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Hui Chong, Cuiyun Nie, Lihong Wang, Sicong Wang, Ying Han, Yang Wang, Chengyin Wang, Chaoguo Yan



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Communication

# Construction and investigation of photo-switch property of azobenzene-bridged pillar[5]arene-based [3]rotaxanes

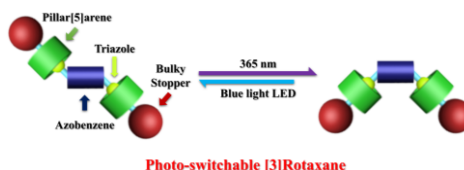
Hui Chong, Cuiyun Nie, Lihong Wang, Sicong Wang, Ying Han\* hanying@yzu.edu.cn, Yang Wang, Chengyin Wang, Chaoguo Yan\* [cgyan@yzu.edu.cn](mailto:cgyan@yzu.edu.cn)

College of Chemistry & Chemical Engineering, Yangzhou University, Yangzhou 225002, China

\* Corresponding authors.

Email addresses : (Y. Han), (C. Yan)

Graphical abstract



A series of unique azobenzene-bridged pillar[5]arene-based [3]rotaxanes with different alkyl chain length of guest were constructed by threading-endcapping method with alkylenetriazole as axil and tetrahydrochromene as endcapping group.

## ABSTRACT

Series of azobenzene-bridged pillar[5]arene-based [3]rotaxanes with different alkyl chain length of guest molecules were constructed by threading-endcapping method with alkylenetriazole as axile and tetrahydrochromene as endcapping group. The encapsulation of pillar[5]arenes were proved by high-resolution mass,  $^1\text{H}$  NMR and NOESY spectra. The photo-responsive property were examined by irradiation of the synthesized [3]rotaxanes with 365 nm and blue light LED, which caused *trans* to *cis* and *cis* to *trans* isomerization, respectively. Irradiation of corresponding model guest compounds without pillar[5]arene encapsulation resulted in near completely *trans* to *cis* and *cis* to *trans* isomerization, indicating the existence of pillar[5]arenes is the determining factor for the comprised photo isomerization efficiency

Keywords: Pillar[5]arene [3]Rotaxane Azobenzene Supramolecule Photo-switch

Stimuli-responsive supramolecular systems nowadays have gained intense research interests as they are potential building blocks for fabrication of memory storage and drug delivery systems, chemical sensors and molecular machines [1-4]. In this aspect, the mechanically interlock molecules (MIMs) are attractive entities because of their potential stimuli caused the shuttle behaviors, which enable their applications as molecular device and machine [5]. A plethora of external stimuli including temperature, pH, light and redox were commonly employed in the fabrication of such functional systems [6-12]. Among them, light is the most facile and promising external stimuli and has attracted considerable attentions [13-15]. Azobenzene was recognized as one of the key light responsive motifs among the photo active moieties because of light triggered *trans/cis* isomerization, which could render substantial structure and property change of azobenzene [15]. Therefore, azobenzene derivates have been widely employed as guest of light-responsive MIMs [16].

