8 t (CH_2). 50,9 q (OCH_3), 77.7 s (C) 126.3 d, 127.7 d, 128.3 d and141.7 s (C_6H_5)

TABLE 2 (continued)

Compound	Bp, °C (p, mm Hg)	PMR spectrum	^{13}C NMR spectrum
		δ, ppm	
2-Methoxy-2-phenylpropane	82–84(20)	1.57 s(6H, CH_3), 3.10 s(3H, OCH_3), 7.35 m (5H, C_6H_5)	
1-Bromo-2-methyl-1-phenylprop-1-ene*	98–100(12)	1.76 s (3H, CH_3), 2.08 s (3H, CH_3)	22.0 q and 24.8 q (CH_3), 126.1 s 127.7 d, 128.2 d, 129.4 d, 135.5 s and 141.3 s($C_6H_5CBr{=}C$)
2-Methyl-2-methoxy-1-phenylpropane	90–91(12)	1.18 s (6H, CH_3), 2.81 s (2H, CH_2), 3.32 s (3H, OCH_3), 7.32 m (5H, C_6H_5)	25.3 q (CH_3). 46.4 t (CH_2). 49.5 q (OCH_3), 75.5 s (C), 126.6 d, 128.2 d, 129.8 d and 136.9 s (C_6H_5)
trans-1-Bromo-2-methoxy-2-phenylcyclohexane	147–9(22)	1.75 m and 2.08 m (8H, CH_2), 3.01 s (3H, OCH_3), 4.47 m (1H, CHBr), 7.40 m (5H, C_6H_5)	19.8 t, 20.6 t, 23.9 t and 30.8 t (CH_2), 50.6 q (OCH_3). 60.5 d(CHBr). 79.04 s (C). 126.7 d, 127.6 d, 128.0 d and 143.1 s (C_6H_5)
trans-1-Bromo-2-methoxy-1-phenylcyclohexane	137–141(22)	1.75 m and 2.08 m (8H, CH_2). 3.4 s (3H, OCH_3). 4.23 m (1H, CH), 7.42 m (5H, C_6H_5)	
trans-1-Bromo-2-methoxycyclohexane	87–89(12)	1.30 m 1.75 m and 2.23 m (8H, CH_2), 3.22 m (1H, $CHOCH_3$), 3.42 s(3H, CH_3O), 3.96 m(1H, CHBr) cf. [12]	
1-Methoxy-1-phenyl-2-chloroethane*	114–115(18)	3.34s (3H, OCH_3), 3.65m (2H, CH_2), 4.38m (1H, CH). 7.37 m (5H, C_6H_5)	
1,2-Dimethoxy-1-phenyl-2-chloroethane	97–99(16)	3.23 s and 3.33 s (6H, CH_3O), 3.49 m (2H, CH_2), 4.33 m (1H, CH), 7.27 m (C_6H_5)	

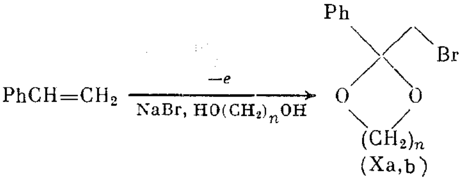
*Composition confirmed by elemental analysis.

In the case of NaCl, the principal oxidation product of (Ia) was 1-methoxy-1-phenyl-2-chloroethane, as a result of the lesser tendency of HCl to undergo elimination as compared with HBr.

The electrooxidation of (Ia) to (IIa) is not sensitive to the anode material, since replacement of Pt by C had little effect on the outcome of the reaction. However, in choosing the cathode material, attention must be paid to possible side reactions. For example, reduction of the intermediate (Va) to α-methoxyethylbenzene (IX) (yields up to 20%) is much enhanced, while at a graphite cathode hydrogenation of the styrene to ethylbenzene became appreciable (yields up to 10%). Even using a brass electrode, which we have found to be the best, at the bromomethoxylation stage reduction of the β-bromoether at the cathode occurs to give the unbrominated ether. This reaction has been discussed in greater detail [8].

Since the Cl^- ion is oxidized at more positive potentials than Br^-, on electrooxidation of (Ia) in the presence of NaCl direct electrochemical oxidation of (Ia) in methanol commences, with the formation of 1,2-dimethoxy-1-phenylethane. A similar reaction has been reported previously [10].

Electrolysis of (Ia) in ethylene and 1,3-propylene glycols in the presence of NaBr proceeds in one step to give the cyclic ketals in yields of 70-80%.



n = 2 (Xa), 3 (Xb).

When the reaction was carried out in 1,3-butylene glycol, a mixture of isomeric 2-bromomethyl-2-phenyl-4-methyl-1,3-dioxanes (XI) in a ratio of 4:3 was obtained in 68% yield. It is likely that the isomer with equatorial methyl and bromomethyl groups is formed preferentially [11].

EXPERIMENTAL

GLC analyses were carried out on an LKhM-8MD chromatograph with a flame ionization detector in a stream of nitrogen, stainless steel columns: 1) 2000 × 3 mm with 15% XF-1150 on Chromosorb W (60-80 mesh); 2) 1000 × 3 mm with 3% OV-1 on Inertone Super (0.16-0.20 mm), and a glass column, 3000 × 3 mm with 5% SE-Superphase on Inertone Super (0.16-0.20 mm).

