Pergamon

SCIENCE DIRECT®

Tetrahedron 59 (2003) 7603–7607

TETRAHEDRON

The role of palladium catalyst and base in stereoselective transformations of (*E*)-2-chlorovinylsulfides

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Received 14 February 2003; revised 26 June 2003; accepted 17 July 2003

Abstract—Stereoselective transformations of 2-chlorovinylsulfides in the presence of soluble (*t*-BuOK) or insoluble (solid KOH or Cs_2CO_3/18-crown-6) base and palladium catalyst (dppb)$Pd(OAc)_2$ have been studied. Depending on the substrate or catalytic system, the reaction leads to the formation of (*E*)-1,2-bis[aryl(or arylmethyl)thio]ethenes and/or (*E*)-1,4-bis[aryl(or arylmethyl)thio]-1-buten-3-ynes in yields of up to 93%.

1. Introduction

The utilization of functionalized unsaturated sulfides in the further synthesis of new organic compounds was been of interest during the last decades.[1–3] However, the synthetic transformations of chlorovinyl sulfides have remained largely unexplored. The first example of chlorovinyl sulfide utilization confirms that the nucleophilic attack of sodium alkyl and aryl thiolates on *cis* and *trans*-chlorovinyl sulfides stereospecifically leads to formation of *cis* and *trans*-dithioethenes, correspondingly.[4a] These reactions occur in HMPA with complete retention of configuration and the products are stable under oxidation of the thio group to a sulfone.[4b] This method was found to be very convenient for the preparation of regular polymers containing thiovinyl fragments by addition of bis(chlorovinyl)arenes to bisthiolates.[5] When the solvent was changed from HMPA to DME or toluene it was necessary to use a catalyst [$(bipy)_2NiBr_2$] and to reflux the reaction mixture to obtain the desired dithioethenes.[6] The alternative way for the introduction of the thio substituent at the double bond is catalytic addition of diaryldisulfides to the C≡C triple bond. Transition metal complexes such as $Pt(PPh_3)_4$, $Pd(PPh_3)_4$, and $Rh(PPh_3)_3Cl$ were used as the catalyst. Pd(II) complexes and $Rh(PPh_3)_3-Cl_2$ did not catalyze the reaction due to poisoning by sulfur.[7] It was found that a rhodium–phosphine complex and trifluoromethanesulfonic acid catalyzed addition of dialkyl disulfides to terminal acetylenes stereospecifically yields (*Z*)-bis(alkylthio)olefins.[8] Polyendiynes were synthesized by metal-mediated coupling reactions of halovinylthioarenes with trimethylsilylacetylene in the presence of a palladium catalyst.[9]

We would like to investigate the transformations of chlorovinylsulfides under basic conditions in the presence or absence of the palladium catalyst. We supposed that ethynyl sulfides would be formed as a result of the dehydrochlorination of chlorovinylsulfides. We expected that in the presence of a Pd catalyst, a Heck reaction would occur, but unexpected results were obtained.

In the current report we present the results of novel nucleophilic transformations of (*E*)-2-chlorovinyl-1-phenyl (2-pyridyl, benzyl, furfuryl) sulfides in the presence of base (*t*-BuOK, KOH) and/or under mixed basic phase transfer–metal complex catalysis conditions. The reaction proceeded in two directions giving (*E*)-1,2-bis[aryl(or arylmethyl)-thio]ethenes and (*E*)-1,4-bis[aryl(or arylmethyl)thio]-1-buten-3-ynes due to the possible carbon–sulfur bond cleavage under basic conditions forming an aryl thiolate anion. The influence of palladium catalyst, the nature of the base and aryl(or hetaryl) substituent in the (*E*)-2-chlorovinylsulfides on the course of the reactions are discussed.