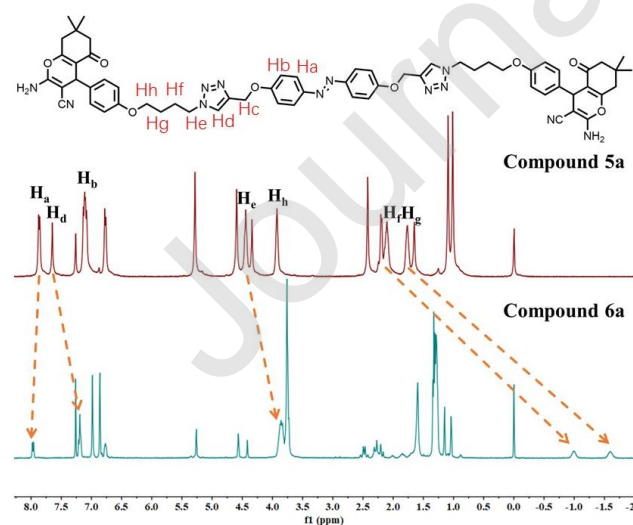
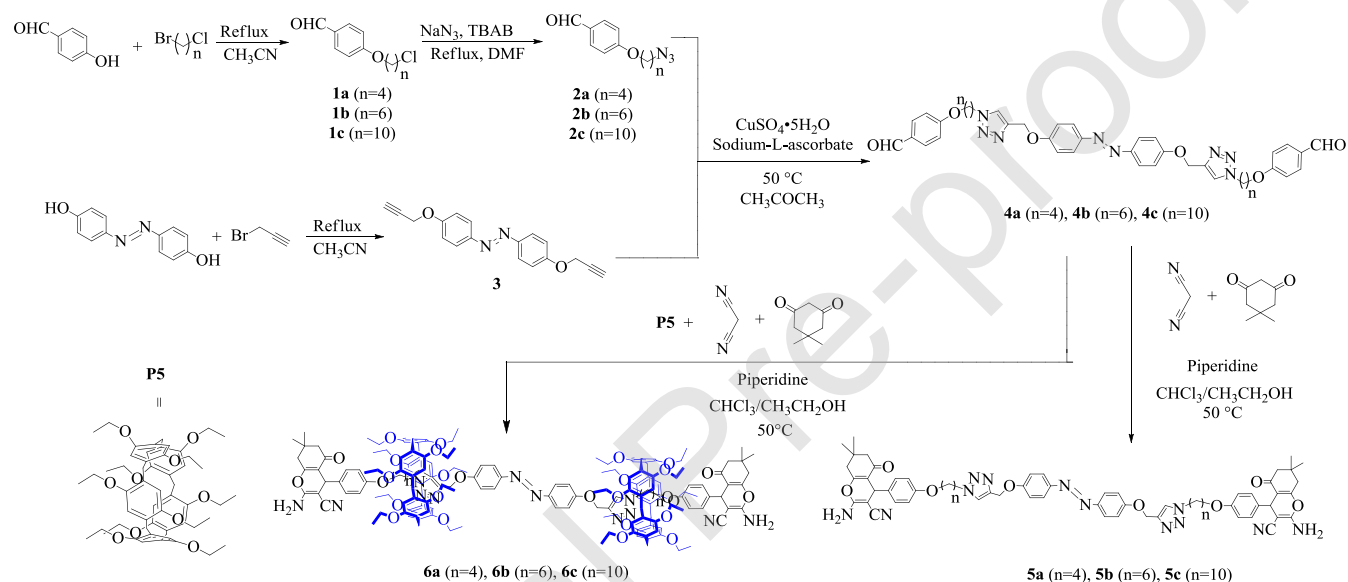
Traditional host molecules for fabricating rotaxanes included crown ethers, cyclodextrins, and cucurbit[*n*]urils [17]. The synthesis and photo-responsive property of cucurbit[7]uril and azobenzene based [3]rotaxane has been reported. The systems exhibited molecular shuttle property upon irradiation of UV/vis light [18]. The novel macrocyclic structure of pillar[*n*]arenes ( $n = 5, 6$ ), first reported in 2008 and described as “fascinating cyclophanes with a bright future”, have been proved to be the promising candidate host for rotaxane and catananes [15]. Recently, some rotaxanes composed of azobenzene derivates and pillar[*n*]arenes have been reported in literatures [19-21]. The photo-switch properties together with the function exploration of pillar[*n*]arenes-based azobenzene-bearing rotaxanes received considerable attentions [15]. Yet, current related investigations mainly focused on pillar[*n*]arene ( $n = 5, 6$ ) based azobenzene bridged [1] and [2]rotaxanes. The light-responsive property of bis-pillar[*n*]arene and azobenzene based [3]rotaxanes remain much unexplored. Herein, we report the design, syntheses and photo-switch property of pillar[5]arene-based azobenzene-bridged [3]rotaxanes.

