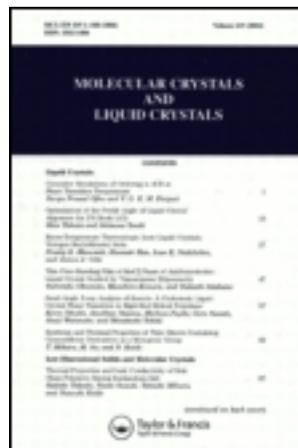


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Azobenzene Liquid Crystals with High Birefringence and Their Effect of Trans-Cis Photoisomerization on Selective Reflection

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A series of four novel azobenzene liquid crystals (LCs) with high birefringence were synthesized by coupling of intermediate molecules with a phenylacetylene fragment. The chemical structures of synthetic intermediates and compounds were confirmed by FT-IR and ¹H-NMR. The LC behavior including transition temperatures and phase sequences were investigated by differential scanning calorimetry (DSC) and polarizing optical microscope (POM). As a very important parameter, the birefringence was also measured using polarizing light interferometry method. Moreover, the characteristics of selective reflection which was associated with the birefringence of the N-LC dropped with azobenzene LCs and the effect of trans-cis photoisomerization of these compounds on selective reflection were studied in detail.*

Keywords Azobenzene; birefringence; isomerization; liquid crystal; selective reflection

1. Introduction

Owing to the rich photochemistry, azobenzene liquid crystals (LCs) are promising materials [1]. They have been extensively studied because of their applicability for photoswitching devices [2, 3], nonlinear optics [4], holography [5], and optical memory [6]. One well-known feature of an azobenzene-containing species was that polarized light could induce reorientation of azobenzene groups through photochemically induced isomerization [7]. In addition, liquid-crystalline polymers with the azobenzene in the side chain have attracted much attention because a photoinduced change in birefringence Δn was large when compared with amorphous counterparts [8, 9]. By contrast, related small molecular compounds have been less explored. The latter have the advantage, among other things, of relatively simple synthesis, being of higher purity, resulting in well-defined structures and corresponding physicochemical properties and providing the option of homogeneously incorporating them into polymeric matrixes [10, 11]. The azobenzene moiety in a trans-form, which is

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rod-like, stabilizes the phase structure of LCs. On the other hand, bent cis-isomers disorganize the phase structure of the LCs [12]. Consequently, trans-cis photoisomerization of the azobenzene moiety in LC compounds led to a large change in Δn by disorganizing the alignment [13].

The Δn is a very important parameter of LCs. LCs with high birefringence at room temperature are useful not only in conventional display devices, such as supertwisted nematic LC displays [14], but also in scattering-type polymer-dispersed LC displays as reflective LC displays and in spatial light modulators [15]. Many high Δn materials have been obtained by increasing the molecular π -electron conjugation length [16] and introducing highly polarizable end groups [17]. Tolane moieties are known as a highly birefringent mesogen because of large π -electron conjugation length and anisotropy of polarizability [18]. Therefore, to obtain larger values of Δn , we synthesized a series of novel azobenzene LCs with tolane or phenyldiacetylene moieties. In addition, we investigated the photoinduced birefringence of the LCs upon exposure to linearly polarized light.

2. Experimental

Reagents were purchased from commercial sources (Aldrich) and used without further purification. Triethylamine (TEA) and tetrahydrofuran (THF) were distilled and purged with argon before use. $^1\text{H-NMR}$ spectra of the samples were recorded with a Bruker DMS-400 spectrometer instrument. Fourier transform infrared spectroscopy (FT-IR, Perkin Elmer Spectrum One) was also measured.

