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The application of Pd-complexes of diphenylphosphinoferrocenyloxazoline ligands to catalytic enantioselective allylic amination

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Abstract

The preparation of cationic palladium (η^3-C_3H_5) complexes, possessing an enantiopure diphenylphosphinoferrocenyloxazoline unit, is described. These complexes were applied to the enantioselective amination of the test substrate, ethyl (2*E*)-1,3-diphenylprop-2-enyl carbonate, with benzylamine in moderate to high conversions with enantioselectivities of up to 72% for the (*S*)-valinol derived oxazoline complex. Enantioselectivities of up to 37% were obtained for the (*S*)-*tert*-leucinol derived oxazoline complex.

Keywords: Allylic amination; Diphenylphosphinoferrocenyloxazoline ligands; Palladium catalysis

1. Introduction

The enantioselective alkylation of allylic acetates is a carbon–carbon bond forming process catalysed by transition metal complexes of palladium and has been the subject of several recent reviews [1–5]. It is an important transformation and has proved to be a useful testing ground for the design of new ligands and for gaining mechanistic insights into organopalladium chemistry [6]. Although less well-studied, significant progress has been made recently with the analogous allylic amination process using palladium complexes previously successful in allylic alkylation [7,8]. A selection of the ligands employed in allylic amination, with the relevant ee values obtained in the standard reaction between benzylamine and ethyl (2*E*)-1,3-diphenylprop-2-enyl carbonate, is shown in Fig. 1. The ferrocenylphosphine ligand **1** tested by Hayashi and Ito gave up to 97% ee [9]. Since that report numerous examples of mixed phosphorous–nitrogen bidentate ligands have been employed and tested with success in allylic amination. Such ligands induce asymmetry through a combination of steric and electronic interactions. Pfaltz and Helmchen obtained 94% ee with the phenylglycine-derived diphenylphosphinooxazoline (**2**) [10] and Saigo reported ees up to 97% with related ligands **3** [11]. Pregosin applied the binaphthalene-based diphenylphosphinooxazoline (**4**) with ees up to 99% [12]. The pyrazole-containing ferrocenylphosphinamine ligand **5** was reported by Togni to afford similarly excellent ees of up to 99% [13,14] and they also noted a remarkable anion effect with this system [15]. Recently Buono has tested the quinolinephosphine (**6**) and obtained up to 93% ee [16].

Part of our research programme is devoted to the design and application in asymmetric catalysis of bidentate phosphinamine ligands [17–22]. One of the ligand classes, which ourselves, and others have prepared, is the diphenylphosphinoferrocenyl oxazolines, represented by **7** and **8** [23–25], and these have been successfully applied in asymmetric catalysis by a range of research groups [26]. We have recently used ligands **7** and **8** in the intermolecular asymmetric phenylation [27] and cyclohexenylation [28] of 2,2-dimethyl-2,3-dihydrofuran and obtained ees of up to 98%. We have also applied these ligands in a synthetic and mechanistic study in palladium-catalysed allylic alkylations in which we obtained up to 92% ee [29]. We now report the application of their palladium complexes to enantio-

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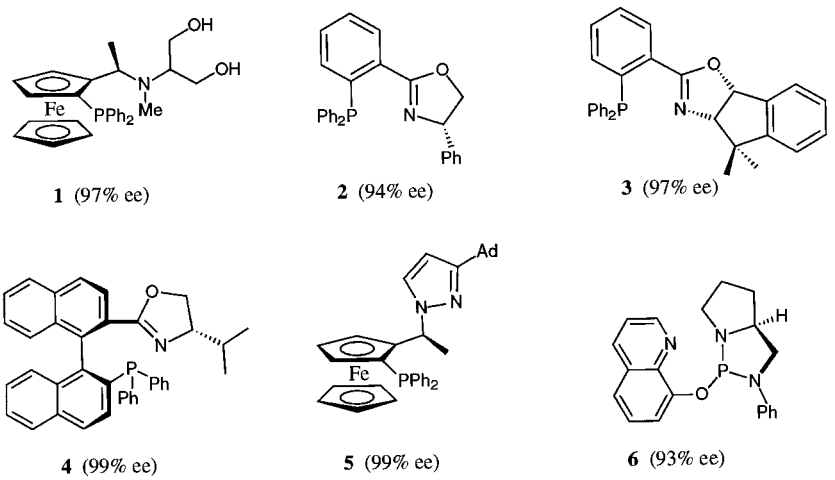
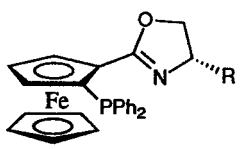


1 (97% ee) **2** (94% ee) **3** (97% ee)

4 (99% ee) **5** (99% ee) **6** (93% ee)

Fig. 1.

selective allylic amination of ethyl (2*E*)-1,3-diphenyl-prop-2-enyl carbonate.