We used a straight-forward strategy to synthesize the pillar[5]arene-based azobenzene-bridged [3]rotaxanes (Scheme 1). The alkylation of 4,4'-dihoxylazobenzene with 3-bromoprop-1-yne readily afforded the triple bond bearing photo-active segments. The triple bonds attached to the functional core facilitate subsequently constructing of precursors of guest molecules by Sharpless

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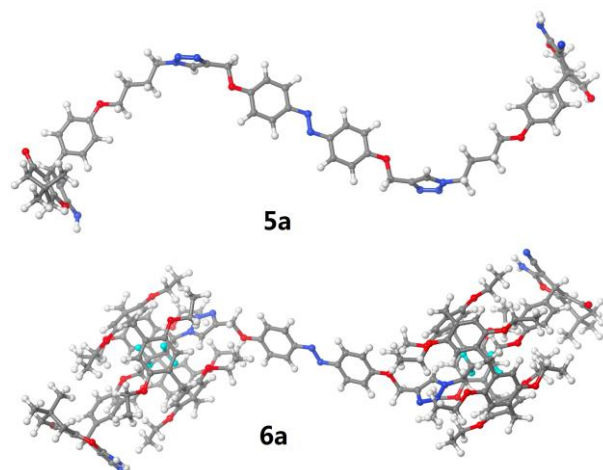
copper(I)-catalyzed Sharpless alkyne-azide click reaction (Scheme 1). The triazole groups formed in this step could help “fix” the pillar[5]arenes by multiple hydrogen bonds interactions. With the consideration that alkyl chain length on the guest molecule may have different influence on the photo-assisted shuttle property of the supramolecules, we synthesized three azide-end capped segments with varying alkyl chain lengths (C4, C6 and C10). The symmetrical azobenzene bearing threads (**5a-5c**) was also synthesized as standard. The alkyl chain length did not show obvious influence on the yield of the click reaction. The threading of pillar[5]arene onto the guest molecule was achieved in chloroform solution. And the benzaldehyde end capped pseudo rotaxanes was immediately reacted with 5,5-dimethylcyclohexane-1,3-dione and malononitrile to install the bulky stopper to block the encapsulated pillar[5]arenes. In contrast to the situation of **6a** and **6b**, the yield for **6c** was obviously reduced (24% for **6a**, 26% for **6b** and 8% for **6c**), may suggest a relatively low threading efficiency of pillar[5]arene in the case of molecular thread of C10 chain.

In addition to these [3]rotaxanes, we also synthesized bulky stopper installed guest molecules (**5a-5c**) without threading of pillar[5]arenes in order to examine the influence of alkyl chain length on photo-responsive property in advance.  $^1\text{H}$ ,  $^{13}\text{C}$  NMR and high-resolution mass spectra data for these compounds were shown in supporting compound **6a** exhibited obvious upfield  $\delta$  movements in information. As shown in Fig. 1, the protons  $\text{H}_d$ - $\text{H}_g$  of contrast to **5a**. The upfield  $\delta$  value of  $\text{H}_d$  ( $\Delta\delta = -0.447$  for  $\text{H}_d$ ) suggested the formation of  $\text{C-H}\cdots\text{O}$  hydrogen bonds between triazole segment and the oxygen atoms on the pillar[5]arene as documented in the literature [22]. The upfield  $\delta$  movements of protons  $\text{H}_e$  to  $\text{H}_h$  suggested inclusion of the methylene units within the cavity of pillar[5]arene as the result of the encapsulation-induced shielding effects [23]. These results clearly indicated formation of unique [3]rotaxanes. In addition to the  $^1\text{H}$  NMR spectra, the exact molecular weight of **6a** measured by high-resolution mass matched the calculated value, further indicating the formation of [3]rotaxanes (Figs. S22, S25 and S28 in Supporting information).



