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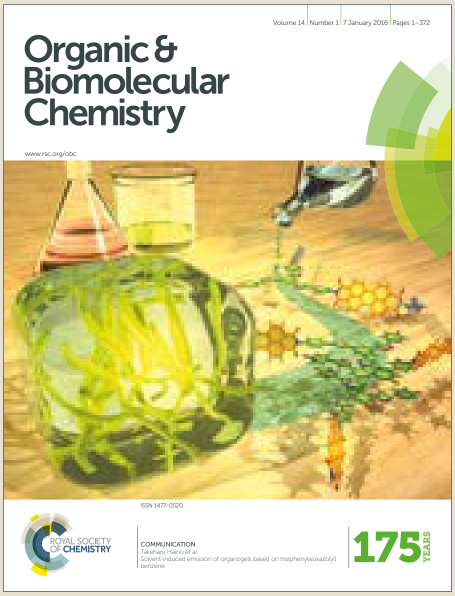

Volume 14 | Number 1 | 7 January 2016 | Pages 1–372

Organic &
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# A dimethoxypillar[5]arene/azastilbene host–guest recognition motif and its application in the fabrication of polypseudorotaxane

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***Received (in XXX, XXX) Xth XXXXXXXXX 20XX, Accepted Xth XXXXXXXXX 20XX***

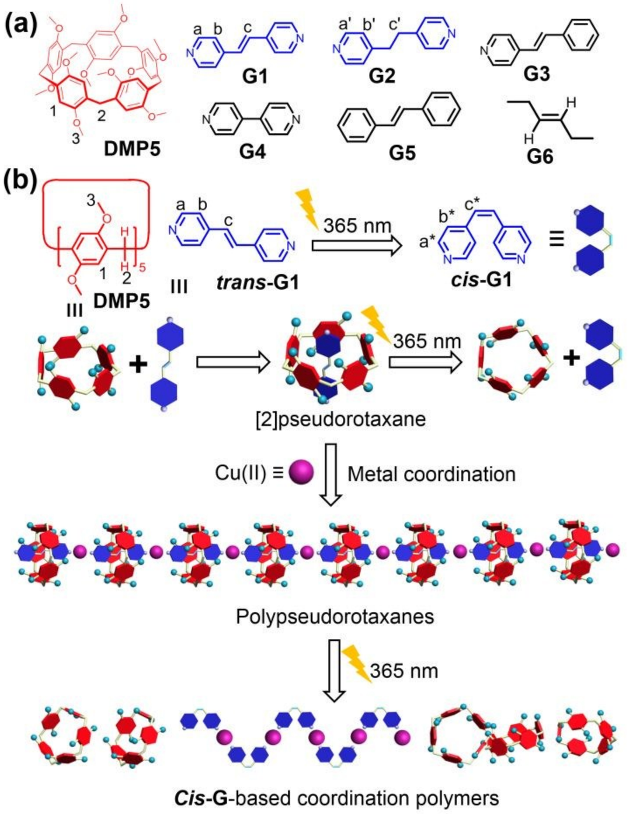
**DOI: 10.1039/c0xx00000x**

Pillar[*n*]arenes, known as the fifth generation of host macrocycles since 2008, have become a popular topic over the past ten years. Until now, the studies of pillar[*n*]arenes have been mainly focused on pillar[5]arenes owing to their easy synthesis and high yields. Especially, 1,4-dimethoxypillar[5]arene (**DMP5**), which shows simple structure, efficient synthesis and high yield, has played important roles in the construction of various advanced supramolecular architechtures. However, **DMP5** has only displayed host−guest binding property towards some guests. Therefore, the investigation of the host−guest chemistry of **DMP5** should be able to greatly promote the development of pillararene chemistry. Herein, a photo-sensitive azastilbene derivative was chosen as a neutral guest to study the host−guest binding and stimuli-responsive behavior with **DMP5**. In addition, the binding behavior of **DMP5** towards a series of analogous neutral guest molecules were investigated to study the driving forces of the host−guest interaction between **DMP5** and the azastilbene guest. Moreover, the [2]pseudorotaxane based on **DMP5** and the azastilbene guest was used to construct a polypseudorotaxane *via* metal coordination.