7 R = *i*-Pr

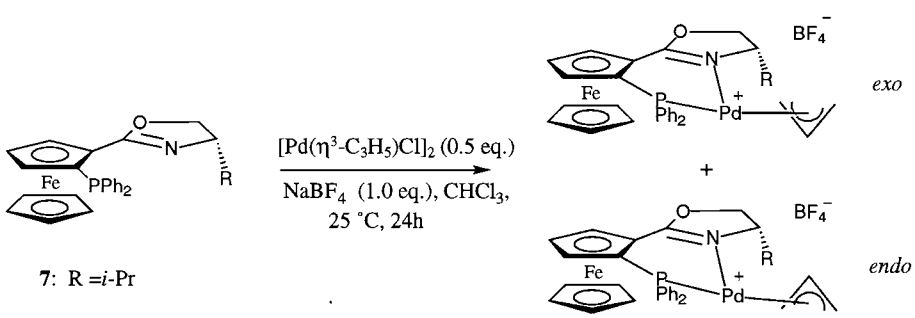
8 R = *t*-Bu

2. Results and discussion

In performing allylic substitutions it is possible to use as the catalyst either pre-formed palladium (η^3-allyl) complexes of the ligand or to assume such complexes are formed when a slight excess of ligand is added to either $Pd_2(dba)_3$ or a η^3-allyl palladium chloride dimer. We prefer to use the pre-formed catalyst and hence we prepared the air-stable η^3-allyl palladium tetrafluoroborate salts (**9** and **10**), in 91 and 90% yields, respectively (Scheme 1).

The structure of [{1-[4-(*S*)-*iso*-propyl-2-oxazolin-2-yl]-2-(*S*)-(diphenylphosphino)ferrocene}-[π-allyl]-palladium] tetrafluoroborate (**9**), prepared from the reaction of di-μ-chloro-bis(π-allyl)dipalladium, 1-[4-(*S*)-*iso*-propyl-2-oxazolin-2-yl]-2-(*S*)-(diphenylphosphino)-ferrocene (**7**) and sodium tetrafluoroborate, was confirmed by spectroscopy. The high field ^{1}H-NMR spectrum was broad and showed the presence of only one unsubstituted cyclopentadienyl ring at 3.83 ppm indicating the existence of one of the two possible diastereomeric *endo*- and *exo*-intermediates in solution [nomenclature note: the *exo*-isomer is defined as that allyl configuration in which the central allyl proton points in the same direction as the C-4 substituent of the oxazoline ring]. ^{31}P-NMR showed a single peak at 17.5 ppm, which confirmed the presence of a single diastereomer.

The *tert*-butyl-substituted analogue, [{1-[4-(*S*)-*tert*-butyl-2-oxazolin-2-yl]-2-(*S*)-(diphenylphosphino) ferrocene}-[π-allyl]palladium] tetrafluoroborate (**10**), was prepared in a similar manner. However, in this case the ^{1}H-NMR spectrum indicated the presence of both the *exo*- and *endo*-intermediates in a 12:1 ratio. As with



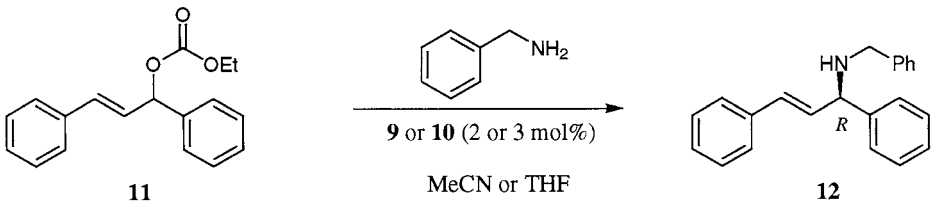
7: R = *i*-Pr

8: R = *t*-Bu

9: R = *i*-Pr (91%) *exo* or *endo*

10: R = *t*-Bu (90%) *exo* and *endo*

Scheme 1.