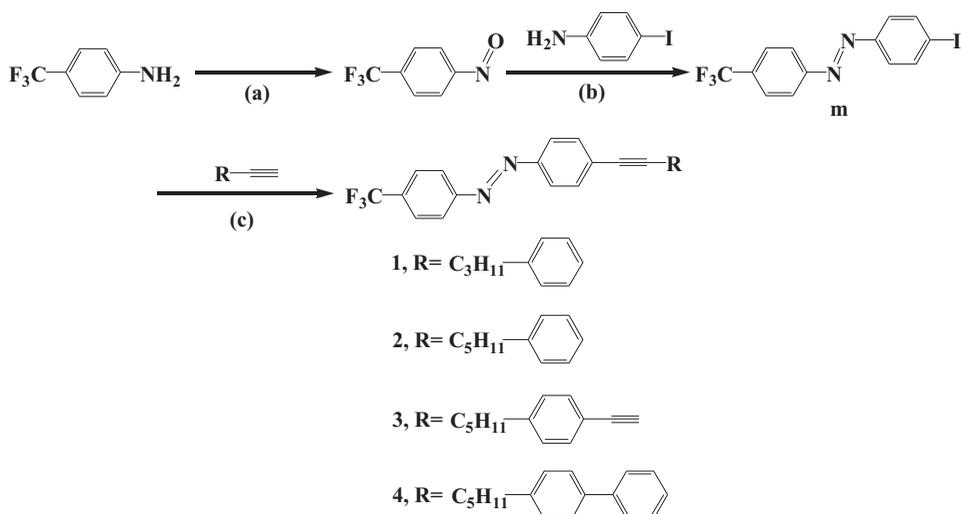
Transition temperatures and phase sequences were measured using a polarizing optical microscope (POM, Olympus BX51) equipped with a hot stage calibrated to an accuracy of ± 0.1 K (Linkam LK-600PM) and then confirmed using differential scanning calorimetry (DSC, Perkin Elmer Pyris 6). The Δn was evaluated using the guest-host method from mixtures containing 10 wt% of each test compound in SLC 1717 (Slichem Liquid Crystal Material Co., Ltd.) and a polarized UV/VIS/NIR spectrophotometer (JASCO V-570) was used to measure the birefringence of the mixtures [19].

The reflectance characteristics were investigated by unpolarized UV/VIS/NIR spectrophotometer (JASCO V-570) in transmission mode at normal incidence. The transmittance of the blank cell was normalized as 100%. The N*-LC was composed of matrix LC, chiral dopant (specific content of 12 wt%) and one of the synthesized compounds. As usual, the matrix nematic LC, SLC1717 and the chiral dopant, and R811 (Merck Co., Ltd.) were used [20]. The $\Delta\lambda$ s were measured from the spectrum by considering respectively the peak bandwidth at half-height.

3. Results and Discussion

3.1. Synthesis

A strategy was proposed on the design of azobenzene-based structures, which consist of three main units: the azobenzene group, the polar terminal substituent, and the π -conjugated tolane or phenyldiacetylene. The polar terminal substituent could both engender the asymmetric structures and influence the intramolecular electron density [20], and the π -conjugated groups could easily extend intramolecular π -conjugation, so as to improve the values of Δn of the LCs. Here, one special design was that the trifluoromethyl group as one polar terminal substituent provided the higher Δn and lower melting point



Scheme 1. Reagents and conditions: (a) Oxone, CH_2Cl_2 , H_2O , r. t., 15 h; (b) AcOH, r. t., 15 h; (c) TAE, THF, $\text{PdCl}_2(\text{PPh}_3)_2$, CuI, PPh_3 , 30°C , 10 h.

[21]. On the basis of the idea mentioned above, a series of azobenzene derivatives with trifluoromethyl groups as terminal substituent have been synthesized that connected with alkylbenzene and alkylphenylacetylenes or alkyl biphenyls linkers. The synthesis of the target compounds was accomplished by 3-step reactions: oxidation procedure (a), azobenzene generation procedure (b), and Hagihara-Sonogashira cross-coupling procedures (c) as showed in Scheme 1. All of above compounds were obtained in good yields and high purities. Their molecular structures and purities were assigned using $^1\text{H-NMR}$ and FT-IR.

3.2. Photophysical Properties

The absorption spectra of the synthesized compounds **2–4** in dilute CH_2Cl_2 solution at room temperature, depicted in Fig. 1, provides some insight into the electronic structure of the systems. All the spectra display a characteristic pattern of two absorption bands between 280 nm and 350 nm. The data were summarized in Table 1. Compound **1** and **2** show the same absorption spectra for the similar molecular structure. Compared with the synthesized compound **2**, compound **4** revealed slight red shifts (~ 10 nm) and compound **3** revealed significant blue shifts (~ 20 nm) of the maxima absorption wavelengths. However, the absorption bands at about 290 nm decreased in the order of **3**, **4**, and **2**. One generally approved explanation was that the maxima absorption wavelengths of azobenzene shifted, when azobenzene was connected with phenylacetylene group in **2** or phenyldiacetylene group in **3** or diphenylacetylene group in **4**.