It should be noticed that the small cavity size of pillar[5]arene (inner diameter of around 4.7 Å) typically does not allow it to encapsulate the azobenzene segment (the diameter of benzene ring is around 5.0 Å). Therefore, it is highly possible that the

pillar[5]arene would be squeezed to locate on the alkyl chain segment of guest molecules. After the threading of pillar[5]arenes,  $\delta$  values of  $H_f$  and  $H_g$  on the alkyl chain **6a** displayed dramatically upfield in movements among all the alkyl chain protons ( $\Delta\delta = -3.370$  and  $-3.180$  for  $H_f$  and  $H_g$ ;  $\Delta\delta = -0.065$  and  $-0.582$  for  $H_e$  and  $H_h$ ). Such alternation in NMR indicated strong C-H $\cdots\pi$  interactions between the pillar[5]arenes [24]. On the other hand, the splitted aromatic protons on pillar[5]arene ( $\delta = 6.99$  and  $6.86$  for aromatic protons in **6a**) suggested different chemical environments of the two rims of pillar[5]arenes upon encapsulation. Based on these  $^1H$  NMR results, it was reasonable to predict the pillar[5]arenes located near the triazole segments of the guest molecule with the cavity covering  $H_{e-g}$  of the alkyl chain. This configuration was further supported by the DFT optimized structures of the compound **5a** and **6a**, based on which the computed upfielded  $\delta$  for  $H_f$  and  $H_g$  is  $\sim 3$  ppm, in good agreement with the experimental observation mentioned above (Fig. 2).



**Fig. 2.** Optimized structures of **5a** and **6a** in dichloromethane solution (Hydrogens  $H_f$  and  $H_g$  are highlighted in cyan).

The  $\delta$  values of alkyl chain protons of the guest molecules of **6b** and **6c** exhibited similar upshift pattern in contrast to the corresponding model guest compounds. This also indicated the formation of corresponding [3]rotaxanes for the compounds **6b** and **6c**, and was also supported by high-resolution mass results (Supporting information). Additionally, in NOESY spectra of **6b**, the clear NOEs signals have been observed between aromatic ( $H_h$ ), methylene bridge ( $H_g$ ) and ethoxyl ( $H_g$  and  $H_f$ ) and the guest alkyl chain protons ( $H_a$  to  $H_d$ , Fig. 3). This further proved host pillar[5]arenes were encapsulated on the guest molecular thread and the location of the host pillar[5]arene was on the alkyl chain of the guest thread.  $^1H$  NMR results revealed more complicated situations of **6b** and **6c**. Couples of new proton resonance signals were observed in these spectra due to the incorporation of longer alkyl chains in the precursors. As judged from the resonance spectra, the shielding effect of pillar[5]arene was influenced by the alkyl chain length. For instance, in the case of compound **6b**, the highest field proton signal of the alkyl chain appeared around  $-2.02$  ppm. However, in compound **6c**, the corresponding appeared around  $-1.51$  ppm, whereas, the corresponding protons in **5b** and **5c** appeared both around  $1.90$  ppm. In addition, aromatic protons of pillar[5]arene in **6b** and **6c** exhibited slightly different  $\delta$  values (corresponding  $\delta$  for **6b** were  $6.94$  and  $6.90$ ;  $\delta$  for **6c** were  $6.92$  and  $6.88$ ). This suggested slightly different shielding effect of pillar[5]arene in synthesized [3]rotaxanes. In compound **6a**, proton  $H_d$  was found to be merged with  $H_b$  in resonance spectrum, in the case of **6b** and **6c**, these two protons clearly split into two separated peaks ( $\delta$  for  $H_d$  in **6b** was  $7.63$ ;  $\delta$  for  $H_d$  in **6c** was  $7.40$ ). This suggested varied C-H $\cdots$ O hydrogen bond situations between  $H_d$  and oxygen atom in pillar[5]arene in **6b** and **6c** in contrast to **6a**. The detailed  $^1H$  NMR spectra for **6b**, **6c** and their corresponding **5b** and **5c** were shown in Figs. S2 and S3 (Supporting information). We attempted to obtain the single crystal of the three compounds to conduct an in-depth analysis of the configurations, yet we failed to obtain the desired single crystals.