11 12

Scheme 2.

Table 1
Application of Pd-complexes **9** and **10** to the asymmetric allylic amination of carbonate **11**

Entry	Catalyst (mol%)	Solvent	Time (d)	Temperature (°C)	% Yield [a]	% ee [b] (configuration [c])
1	**9** (2)	CH_3CN	5.5	25	90	16 (*R*)
2	**9** (2)	CH_3CN	5.5	0	70	18 (*R*)
3	**9** (3)	THF	5.5	0	75	72 (*R*)
4	**9** (3)	THF	5.5	25	83	50 (*R*)
5	**9** (3)	THF	8	0	94	63 (*R*)
6	**10** (2)	CH_3CN	3	0	50	18 (*R*)
7	**10** (2)	CH_3CN	4.5	40	90	11 (*R*)
8	**10** (3)	THF	5.5	25	87	37 (*R*)
9	**10** (3)	THF	4.5	40	89	27 (*R*)
10	**10** (2)	THF	8	60	80	22 (*R*)

[a] Isolated yields.
[b] Enantiomeric excesses were determined by HPLC on a Daicel Chiracel OJ column: *n*-hexane–2-propanol 87:13; $T = 25$°C; v/v: 0.5 ml min^{-1} ($t_R = 18.3$ (*S*) and 20.9 (*R*) min) for amine **12**.
[c] Assignment is based on the sign of the optical rotation and comparison with Togni's work [13].

complex **9** we are unable to unambiguously assign the allyl cofiguration in this mixture although in a related mechanistic study we have successfully assigned the allyl configurations of the corresponding 1,3-diphenylallyl complexes [29]. The unsubstituted cyclopentadienyl ring of the major diastereomer appeared as a singlet at 3.79 ppm whilst the corresponding peak occurred at 3.85 ppm in the minor diastereomer. Further evidence for the existence of two diastereomers was obtained from ^{31}P-NMR which showed peaks at 17.1 and 19.0 ppm.

Once prepared, we wished to test the asymmetry-inducing ability of ligands **7** and **8** in what has become one of the standard test reactions in allylic amination, namely that between benzylamine and ethyl (2*E*)-1,3-diphenylprop-2-enyl carbonate (**11**) (Scheme 2). The use of diastereomeric allyl mixtures is acceptable in allylic substitutions as the initial allyl is removed from the metal by nucleophilic attack and subsequent olefin dissociation. This generates a Pd(0) species which then associates and oxidatively adds racemic allylic carbonate (**11**), giving diastereomeric 1,3-diphenylallyl intermediates which readily interconvert, thus allowing racemic allylic carbonate the possibility to afford enantiomerically enriched product. The results of our allylic amination studies are given in Table 1.

The *iso*-propyl substituted diphenylphosphinoferrocenyloxazoline complex **9** was first tested in acetonitrile, the best solvent for the corresponding alkylation [29]. At ambient temperature the chemical yield of the product amine (**12**) was high (90%) but the ee a disappointingly low 16%, favouring the (*R*)-enantiomer. This was increased slightly by lowering the temperature but a lower yield of 70% was obtained (entry 2). Changing the solvent to THF, the solvent of choice in previous amination studies [10,13] gave similarly good chemical yields but improved ees. Our optimal ee obtained was 72% when the reaction was carried out at 0°C in THF (entry 3). Carrying out the reaction at ambient temperature gave a higher yield but lowered ee of 50% (entry 4). Increasing the reaction time at 0°C gave **12** in 63% ee and 94% yield. The use of acetonitrile as solvent with the bulkier *tert*-butyl containing palladium complex **10** gave poor ees of 8–11% in moderate to good yield (entries 6, 7). A change of solvent again to THF proved beneficial in terms of reactivity with yields of 80–89% being observed. However, the best ee obtained with complex **10** in THF was 37% (entry 8). The palladium complexes of the analogous diphenylphosphinooxazolines (**2**) [$R = i$-Pr, t-Bu] gave higher ees of 87 and 88%, respectively, again favouring the (*R*)-enantiomer [10].