3.3. Phase Transitions

DSC was used to determine the phase transition temperatures during cooling at a scanning rate of $10^\circ\text{C} \cdot \text{min}^{-1}$, the phase transition temperatures of the compounds were listed in Table 1. It was shown that the compounds **1–4** all exhibit mesomorphism. Moreover, compared with the synthesized compound **2**, compound **1** exhibits a higher melting point

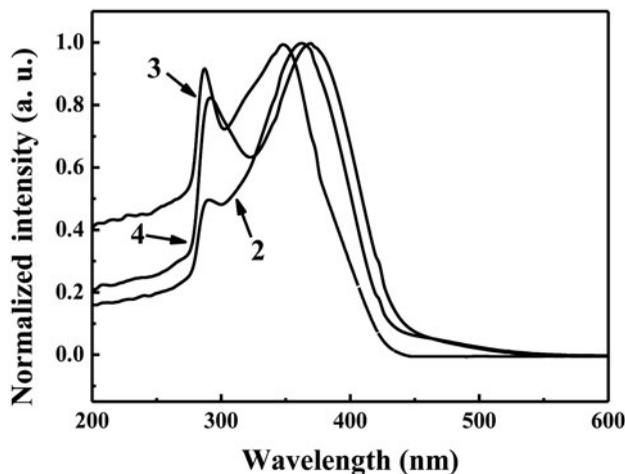


Figure 1. UV-Vis absorption spectra of 2–4 in CH_2Cl_2 under room temperature.

and a narrower temperature range of mesophase, this is because the alkyl carbon number in compound **1** was less than compound **2**. The shorter alkyl chain leads to higher crystallinity. However, in compound **3** and compound **4**, the increasing of π -electron conjugation length was contributed to increase melting point or clearing point and to widen mesophase. It is usually believed that phenyl group possesses richer electron density than alkynyl group [22], so the π -electron conjugation degree of compound **4** are higher than that of compound **3**, that caused an increase the melting point, clearing point, and the temperature range of mesophase.

3.4. Mesomorphic Properties

The mesomorphic properties of the synthesized compounds **1–4** were investigated primarily by POM. The phases were identified through the comparison of the observed textures with reference textures [23] from POM. Figure 2 shows POM photographs of compounds **1**, **2**, **3**, and **4** observed in their LC phases in the course of cooling. The observations above indicate that the compounds show enantiotropic phase behavior with the existence of mesophase between the crystal and the isotropic liquid, which are consistent with the DSC results. Compared with compound **2**, compound **1** and **4** tend to form typical smectic textures for the shorter alkyl chain (compound **1**) and higher electron density (compound **4**).

Table 1. Phase transition temperatures and Δn of the compounds. Cr = crystal, Sm = smectic phase, N = nematic phase, I = isotropic

Compounds	λ^{abs} (nm)	Phase transition temperatures		Δn
		(°C)		
1	362	Cr 151 Sm 178	I	0.469
2	362	Cr 117 Sm 190	I	0.428
3	348	Cr 124 Sm 203	I	0.531
4	369	Cr 153 Sm 246	I	0.553