2. Results and discussions

The direction of transformation of (*E*)-2-chlorovinylsulfides strongly depends on the nature of base, presence of palladium catalyst and substrate properties. For investigation of the influence of the base, *t*-BuOK (soluble in the reaction mixture) and KOH and Cs_2CO_3 (insoluble) were chosen. Phase transfer catalysis (PTC) using 18-crown-6 is

Keywords: palladium catalyst; phase transfer catalysis; sulfides; alkenes; enynes.
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0040–4020/$ - see front matter © 2003 Elsevier Ltd. All rights reserved.
doi:10.1016/S0040-4020(03)01125-6

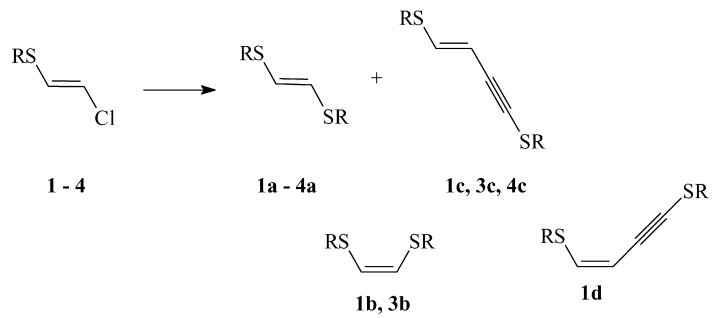
necessary in the case of insoluble bases. High activity of the Pd catalyst with 1,4-bis(diphenylphosphino) butane (dppb) ligand[10] was demonstrated in the reaction of allenylcyclobutanols with aryl halides. Palladium catalyzed reactions of thiols with aryl halides were recently described.[11] Therefore the reaction was carried out in the presence of $Pd(OAc)_2$/dppb and for comparison in the absence of it. (*E*)-2-Chlorovinylthiobenzene (**1**), (*E*)-2-chlorovinyl(2-pyridyl)sulfide (**2**), (*E*)-2-chlorovinyl (benzyl)sulfide (**3**), and (*E*)-2-chlorovinyl(2-furfuryl)sulfide (**4**) were used as the substrates and the results are shown in Table 1.

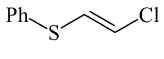
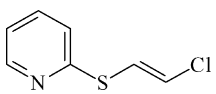
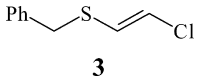
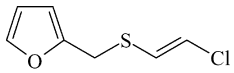
(*E*)-2-Chlorovinylthiobenzene (**1**) in the *t*-BuOK/$Pd(OAc)_2$/dppb/toluene system transforms to (*E*)-1,2-bis(phenylthio)-ethene (**1a**) almost stereoselectively in 70% yield. The similar reaction of alkene **1** in the absence of palladium catalyst affords alkene **1a** in 10% yield along with both isomers of 1,4-bis(phenylthio)-1-buten-3-yne: *E*-isomer (**1c**, 25%) and (*Z*)-isomer (**1d**, 18%). The system of solid KOH and palladium complex gave the mixture of (*E*) and (*Z*)-1,2-bis(phenylthio)ethenes (**1a**) and (**1b**) in 50 and 30% yields, correspondingly. It should be noted that reaction does not take place under PTC conditions in the absence of palladium complex. Application of a weaker base (Cs_2CO_3) accompanied by palladium catalyst lowers the yield of product **1a** (10%).

The mixed PTC–metal complex catalytic system is preferable for the transformation of (*E*)-2-chlorovinyl(2-pyridyl)sulfide (**2**). (*E*)-1,2-Bis[(2-pyridyl)thio]ethene **2a** was obtained in 70% yield in the solid KOH/18-crown-6/$Pd(OAc)_2$/dppb/toluene system. Cesium carbonate was practically inactive as a base under PTC conditions and led to the formation of ethene **2a** in 10% yield. In the presence of *t*-BuOK and (dppb)$Pd(OAc)_2$ the yield of product **2a** is 56%, but without palladium complex (*E*)-2-chlorovinyl(2-pyridyl)sulfide (**2**) completely decomposes. Thus, the mild PTC conditions seem appropriate for the pyridyl derivative **2** successful conversions.