A model for the reaction transition-state to explain the preferred formation of the (*R*)-enantiomer in our work can be proposed. The model is based on the mechanistic studies of Brown [30], Pfaltz [31], Togni [32] and Helmchen [33] on the related allylic alkylation

employing palladium phosphinamine complexes. They suggest that the ground state of of the 1,3-diphenylallyl complex contains an allyl which has reoriented itself into a product-like geometry, thus facilitating nucleophilic attack on that allyl terminal carbon *trans* to the phosphorus donor atom. The labilty of the 1,3-diphenylallyl complexes is influenced by such electronic factors and also by intra-complex steric clashes between the ligand and the allyl. Attack *trans* to phosphorus of the *endo*-diastereomer **13a** engenders unwanted steric interactions between the oxazoline C-4 substituent and the phenyl group of the diphenylallyl as it rolls into a product-like geometry to afford (*S*)-**12** after olefin dissoaciation, Scheme 3. Attack *trans* to phosphorus of the *exo*-diastereomer (**13b**), formed from **13a** via a π–σ–π mechanism, is not as sterically disfavoured thus leading to the enantiomer formed in excess, (*R*)-**12**.

It is clear from our amination results that these ligands do not afford enantioselectivities as high as those observed in the analogous alkylations. In both amination and alkylation we assume that the ligand electronic disparity favours attack of the nucleophile at the allylic carbon *trans* to phosphorus. Therefore, the different enantioselectivities must be due to the different reactivities of the nucleophiles involved with the diastereomeric intermediates (**13a** and **13b**), with benzylamine clearly being less selective.

In conclusion, we have prepared new palladium complexes of diphenylphosphinoferrocenyloxazoline ligands **7** and **8** and applied them with modest enantioselectivities in a test allylic amination. Further work will be disclosed on their application in allylic alkylation and on our NMR studies of substituted allyl complexes of these ligands [29].

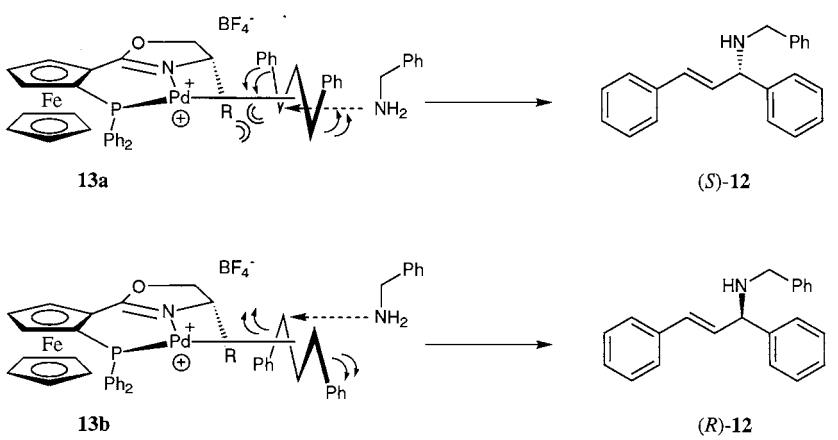
3. Experimental

Solvents were dried immediately before use by distillation from standard drying agents and subjected to degassing by three freeze–thaw cycles. Benzylamine was commercially available (Aldrich Chemical Co.) and was used as purchased. Pd salts were obtained on loan from Johnson Matthey. Di-μ-chloro-bis(π-allyl)dipalladium [34], ligands **7** and **8** [24,25], ethyl-(*E*)-1,3-diphenyl-2-propenyl-carbonate [9] were prepared by literature procedures. Separation by column chromatography was performed using Merck Kieselgel 60 (Art. 7734).

NMR spectra were recorded on a Jeol 270 MHz or a Varian Unity 500 MHz spectrometer. ^{1}H chemical shifts are reported in δ ppm relative to $CHCl_3$ (7.27 ppm), ^{13}C chemical shifts are reported relative to the central peak of $CDCl_3$ (77.0 ppm), and ^{31}P chemical shifts are reported relative to 85% aqueous phosphoric acid (0.0 ppm). Elemental microanalyses were carried out in house using a Carlo Erba 1106 elemental analyser. Electrospray mass spectra were recorded on a VG (Micromass) Quattro with electrospray probe. IR spectra were recorded on a Perkin–Elmer Paragon 1000 FT spectrometer. Optical rotations were recorded on a Perkin–Elmer 241 polarimeter. Melting points were recorded on a Gallenkamp melting point apparatus and are uncorrected.