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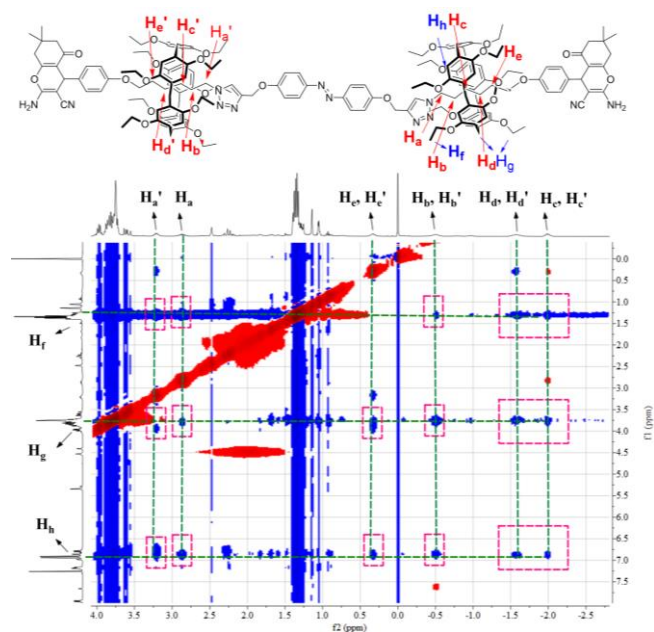


Fig. 3. NOESY spectrum of **6b** in  $\text{CDCl}_3$ .

In order to examine the photo-switch properties of [3]rotaxanes **6a**, **6b** and **6c**, we conducted the light irradiation experiments. As shown in Figure 4, compound **6a** exhibited partially photo-switch property as characterized by UV-vis. Upon irradiation with UV light of 365 nm with the light intensity of  $2.88 \text{ mW/cm}^2$  for 30 min, the absorbance of **6a** centered at 350 nm, which corresponds to the  $\pi$ - $\pi^*$  electronic transition of *cis*-azobenzene isomer, reduced dramatically. This suggested the occurrence of *trans* to *cis* under UV irradiation of **6a**. Subsequently, irradiating **6a** with blue light LED resulted in the 350 nm absorption recovery, meaning occurrence of reverse isomerization [25]. But the absorbance did not completely restore, meaning comprised *cis* to *trans* photo-isomerization efficiency. Compounds **6b** and **6c** displayed similar incomplete reversible transformation upon light irradiation according to the UV-vis spectra (Figs. S3 and S4).

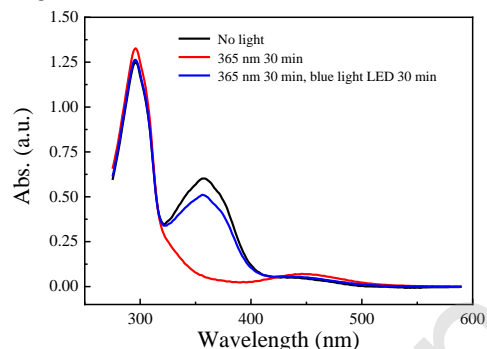
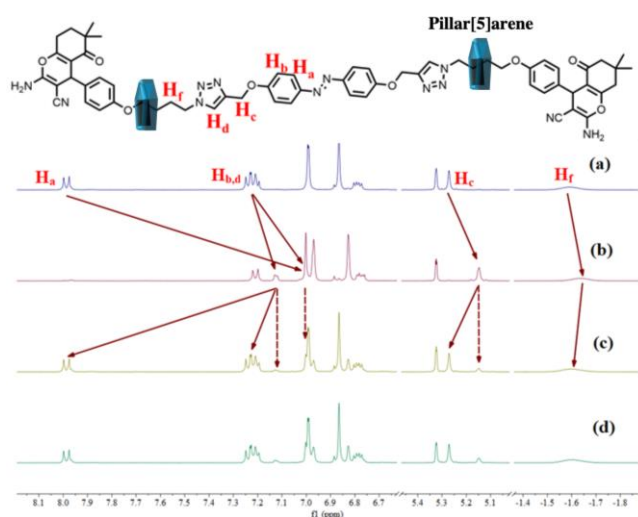