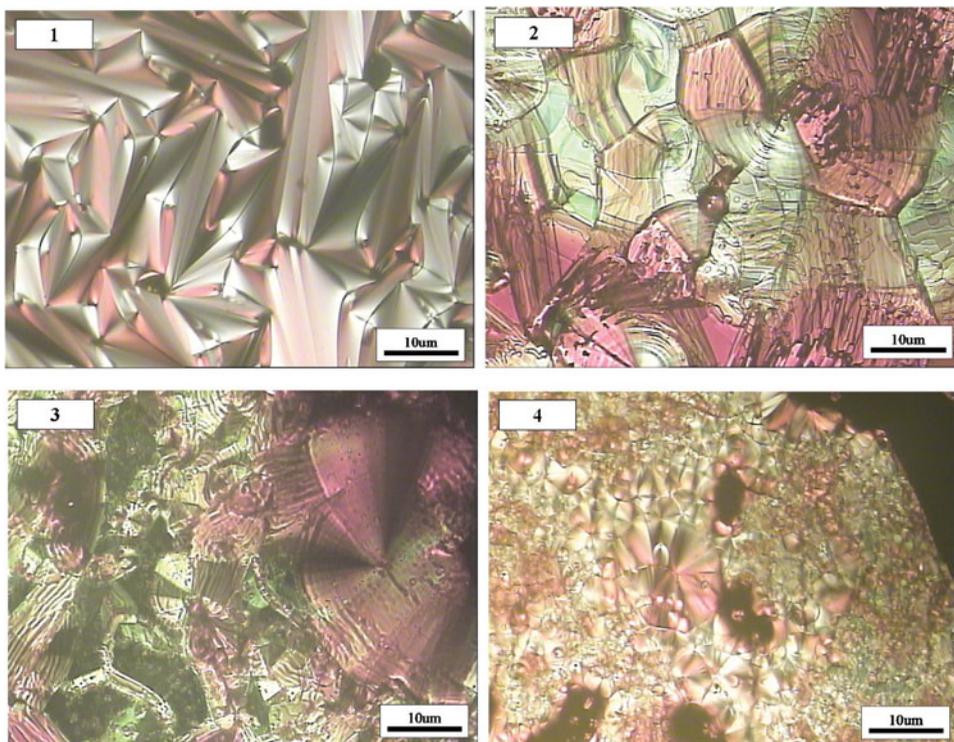


Figure 2. Polarized optical microscope images of the compounds **1–4** in mesophase, cooling rate: $10^{\circ}\text{C} \cdot \text{min}^{-1}$.

3.5. Birefringence

Having demonstrated the LC properties of the azobenzene derivatives, we focused our attention on their Δn properties. The Δn was evaluated from mixtures containing 10 wt% of each test compound in SLC 1717 using polarizing light interferometry method [19]. The Δn data of the compounds were collected in Table 1. Among compound **1** and **2**, the length of flexible chain is important evaluation factor for Δn . Longer carbon chain decreases π -electron conjugation slightly, the Δn value decreased correspondingly [24]. Then, Δn was increased by exchanging a benzene ring for the biphenyl or phenylacetylene group, the Δn of compound **3** or **4** is higher than compound **1**. However, it was well known that although the LC compounds possess lower viscosity and melting point when a phenyl group was replaced by alkynyl group, the Δn values would reduce with the decreasing of the π -electron conjugation. For all the above reasons, the highest Δn in the series was belonged to compound **4**, the values was 0.553.

3.6. Selective Reflection

In order to investigate the effect of trans-cis photoisomerization of the synthesized azobenzene LC compounds on the reflection behaviors of the N^* -LC, the synthesized compounds doped in N^* -LC were studied with the same content of 10 wt%. The composition and content of the N^* -LC were shown in Table 2.

Table 2. The composition and content of the N*-LC

Sample	Compound	Weight ratio ^a
A	—	0.0/78.0/12.0
B	1	10.0/68.0/12.0
C	2	10.0/68.0/12.0
D	3	10.0/68.0/12.0
E	4	10.0/68.0/12.0

^aWeight ratio of Compound/SLC1717/S811.

Due to the law of selective reflection, the reflection bandwidth ($\Delta\lambda$) was related to Δn only when the concentration of the chiral dopant was changeless [25].

Figure 3 showed the dependence of the spectra of the N*-LC samples on the different compounds of **1**, **2**, **3**, and **4**, respectively. In Fig. 3, $\Delta\lambda$ was about 140 nm for sample A, whereas it was 234, 222, 252, and 260 nm for **B**, **C**, **D**, and **E**, respectively. It was clearly seen that the reflection band remarkably become more broad with the increasing in Δn of the compounds of **1–4**. Whereas, when samples **B–E** exposed to UV light ($\lambda = 365$ nm, 6.5 mW cm⁻²) for 30 s at room temperature, the Δn dropped as a result the trans-cis isomerization-induced destabilization of aligned LC molecules [26, 27]. The comparison spectra of **B–E** before and after UV light were shown in Fig. 4. The $\Delta\lambda$ s of samples **B–E** would changed with the decreasing of Δn according to $\Delta\lambda = \Delta n \times P$.