3.1. Preparation of [{1-[4-(S)-iso-propyl-2-oxazolin-2-yl]-2-(S)-(diphenylphosphino)ferrocene}-[π-allyl] palladium]tetrafluoroborate, (9)

Di-μ-chloro-bis(π-allyl)dipalladium (0.013 g, 0.032 mmol), 1-[4-(*S*)-*iso*-propyl-2-oxazolin-2-yl]-2-(*S*)-(diphenylphosphino)ferrocene (0.036 g, 0.064 mmol), and



Scheme 3.

sodium tetrafluoroborate, (0.022 g, 0.2 mmol), were placed in a Schlenk under nitrogen. Dried and degassed chloroform was added via syringe to give an orange suspension which was stirred at ambient temperature for 24 h. The solid was removed by filtration and the solvent was removed in vacuo to give diastereomerically pure [{1-[4-(*S*)-*iso*-propyl-2-oxazolin-2-yl]-2-(*S*)-(diphenylphosphino)ferrocene} - [π - allyl]palladium] tetrafluoroborate (0.42 g, 91%), as a brown solid, m.p. 118–120°C; $[\alpha]_D^{23} - 35$ (*c* 0.33, chloroform); (Found: C, 51.86; H, 4.46; N,1.81. $C_{31}H_{33}NOPFePdBF_4$ requires C, 52.03; H, 4.62; N, 1.96%); ν_{max} (KBr) 1620 (C=N), 1054 (B–F) cm^{-1}; δ_H (270 MHz, $CDCl_3$) 1.01 (3H, br s, (CH_3)), 1.09 (3H, br s, (CH_3)), 1.7 (1H, br s, allyl), 2.4 (1H, s, $CH(CH_3)_2$), 3.01 (1H, d, *J* 11.7, $–OCH_2$), 3.6 (1H, s, allyl), 3.83 (5H, s, $–C_5H_5$), 4.08 (1H, d, *J* 6.45, $–OCH_2$), 4.33 (br s, allyl), 4.51 (br s, allyl), 4.65 (1H, br s, –Fc), 4.91 (1H, br s, –Fc), 5.18 (1H, br s, –Fc), 5.4 (m, CHN), 5.9 (br s, allyl), 7.08 (2H, br m, Ph), 7.35 (3H, br s, Ph), 7.68 (3H, br s, Ph), 7.9 (2H, br s, Ph); δ_C (67.5 MHz, $CDCl_3$) 15.07 (CH_3), 19.01(CH_3), 30.69 ($CH(CH_3)_2$), 68.8 ($–OCH_2$), 71.96 (C_5H_5), 72.00 (*J* 6.4, *ipso*-Fc), 74.26 (*J* 7.5), 75.56, 75.88, 76.32, 77.24, 128.85, 128.9, 129.44, 129.61, 130.34, 130.95 (*J* 11.8), 132.65, 132.68, 135.04 (*J* 15.1) and 171.2 (C=N); δ_P (109.3 MHz, $CDCl_3$) 17.5, 18.5; *m*/*z* (ESI/pos in CH_3OH) 628 M–BF_4.

3.2. [{1-[4-(S)-tert-butyl-2-oxazolin-2-yl]-2-(S)-(diphenylphosphino)ferrocene}-[π-allyl]palladium] tetrafluoroborate, (10)