It was found that the system *t*-BuOK/$Pd(OAc)_2$/dppb/toluene is the most favorable for (*E*)-2-chlorovinyl(benzyl)-sulfide (**3**) transformation. The desired product (*E*)-1,2-bis(benzylthio)ethene (**3a**) is stereospecifically obtained in 86% yield. The change of *t*-BuOK to solid KOH/18-crown-6 decreases the yield of product **3a** to 65%. Using of Cs_2CO_3

Table 1. Transformation of (*E*)-2-chlorovinylsulfides



Substrate	System	Time (h)	Isolated yield (%)[a]			
			a	**b**	**c**	**d**
 **1**	KOH/18-crown-6	42	Traces			
	t-BuOK	20	10	Traces	25	10
	KOH/18-crown-6/$Pd(OAc)_2$/dppb	43	50	30		
	t-BuOK/$Pd(OAc)_2$/dppb	20	70	Traces		
	Cs_2CO_3/18-crown-6/$Pd(OAc)_2$/dppb	42	10			
 **2**	*t*-BuOK	20		[b]		
	KOH/18-crown-6/$Pd(OAc)_2$/dppb	44	70			
	t-BuOK/$Pd(OAc)_2$/dppb	48	56			
	Cs_2CO_3/18-crown-6/$Pd(OAc)_2$/dppb	44	10			
 **3**	*t*-BuOK	20	65	8	22	
	KOH/18-crown-6/$Pd(OAc)_2$/dppb	43	65	Traces		
	t-BuOK/$Pd(OAc)_2$/dppb	20	86	Traces	9	
	Cs_2CO_3/18-crown-6/$Pd(OAc)_2$/dppb	42	20	Traces		
 **4**	*t*-BuOK	20			57	
	KOH/18-crown-6/$Pd(OAc)_2$/dppb	43	Traces		26	
	t-BuOK/$Pd(OAc)_2$/dppb	20	5		60	
	Cs_2CO_3/18-crown-6/$Pd(OAc)_2$/dppb	42			8	

[a] Compounds **1a**,**b** and **3a**,**b** have been described.[12]
[b] Polymerization.

gives (*E*)-1,2-bis(benzylthio)ethene in 20% yield. The stereoselectivity of the reaction and yield of the major product **3a** in the absence of palladium catalysts are also considerably lower. Moreover, (*E*)-1,4-bis(benzylthio)-1-buten-3-yne **3c** [MS, *m/z* (I, %): 296 (M^+, 17), 173 (M^+-PhCH_2S, 100)] is obtained in 22% yield using only *t*-BuOK. Thus, the introduction of the methylene group between the phenyl ring and the sulfur atom in chlorovinylthiosulfides strongly decreases the influence of the aromatic ring to the thiovinyl fragment. As a result the stereoselectivity and the overall yield are higher in the case of benzyl derivative **3** in comparison with phenyl analogue **1**.

Inspection of the experimental data in the case of (*E*)-2-chlorovinyl(2-furfuryl)sulfide (**4**) shows that the transformation proceeds without catalyst. The conversion of sulfide **4** in the presence of soluble *t*-BuOK in refluxing toluene unexpectedly leads to the formation of (*E*)-1,4-bis(2-furfurylthio)-1-buten-3-yne (**4c**) in 57% yield. The addition of palladium catalyst to the reaction mixture slightly increases the yield of enyne **4c**, but simultaneously it diminishes the selectivity of the process: the formation of (*E*)-1,2-bis(2-furfurylthio)ethene (**4a**) as minor product in 5% yield was detected. Using solid KOH accompanied by $(dppb)Pd(OAc)_2$ in the transformation of sulfide **4** was less effective and led to enyne **4c** in considerably lower yield (26%). Cs_2CO_3 was practically ineffective.