4. Synthesis

4.1. General Procedure for the Synthesis of Intermediate *m*

The 4-Trifluoromethyl-aniline (16.1 g, 0.100 mol) dissolved in 200 mL of CH₂Cl₂ and Oxone (123.0 g, 0.200 mol) dissolved in 200 mL of water were added in a round bottom flask, the reaction mixture then stirred at room temperature for 15 h under an N₂ atmosphere [28, 29]. After separation of the layers, the aqueous layer was extracted with CH₂Cl₂ (3×). The combined organic layers were washed with 1 N HCl, saturated sodium bicarbonate solution, water, brine, and dried with MgSO₄. After filtration, removal of the solvent from

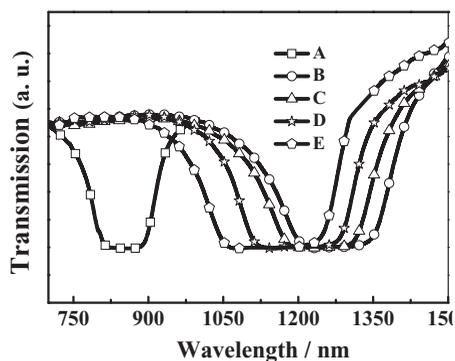


Figure 3. Dependence of the reflection band on the series compounds.

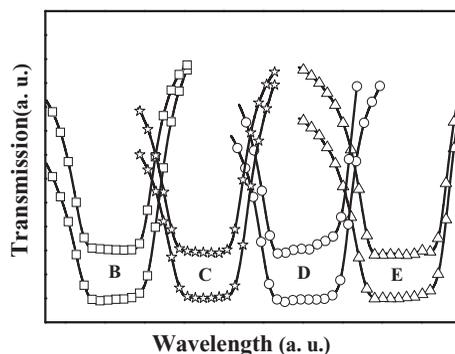


Figure 4. The comparison spectra of **B–E** before and after UV light.

the filtrate in vacuo yielded the corresponding labile nitrosoarene, which was submitted to the next condensation step without further purification. To the nitrosoarene dissolved in 200 mL of acetic acid was added 4-Iodo-aniline (21.9 g 0.100 mol). The resulting mixture was stirred at room temperature for 15 h. The precipitate was separated by filtration and the collected solid was washed with acetic acid and water and dried in a desiccator over P_2O_5 under reduced pressure for 24 h.

(4-Iodo-phenyl)-(4-trifluoromethyl-phenyl)-diazene (**m**). Yellow solid, yield: 72.3%, 27.2 g. 1H -NMR (400 MHz, $CDCl_3$) (δ , ppm): 7.66 (d, 2H, $J = 8.8$ Hz), 7.76 (d, 2H, $J = 8.4$ Hz), 7.88 (d, 2H, $J = 8.8$ Hz), 7.98 (d, 2H, $J = 8.4$ Hz). IR (cm^{-1}): 2109, 1932, 1600, 1492, 1409, 1326, 1174, 1139, 1066, 856, 810.

4.2. General Procedure for the Synthesis of the Azobenzene Liquid Crystals with High Birefringence

The **m** (3.76 g, 10.0 mmol) and the terminal alkyne compound (10.0 mmol) were dissolved in the mixed solvent of 10 mL TEA and 10 mL THF in a round bottom flask [30, 31]. The solution was purged for 30 min with bubbling A_r followed by addition of Pd (PPh_3) $_3Cl_2$ (0.211 g, 0.300 mmol), CuI (0.029 g, 0.150 mmol), and PPh_3 (0.040 g, 0.150 mmol). The reaction mixture then stirred at 30°C for 10 h under an A_r atmosphere. Upon completion, the mixture was concentrated, rediluted with CH_2Cl_2 , and filtered through a plug of silica gel. The solvent was removed in vacuo and the crude material was purified by chromatography of silica gel (2:1, hexanes: CH_2Cl_2).