Di-μ-chloro-bis(π-allyl)dipalladium (0.008 g, 0.02 mmol), 1-[4-(*S*)-*tert*-butyl-2-oxazolin-2-yl]-2-(*S*)-(diphenylphosphino)ferrocene (0.02 g, 0.04 mmol), and sodium tetrafluoroborate, (0.013 g, 0.121 mmol), were placed in a Schlenk under nitrogen. Dried and degassed chloroform was added via syringe to give an orange suspension which was stirred at ambient temperature for 24 h. The solid was removed by filtration and the solvent was removed in vacuo to give [{1-[4-(*S*)-*tert*-butyl-2-oxazolin-2-yl]-2-(*S*)-(diphenylphosphino) ferrocene}-[π-allyl]palladium] tetrafluoroborate (0.02 g, 90%), as a brown solid, m.p. 150–152°C; (Found: C, 52.41; H, 4.53; N, 1.89. $C_{32}H_{35}NOPFePdBF_4$ requires C, 52.67; H, 4.79; N, 1.92%); ν_{max} (KBr) 1616 (C=N), 1054 (B–F) cm^{-1}; δ_H (270 MHz, $CDCl_3$) *major diastereomer* 1.13 (9H, s, $(CH_3)_3$), 1.7 (1H, br s, allyl), 3.03 (1H, br s, allyl), 3.28 (1H, br s, allyl), 3.79 (5H, s, $–C_5H_5$), 4.03 (1H, br s, allyl), [4.36 (1H, dd, *J* 4.2 9.04), 4.59 (1H, m), 4.62 (1H, br s) $–OCH_2$, CHN], 4.57 (1H, s, –Fc), 4.88 (1H, s, –Fc), 5.19 (1H, s, –Fc), 5.68 (1H, br s, allyl), *minor diastereomer* 1.33 (9H, s, $(CH_3)_3$), 2.88 (1H, br s, allyl), 3.85 (5H, s, $–C_5H_5$), 4.22 (1H, d, *J* 8.9, allyl), [4.39 (1H, s), 4.8 (1H, t) $–OCH_2$, CHN], *major and minor diastereomers* 7.12 (app t, *J* 10.15 7.33), 7.42–7.32 (4H, m), 7.74–7.65 (3H, br m), 8.3–7.86 (2H, v br s, 2H); δ_C (67.5 MHz, $CDCl_3$) 26.47(($CH_3)_3$), 34.33 ($–C(CH_3)_3$), 70.44 ($–OCH_2$), 71.99 (C_5H_5), 74.47, 76.63, 128.81, 128.97, 129.35, 129.52, 130.31, 131.1, 132.89, 135.48; δ_P (109.3 MHz, $CDCl_3$) 17.1, 19.0; *m*/*z* (ESI/pos in CH_3OH) cation 642 M–BF_4.

3.3. General procedure for allylic amination

Ethyl-(2*E*)-1,3-diphenyl-2-propenyl carbonate (0.078 g, 0.276 mmol) in dry and degassed acetonitrile (0.5 ml) was added to a solution of [{1-[4-(*S*)-trisubstituted-2-oxazolin-2-yl]-2-(*S*)-(diphenylphosphino) ferrocene}-[π-allyl]palladium]tetrafluoroborate (2 mol%) in dry and degassed acetonitrile (0.5 ml). To this was added benzylamine (0.037 ml, 0.331 mmol) and the reaction was stirred at the relevant temperature under a nitrogen atmosphere. Reaction progress was monitored by TLC (hexane:ethyl acetate; 3:1 as the eluent). The reaction was purified directly using silica-gel plates (eluent = hexane–ethyl acetate, 3:1) to afford (*R*)-*N*-benzyl-(1,3-diphenyl-1,2-propenyl)amine (**12**) as a clear oil, $R_f = 0.55$ (hexane:ethyl acetate; 3:1); ν_{max} (Nujol); δ_H (270 MHz, $CDCl_3$) 1.77 (1H, br s, NH), 3.77 (2H, AB, *J* 13.4, CH_2Ph), 4.38 (1H, d, $J_{1,2}$ 7.3, H_1), 6.3 (1H, dd, $J_{1,2}$ 7.3 $J_{2,3}$ 15.7, H_2),. 6.57 (1H, d, $J_{2,3}$ 15.8, H_3) and 7.44–7.16 (15H, m, –Ph); *m*/*z* 299 (M^+, 9%), 208 (40), 115 (28), 91 (100) and 77 (30). Enantiomeric excesses were determined by HPLC using a Daicel Chiralcel OJ column and eluting with a hexane–*i*-PrOH mixture (87:13 v/v, 0.5 ml min^{-1}; $T = 25$°C, retention times: (*S*), 18.3 min; (*R*), 20.9 min).

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