## 1. Introduction

Supramolecular chemistry, which plays a major role in the development of chemistry, has experienced remarkable progress over the last five decades.[1-4] Macrocycles are the basic building blocks to construct various supramolecular architectures and materials due to their special excellent host–guest properties.[5-11] Therefore, the exploration of new macrocycles and their host−guest recognition is an important and essential part in supramolecular chemistry.[12-14] Pillar[*n*]arenes,[15,16] known as the fifth generation of host macrocycles, have become a popular topic over the past ten years.[17] Due to their distinct rigid and pillar structures, electron-donating cavities, and easy functionalization, pillararenes have displayed excellent host–guest recognition properties with diversiform guest molecules, setting a stage for the fabrication of various interesting supramolecular systems in the application of many fields.[18-35]

Until now, the studies of pillar[*n*]arenes have been mainly focused on pillar[5]arenes owing to their easy synthesis and high yields. The host–guest chemistry of pillar[5]arenes has been widely explored. However, among diverse pillar[5]arene derivatives, 1,4-dimethoxypillar[5]arene (**DMP5**), which shows the highest yield until now, has only displayed host–guest binding property towards some guests. For instance, Stoddart *et al*. reported the host–guest interaction between **DMP5** and 1,8-diaminooctane;[36] Huang *et al*. reported the host–guest interaction bewteen **DMP5** and *n*-octylethyl ammonium hexafluorophosphate;[37] Li *et al*. reported some neutral guests that can complex with **DMP5**, which are 1,4-dibromobutane,[38] 1,4-dicyanobutane,[39] 1,4-[1,2,4]triazol-1-yl-butane[40] and [1,2,3]triazol-1-yl-4-cyanno-butane;[12] Ogoshi *et al*. reported the complexation between **DMP5** and *N*-alkyl-pyridine bromide.[41] These **DMP5**-based host–guest systems played important roles in the construction of various advanced supramolecular architechtures, such as threaded structures, supramolecular polymers and stimuli-responsive systems.[12,29,41-45] It can be seen that neutral **DMP5** can complex with cationic and neutral guests due to its electron-rich cavity. Between them, neutral guests are more advantageous because of their relatively higher solubility in nonpolar solvents than cationic guests which is the same as **DMP5**. Therefore, the study of **DMP5**-based host−guest reconition property with neutral guests will facilitate its further application in the construction of advanced supramolecular systems. Furthermore, introducing stimuli-responsiveness to host–guest systems are of great importance for the construction of functional supramolecular structures, such as molecular machine, stimuli-responsive self-assemblies and adaptive materials.[46-48] Herein, a photo-sensitive azastilbene derivative, *trans*-4,4′-vinylenedipyridine (***trans*-G1**), was chosen as a neutral guest to study the host–guest binding behavior and stimuli-responsive complexation with **DMP5**. In addition, the binding behavior of **DMP5** towards a series of analogous neutral guest molecules, 4,4′-ethylenedipyridine (**G2**), *trans*-4-styrylpyridine (**G3**), 4,4′-bipyridine (**G4**), *trans*-stilbene (**G5**) and *trans*-3-hexene (**G6**), were investigated to study the driving forces of the host–guest interaction between **DMP5** and ***trans*-G1**. Moreover, the [2]pseudorotaxane formed by **DMP5** and ***trans*-G1** was used to construct a polypseudorotaxane *via* metal coordination (Scheme 1).



**Scheme 1.** (a) Chemical structures of compounds **DMP5**, ***trans*-G1**, **G2**, **G3**, **G4**, **G5** and **G6**; (b) cartoon representation of the photo-responsive host−guest recognition between **DMP5** and ***trans*-G1** and the Cu(II) linked polypseudorotaxane.

## 2. Experimental section

All reagents were commercially available and used as supplied without further purification. Solvents were either employed as purchased or dried according to procedures described in the literature. **DMP5** was prepared according to published procedures.[15] Compounds ***trans*-G1**, **G2**, **G3**, **G4**, **G5**, **G6** and Cu(OAc)$_2$ were commercially available. NMR spectra were recorded with a Bruker Avance DMX 600 spectrophotometer. FT-IR spectra were recorded with a Thermo Scientific Nicolet iS50 instrument. XRD were performed with a Bruker D2 PHASER. Scanning electron microscopy investigations were carried out on a TASCAN (LYRA3) instrument. Atomic force microscopy experiments were performed by a Bruker Multi-Mode 8.0 instrument.