[4-(4-Propyl-phenylethynyl)-phenyl]-(4-trifluoromethyl-phenyl)-diazene (**1**). Yellow solid, yield: 72.8%, 2.89 g. 1H -NMR (400 MHz, $CDCl_3$) (δ , ppm): $\delta = 0.94$ (t, 3H), 1.65 (m, 2H), 2.60 (t, 2H), 7.17 (d, 2H, $J = 7.2$ Hz), 7.46 (d, 2H, $J = 7.2$ Hz), 7.66 (d, 2H, $J = 7.6$ Hz), 7.70 (d, 2H, $J = 8.0$ Hz), 7.92 (d, 2H, $J = 7.6$ Hz), 7.99 (d, 2H, $J = 7.6$ Hz). IR (cm^{-1}): 3029, 2964, 2849, 2202, 1920, 1607, 1530, 1460, 1333, 1263, 1129, 1110, 1065, 1014, 836, 810.

[4-(4-Pentyl-phenylethynyl)-phenyl]-(4-trifluoromethyl-phenyl)-diazene (**2**). Yellow solid, yield: 74.5%, 3.13 g. 1H -NMR (400 MHz, $CDCl_3$) (δ , ppm): $\delta = 0.88$ (t, 3H), 1.31 (m, 4H), 1.60 (m, 2H), 2.66 (t, 2H), 7.17 (d, 2H, $J = 7.2$ Hz), 7.46 (d, 2H, $J = 7.2$ Hz), 7.66 (d, 2H, $J = 7.6$ Hz), 7.77 (d, 2H, $J = 7.2$ Hz), 7.92 (d, 2H, $J = 7.6$ Hz), 7.99 (d, 2H, $J = 7.6$ Hz). IR (cm^{-1}): 3030, 2965, 2860, 2212, 1920, 1594, 1511, 1467, 1320, 1161, 1129, 1066, 1015, 856, 806.

[4-(4-Pentyl-phenylbiethynyl)-phenyl]-(4-trifluoromethyl-phenyl)-diazene (**3**). Yellow solid, yield: 52.6%, 2.34 g. $^1\text{H-NMR}$ (400 MHz, CDCl_3) (δ , ppm): $\delta = 0.88$ (t, 3H), 1.31 (m, 4H), 1.61 (m, 2H), 2.61 (t, 2H), 7.17 (d, 2H, $J = 7.2$ Hz), 7.45 (d, 2H, $J = 7.2$ Hz), 7.67 (d, 2H, $J = 7.2$ Hz), 7.78 (d, 2H, $J = 7.6$ Hz), 7.92 (d, 2H, $J = 7.6$ Hz), 7.98 (d, 2H, $J = 7.6$ Hz). IR (cm^{-1}): 3026, 2960, 2861, 2212, 1919, 1594, 1598, 1467, 1320, 1269, 1097, 1021, 862, 804.

[4-(4'-Pentyl-biphenylethynyl)-phenyl]-(4-trifluoromethyl-phenyl)-diazene (**4**). Yellow solid, yield: 54.6%, 2.71 g. $^1\text{H-NMR}$ (400 MHz, CDCl_3) (δ , ppm): $\delta = 0.89$ (t, 3H), 1.34 (m, 4H), 1.64 (m, 2H), 2.63 (t, 2H), 7.20 (d, 2H, $J = 6.4$ Hz), 7.49 (d, 2H, $J = 6.4$ Hz), 7.56 (d, 2H, $J = 6.8$ Hz), 7.59 (d, 2H, $J = 6.4$ Hz), 7.69 (d, 2H, $J = 6.8$ Hz), 7.77 (d, 2H, $J = 6.4$ Hz), 7.93 (d, 2H, $J = 6.8$ Hz), 7.99 (d, 2H, $J = 6.8$ Hz). IR (cm^{-1}): 3027, 2957, 2848, 2250, 1940, 1575, 1505, 1480, 1333, 1161, 1136, 1072, 1008, 856, 820.

5. Conclusions

A series of azobenzene LCs were designed and synthesized as potential new mesogens having large birefringence values, and the effect of these replaced modifications on LC properties was obtained. The shorter alkyl chain and the π -electron conjugation strength, which were contributed to increased melting point or clearing point and to widen the mesophase, were confirmed to have higher Δn values. The study on selective reflection behavior of the compounds proved that the higher Δn had the more broad reflection band of the N^* -LC and the trans-cis isomerization produced important influence on the selective reflection behavior according to Δn change on the isomerization.

Acknowledgments

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