The PMR spectra of solutions of organic compounds in $CDCl_3$ were recorded on Tesla BS-497 (100 MHz) and Bruker WM-250 (250 MHz) spectrometers, and ^{13}C NMR spectra on a Bruker AM-300 (300 MHz), standard TMS.

Styrene, p-methylstyrene, anethole, α-methylstyrene, and 1-phenylcyclohexene were of reagent grade purity, purified by redistillation under reduced pressure. Sodium bromide (pure grade) and LiBr (pure for analysis) were further dried in vacuo.

m-Chlorostyrene was obtained by reacting m-chlorobenzaldehyde with methylmagnesium iodide and dehydration of the alcohol obtained.

2-Methyl-1-phenylprop-1-ene was obtained by reacting benzyl methyl ketone with methylmagnesium iodide, and dehydration of the resulting alcohol.

Electrochemical Oxidation of Olefins. General Method. In a nondiaphragm cell with external cooling, fitted with a magnetic stirrer, thermometer, and reflux condenser, were placed 20 mmoles of the olefin, 30 mmoles of NaBr, and 20 ml of alcohol (in the case of 1,3-propylene glycol and 1,3-butylene glycol, in admixture with acetonitrile, 3:1), and electrolysis carried out at 60°C, the amount of electricity shown in Table 1 being passed through. The reaction mixture was analyzed by GLC, evaporated, extracted with ether, washed with water, and the ether layer dried over Na_2SO_4, the ether removed, and the residue distilled under reduced pressure to give the products. The conversion of the olefin and the yields of products by GLC are shown in Table 1.

The physicochemical data and spectral characteristics of the products are given in Table 2.

2-Bromo-1-methoxy-1-phenylethane (Va) and 2-bromo-1-methoxy-1-(4-methoxy-phenyl)propane (Ve) were obtained by electrochemical bromomethoxylation of (Ia) and (Ie), respectively, in 90% yield [8].

1-Methoxy-1-phenylethane (IX) and 2-methoxy-2-phenylpropane were obtained by the electrochemical addition of methanol to (Ia) and (IIIa), respectively [8]. Compounds (Va) and (IX) have been reported previously [8].

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MECHANISM OF REACTION OF PHENYL GLYCIDYL ETHER WITH PHENYL BENZOATE IN THE PRESENCE OF WATER

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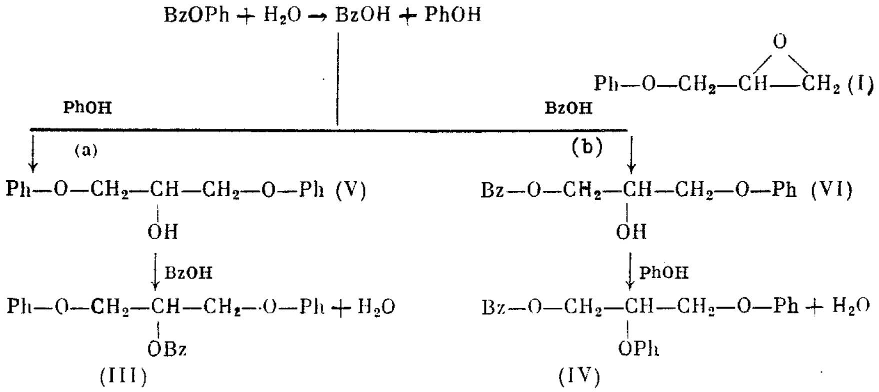
UDC 541.124:542.91:547.474.
3'562:547.581.2'562

The reaction of phenyl glycidyl ether with phenyl benzoate in the presence of water was investigated by adsorption liquid chromatography.

One of the novel reactions of α-oxy compounds that are now being widely studied is their reaction with ester groups [1]. This reaction is finding greater and greater use in the preparation of novel heterochain polymers [2] and also for crosslinking of polyesters [3, 4]. As a result of the reaction, normal cleavage of the α-oxy ring occurs (at the CH_2–O bond) with its subsequent addition at the ester bond. Thus, it has been shown [3, 5] that the reaction of phenyl glycidyl ether (I) with BzOPh (II) affords 1,3-diphenyl-2-benzoylglycerol (III).

In a number of cases [6, 7], the occurrence of polymer-degradation processes due to hydrolysis of ester bonds by sorption moisture has been noted in the reaction of epoxy oligomers with aromatic polyesters (polyarylates) under severe conditions (180°C for 10 h). With model compounds, it has been shown [3] that the reaction of phenyl benzoate (II) with glycerol α-phenyl ether, a product of the hydrolysis of phenyl glycidyl ether (I), does not afford addition product (III) or 1,2-diphenyl-3-benzoylglycerol (IV).

However, another route of the reaction involving water which affords product (III) or (IV) during normal cleavage of the α-oxy ring of phenyl glycidyl ether is possible:



But in the case of anomalous cleavage of the α-oxy ring (at the CH–O bond), the occurrence of the reaction by route (a) in the proposed scheme should afford (IV), and its occurrence by route (b) should afford (III). In addition, in both cases, there is no event of direct addition of an α-oxy ring at the ester bond of phenyl benzoate.

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