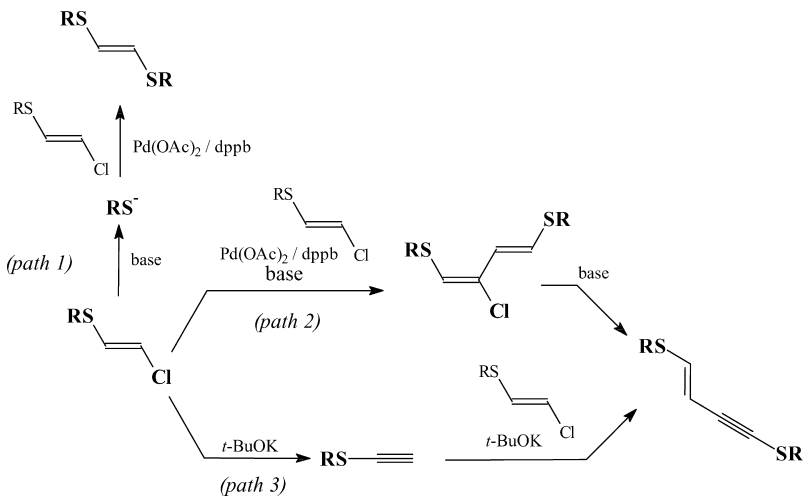
The experimental data suggest that the process of transformation of (*E*)-2-chlorovinylsulfides can be explained in three general ways (Scheme 1). The first route includes the interaction of (*E*)-2-chlorovinylsulfides with a solid base (*t*-BuOK or KOH/18-crown-6) leading to the formation of thiolate anion (RS^-). Then the second molecule of (*E*)-2-chlorovinylsulfide (RSCH=CHCl) in the presence $(dppb)Pd(OAc)_2$ interacts with thiolate anion giving the final dithioalkenes RSCH=CHSR (path 1). The selectivity of alkene formation (RSCH=CHSR) usually decreased if the conversion of RSCH=CHCl occurred in the absence of palladium catalyst (see Table 1, phenyl (**1**) and benzyl (**3**) derivatives in reaction with *t*-BuOK).

The second route is based on a palladium catalyzed Heck reaction between two molecules of (*E*)-2-chlorovinylsulfides leading to diene intermediate RSCH=CCl–CH=CHSR with the following base mediated elimination of HCl yielding the desired enynes (path 2).

The alternative pathway for the formation of enynes **1c**,**d**, **3c**, **4c** is the reaction of RSCH=CHCl (R=Ph, $PhCH_2$, 2-furfuryl) with *t*-BuOK in the absence of palladium catalyst (path 3). In this case the β-elimination of HCl from (*E*)-2-chlorovinylsulfides occurred leading to ethynylsulfides. The latter undergoes nucleophilic substitution with the second molecule of RSCH=CHCl giving enynes **1c**,**d**, **3c**, **4c**.

It should be concluded that phenyl, pyridyl, and benzyl thiochloroethenes **1**–**3** are transformed into the corresponding (*E*)-1,2-bis(arylthio)ethenes as a major product under basic conditions in the presence of palladium complex. The absence of the latter leads to the appearance of enynes **1c**,**d**, **3c** indicating that path 3 also takes place. Transformation of (*E*)-2-chlorovinyl(2-furfuryl)sulfide (**4**) practically afforded (*E*)-1,4-bis(2-furfurylthio)-1-buten-3-yne (**4c**).

For the understanding of the difference in the transformations of our substrates the quantum chemical calculations of (*E*)-2-chlorovinyl(benzyl)sulfide (**3**) and (*E*)-2-chlorovinyl(2-furfuryl)sulfide (**4**) structures were realized. Semiempirical AM1 method was used.[14] The charges calculated for the S atom for both compounds were similar (0.238 e for **3**, and 0.243 e for **4**). The σ-component of density matrix is also equal: 1.837. However, there is the difference in the distribution of the π-electron density on S atom in these compounds. The p_x component in the both compounds has the minimal value (0.973 for **3**, and 0.964 for **4**), but it is lying in the chlorovinyl substituent plane being sterically hindered. Probably the nucleophilic attack on the S atom in (*E*)-2-chlorovinyl(benzyl)sulfide (**3**) occurs in direction of the second minimal component—p_y (1.256). On contrary, in the (*E*)-2-chlorovinyl(2-furfuryl)sulfide (**4**) the minimal p_z (1.071) component is located



Scheme 1. Mechanism of the transformation of 2-chlorovinylsulfides.

parallel to furan ring being practically inaccessible. It could explain the lower probability of thiolate anion formation in the case of (*E*)-2-chlorovinyl(2-furfuryl)-sulfide as we propose.

3. Experimental

3.1. General

^{1}H and ^{13}C NMR spectra were recorded on a Varian Mercury 200 spectrometer at 200.06 and 50.31 MHz correspondingly at 303 K. The chemical shifts are given relative to hexamethyldisiloxane (HDMSO, δ_H=0.055) from solvent ($CDCl_3$) signal (δ_H=7.25). Mass spectra were recorded on a GC–MS HP 6890 (70 eV). GC analysis was performed on a Chrom-5 instrument equipped with flame-ionization detector using glass column packed with 5% OV-101/Chromosorb W-HP (80–100 mesh) (1.2 m×3 mm).