Fig. 4. UV-visible absorption spectra of **6a** (black line); irradiated with 365 nm light (red line) and subsequently irradiated with blue light LED (blue line). Irradiation time was 30 min.

Irradiating **6a** with blue light LED for 30 min resulted in partially regain of the chemical shifts for these protons, indicating the location regain of the pillar[5]arenes (Fig. 5). The coexistence of both representative chemical shifts for these isomers proved the incomplete *cis/trans* isomerization. This is consistent with the result of UV-vis measurement. The detailed *cis/trans* ratio was determined by comparing the integration of  $\text{H}_c$  and  $\text{H}_c'$  to be approximately 77/23. Extended time irradiating experiment (150 min of blue light LED) afforded the identical  $^1\text{H}$  NMR results as that of 30 min irradiation, indicating the *cis* to *trans* photo-isomerization for compound **6a** has reached chemical equilibrium regardless of irradiation time and dose. The further irradiation experiment using the end capped guest model molecule **5a** displayed nearly completely photo-isomerization in both *trans-cis* and *cis-trans* directions as judged by  $^1\text{H}$  NMR spectra (Fig. S5 in Supporting information). This suggested the installation of pillar[5]arene caused the comprised *trans* to *cis* photo-isomerization efficiency.



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**Fig. 5.** Partial  $^1\text{H}$  NMR spectra of compound **6a** upon irradiated with 365 for 30 min and blue light LED, a: no irradiation, b: 365 nm for 30 min, c: blue light LED for 30 min, d: blue light LED for 150 min. ( $\text{CD}_2\text{Cl}_2$ , 400 MHz).

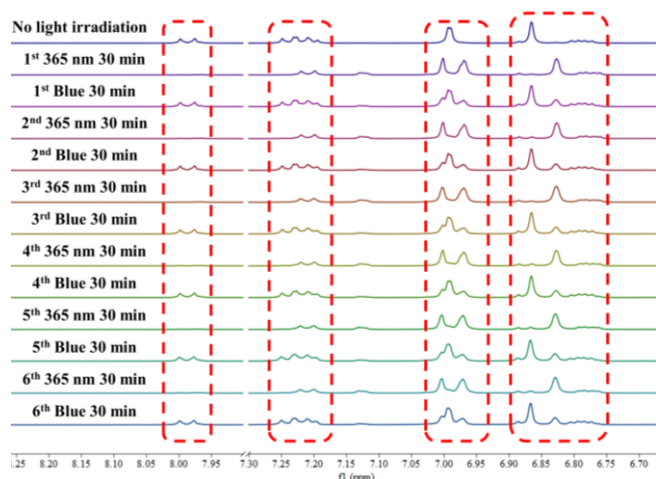
Irradiation with 365 nm light also resulted in nearly complete *trans/cis* conversion of compounds **6b** and **6c**, as proved by disappearance of corresponding  $\text{H}_a$  from its original position in  $^1\text{H}$  resonance spectra. In the backwards photo-isomerization of **6b** using blue light LED,  $\text{H}_d$  splitted into two peaks in resonance spectra with  $\delta$  of 7.63 and 7.58, respectively. The peak of  $\delta$  7.63 matched the corresponding peak in **6b** without light irradiation. This may suggest a different  $\text{C-H}\cdots\text{O}$  hydrogen bonding situation between  $\text{H}_d$  and oxygen atom on pillar[5]arenes in *cis* and *trans* isomers of **6b**. Also, the *cis/trans* ratio was determined to be 71/29 by comparing the intergration of the split  $\text{H}_d$ . Corresponding  $\text{H}_d$  in compound **6c** ( $\delta$  of 7.40 without light irradiation) shifted upfield to 7.32 ppm after UV light irradiation, and partially regained the original chemical shift in  $^1\text{H}$  NMR resonance spectra. The corresponding *cis/trans* ratio was calculated to be 71/29 as well. The similar *cis/trans* ratio of synthesized [3]rotaxanes indicated the longer alkyl chain of the guest molecular thread did not result in improved photo-isomerization efficiency. The results of irradiation experiment using corresponding **5b** and **5c** resembles the case of **5a**, further proved the threading of pillar[5]arene could be the main reason for incompletely *trans* to *cis* photo-isomerization (Figs. S7 and S8 in Supporting information).

We subsequently investigated the light-triggered shuttle behaviors of **6b** and **6c** using  $^1\text{H}$  NMR. The shielding effects of pillar[5]arene were observed to increase with alkyl chain length (the most pronounced upfield  $\delta$  variations of the alkyl chain protons of **6b** and **6c** amounted to -0.07 and -0.09, respectively). The enhanced  $\Delta\delta$  of alkyl chain of **6b** and **6c** in contrast to **6a** in the process of photo irradiation suggested longer alkyl chain could facilitate the shuttle behavior of pillar[5]arene to a certain extent. However, the photo-triggered shuttle behavior of [3]rotaxanes was believed to be confined to a small extent independent of the alkyl chain lengths. One plausible reason could be the strong “hydrogen bond locking effect” between the triazole and pillar[5]arenes that restricted the shifting of pillar[5]arenes even in the sterically crowded *trans* form [3]rotaxanes. The partial  $^1\text{H}$  NMR spectra of compounds **6b** and **6c** after light irradiation were shown in Figs. S9 and S10 (Supporting information).

Repeated photo-isomerization of compound **6a** can be realized by alternatively irradiation with 365 nm light and blue light LED, respectively. As shown in Fig. 6, the protons in aromatic region of **6a** displayed reproducible chemical shift changes within five irradiation cycles. Compounds **6b** and **6c** also showed repeated photo-isomerization properties as characterized by alternatively irradiation experiments. The partial  $^1\text{H}$  NMR spectra of **6b** and **6c** were shown in Figs. S11 and S12 (Supporting information).

To best of our knowledge, we have for the first time constructed series of azobenzene bridged pillar[5]arene-based [3]rotaxanes with different alkyl chain guest molecule (**6a**, **6b** and **6c**).  $^1\text{H}$  NMR and NOESY and high-resolution mass spectra proved the inclusion of the pillar[5]arenes onto the guest molecule, the main driving force for formation of the [3]rotaxanes include  $\text{C-H}\cdots\text{O}$  hydrogen bond and  $\text{C-H}\cdots\pi$  interactions. As characterized by UV-vis and  $^1\text{H}$  NMR spectroscopy, the synthesized rotaxanes could undergo completely *trans* to *cis* isomerization under irradiation with 365 nm light for 30 min. Illumination of rotaxanes by blue light did not yield completely backward *cis* to *trans* isomerization even in extended irradiation time and intensity. The installation of pillar[5]arene was believed to be the reason for incomplete photo-isomerization. The slightly photo-triggered shuttle behavior of [3]rotaxanes have been observed in the  $^1\text{H}$  NMR spectra. We are currently focusing on the developing photo responsive [3]rotaxanes with improved shuttle behavior.

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**Fig. 6.** Partial  $^1\text{H}$  NMR spectra of compound **6a** upon irradiated with 365 nm and blue light LED for 30 min for 6 cycles ( $\text{CD}_2\text{Cl}_2$ , 400 MHz). Protons in the red squares referred to the aromatic protons.

## Acknowledgments

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