(*E*)-2-Chlorovinylthio derivatives **1**–**4** were prepared from corresponding thiols and $ClCH_2CHCl_2$ using solid K_2CO_3 (then KOH)/18-crown-6/toluene or xylene as described.[13]

3.2. Transformation of (*E*)-2-chlorovinylsulfides by *t*-BuOK

To the solution of substrate **1**–**4** (1 mmol) in dry toluene (2 mL) under argon atmosphere *t*-BuOK (137 mg, 1.4 mmol) was added. The reaction mixture was stirred at 110°C up to substrate disappearance (GC control). Then the precipitate (KCl) was filtered off, and toluene was evaporated under reduced pressure. The crude residue was purified by column chromatography on silica gel using hexane/toluene or hexane/ethyl acetate as eluent.

3.3. Transformation of (*E*)-2-chlorovinylsulfides in the system KOH/18-crown-6/$Pd(OAc)_2$/dppb

The substrate **1**–**4** (1 mmol) was added to the mixture of $Pd(OAc)_2$ (4.5 mg, 0.02 mmol) and 1,4-bis(diphenylphosphino)butane (dppb) (21.3 mg, 0.05 mmol) in dry toluene (2 mL) under argon atmosphere. After five-minute stirring KOH (78.4 mg, 1.4 mmol) and 18-crown-6 (26 mg, 0.1 mmol) were added. The reaction mixture was stirred at 110°C up to substrate disappearance (GC control), filtered; solvent was removed under reduced pressure. The crude residue was purified on silica gel using hexane/toluene or hexane/ethyl acetate as eluent.

3.4. Transformation of (*E*)-2-chlorovinylsulfides in the system *t*-BuOK/$Pd(OAc)_2$/dppb

The substrate **1**–**4** (1 mmol) was added to the mixture of $Pd(OAc)_2$ (4.5 mg, 0.02 mmol) and 1,4-bis(diphenylphosphino)butane (dppb) (21.3 mg, 0.05 mmol) in dry toluene (2 mL) under argon atmosphere. After five-minute stirring *t*-BuOK (137 mg, 1.4 mmol) was added. Reaction mixture was stirred at 110°C up to substrate disappearance (GC control), filtered; solvent was removed under reduced pressure. The crude residue was purified by column chromatography on silica gel using hexane/toluene or hexane/ethyl acetate as eluent.

3.5. Transformation of (*E*)-2-chlorovinylsulfides in the system Cs_2CO_3/18-crown-6/$Pd(OAc)_2$/dppb

The substrate **1**–**4** (1 mmol) was added to the mixture of $Pd(OAc)_2$ (4.5 mg, 0.02 mmol) and 1,4-bis(diphenylphosphino)butane (dppb) (21.3 mg, 0.05 mmol) in dry toluene (2 mL) under argon atmosphere. After five-minute stirring Cs_2CO_3 (456 mg, 1.4 mmol) and 18-crown-6 (26 mg, 0.1 mmol) were added. The reaction mixture was stirred at 110°C up to substrate disappearance (GC control), filtered; solvent was removed under reduced pressure. The crude residue was purified on silica gel using hexane/toluene or hexane/ethyl acetate as eluent.

3.5.1. (*E*)-1,4-Bis(phenylthio)-1-buten-3-yne (1c). Oil; R_f 0.5 (SiO_2, hexane/toluene 10:1). MS, *m*/*z* (I, %): 268 (M^+, 88), 235 (34), 234 (68), 202 (18), 191 (40), 190 (47), 158 (20), 147 (74), 121 (40), 115 (84), 114 (27), 102 (18), 89 (20), 77 (61), 69 (50), 51 (100), 39 (23). ^{1}H NMR δ ppm: 5.73 (d, 1H, *J*=16 Hz, =CH), 6.94 (d, 1H, *J*=16 Hz, =CH), 7.15–7.46 (m, 10H, Ph). ^{13}C NMR δ ppm: 85.7 (≡C), 95.8 (≡C), 107.4 (=C), 125.3, 126.1, 126.4, 126.7, 128.1, 129.1, 131.5, 132.9, 139.8 (=C). Anal. calcd for $C_{16}H_{12}S_2$: C, 71.60; H, 4.51; S, 23.8. Found: C, 71.49; H, 4.56; S, 24.04.

3.5.2. (*Z*)-1,4-Bis(phenylthio)-1-buten-3-yne (1d). Oil, R_f 0.67 (SiO_2, hexane/toluene 10:1). MS, *m*/*z* (I, %): 268 (M^+, 70), 235 (35), 234 (55), 221 (10), 202 (12), 191 (31), 190 (45), 159 (15), 158 (20), 147 (60), 146 (20), 145 (37), 115 (83), 114 (26), 103 (14), 102 (16), 89 (20), 82 (32), 77 (63), 69 (52), 51 (100), 50 (28), 35 (35), 39 (24). ^{1}H NMR δ ppm: 5.80 (d, 1H, *J*=10 Hz, =CH), 6.75 (d, 1H, *J*=10 Hz, =CH), 7.17–7.53 (m, 10H, Ph). ^{13}C NMR δ ppm: 83.9 (≡C), 94.3 (≡C), 105.9 (=C), 124.9, 126.1, 126.4, 127.5, 129.1, 129.2, 130.3, 134.4, 139.2 (=C). Anal. calcd for $C_{16}H_{12}S_2$: C, 71.60; H, 4.51; S, 23.8. Found: C, 71.57; H, 4.50; S, 24.06.

3.5.3. (*E*)-1,4-(*E*)-Bis[(2-pyridyl)thio]ethene (2a). Oil, R_f 0.53 (SiO_2, hexane/ethyl acetate 2:1). MS, *m*/*z* (I, %): 246 (M^+, <1), 136 (100), 78 (27), 67 (10), 51 (17). ^{1}H NMR δ ppm: 7.04 (m, 2H, H-5, H-5′), 7.21 (m, 4H, H-4, H-4′, CH=CH), 7.49–7.58 (m, 2H, H-3, H-3′), 8.45 (m, 2H, H-6, H-6′). ^{13}C NMR δ ppm: 120.3 (=C), 121.7, 122.3, 136.5, 149.7, 157.6. Anal. calcd for $C_{12}H_{10}N_2S_2$: C, 58.51; H, 4.09; N, 11.37; S, 26.03. Found: C, 58.64; H, 4.13; N, 11.40; S, 26.13.

3.5.4. (*E*)-1,2-Bis(2-furfurylthio)ethene (4a). Oil, R_f 0.54 (SiO_2, hexane/ethyl acetate 10:1). MS, *m*/*z* (I, %): 252 (M^+, 10), 81 (93), 53 (36). ^{1}H NMR δ ppm: 6.02 (s, 2H, HC=CH), 6.20 (m, 2H, H-3, H-4), 7.29 (m, 1H, H-5). ^{13}C NMR δ ppm: 11.9, 103.9 (C=C), 108.6, 111.2, 132.1. Anal. calcd for $C_{12}H_{12}O_2S_2$: C, 57.11; H, 4.79; S, 25.41. Found: C, 57.07; H, 4.74; S, 25.51.

3.5.5. (*E*)-1,4-Bis(2-furfurylthio)-1-buten-3-yne (4c). Oil, R_f 0.54 (SiO_2, hexane/ethyl acetate 10:1). MS, *m*/*z* (I, %): 276 (M^+, 100), 137 (45), 106 (15), 94 (12), 78 (10), 65 (15), 51 (18), 43 (10). ^{1}H NMR δ ppm: 6.11 (d, 1H, *J*=13 Hz,

=CH), 6.21 (m, 1H, H-4), 6.34 (m, 2H, H-3″, H-4″), 6.39 (d, 1H, *J*=13 Hz, =CH), 6.63 (m, 1H, H-3), 7.23 (m, 1H, H-5), 7.36 (m, 1H, H-5′). ^{13}C NMR δ ppm: 13.1, 86.2 (≡C), 94.0 (≡C), 100.5 (=C), 104.3, 108.6, 110.4, 111.2, 117.5 (=C), 140.5, 147.8, 148.9. Anal. calcd for $C_{14}H_{12}O_2S_2$: C, 60.84; H, 4.38; S, 23.20. Found: C, 60.80; H, 4.36; S, 23.24.

Acknowledgements

The authors thank Latvian Council of Science for the financial support (Grant No. 166).

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