## 3. Results and discussion

First, $^1$H NMR spectroscopy was used to study the host–guest complexation behaviour between **DMP5** and these neutral guests, ***trans*-G1**, **G2**, **G3**, **G4**, **G5** and **G6**. As shown in Fig. 1, the host–guest interaction between **DMP5** and ***trans*-G1** was clearly observed. The peaks related to the protons H$_a$, H$_b$ and H$_c$ on ***trans*-G1** shifted upfield and peaks related to H$_b$ and H$_c$ became broad of the equimolar mixture of **DMP5** and ***trans*-G1** compared to ***trans*-G1** alone. The remarkable changes of the signals for ***trans*-G1** resulted from the formation of a threaded structure **DMP5⊃*trans*-G1**, and shielded by the electron-rich cyclic structure of **DMP5**. Moreover, the broadening effect of the signals was due to complexation dynamics.[39] In addition, a 2D NOESY NMR experiment was employed to study the relative positions of the components in **DMP5⊃*trans*-G1** (Fig. S1). NOE correlation signals were observed between proton H$_a$ of ***trans*-G1** and protons H$_3$ of **DMP5** (Fig. S1, A), between proton H$_b$ of ***trans*-G1** and protons H$_1$ and H$_3$ of **DMP5** (Fig. S1, B and C), and between proton H$_c$ of ***trans*-G1** and proton H$_3$ of **DMP5** (Fig. S1, D). These results provided convincing proof for the formation of a [2]pseudorotaxane between **DMP5** and ***trans*-G1**. Moreover, the stoichiometry and association constant between **DMP5** and ***trans*-G1** were investigated by $^1$H NMR titration experiments. The titration experiments were carried out with solutions which had a constant concentration of ***trans*-G1** (1.00 mM) and different concentrations of **DMP5** (Fig. S2). By a mole ratio plot and a non-linear curve-fitting method, the stoichiometry and association constant between **DMP5** and ***trans*-G1** were calculated to be 1:1 (Fig. S3) and $(4.45 \pm 0.23) \times 10^2$ M$^{-1}$ (Fig. S4), respectively.

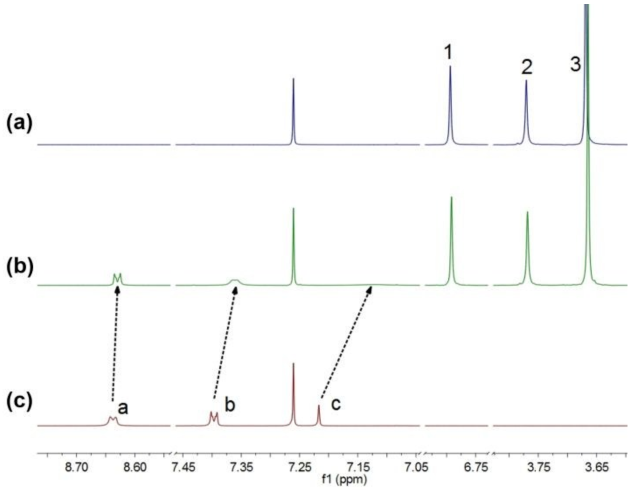


**Fig. 1** Partial $^1$H NMR spectra (600 MHz, CDCl$_3$, room temperature): (a) **DMP5** (5.00 mM); (b) ***trans*-G1** (5.00 mM) and **DMP5** (5.00 mM); (c) ***trans*-G1** (5.00 mM).

Similarly, the host–guest complexation behaviour between **DMP5** and **G2** was also studied by $^1$H NMR spectroscopy. As shown in Fig. S5, significant chemical shift changes of the signals for the protons on **G2** appeared upon the addition of **DMP5**. The peaks related to H$_{a'}$, H$_{b'}$ and H$_{c'}$ shifted upfield and became broad. Meanwhile, NOE correlation signals were observed between proton H$_{a'}$ of **G2** and protons H$_1$, H$_2$ and H$_3$ of **DMP5** (Fig. S6, A,

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B and C). These results suggested the host−guest complexation between **DMP5** and **G2**. In addition, $^1$H NMR titration experiments were carried out to determine stoichiometry and association constant between **DMP5** and **G2** (Fig. S7), which were calculated to be 1:1 (Fig. S8) and $(4.55 \pm 0.31) \times 10^2$ M$^{-1}$ (Fig. S9), respectively. Next, the host–guest complexation properties of **DMP5** towards **G3**, **G4**, **G5** and **G6** were studied. From the $^1$H NMR spectroscopy data, it can be seen that there were no chemical shift changes or broadening phenomena of the peaks related to the protons on these neutral guests or **DMP5** of their equimolar mixtures compared to **DMP5** and these guests alone, indicating that there were no host–guest interactions between **DMP5** and these guest molecules (Fig. S10, S11, S12 and S13).

From the host−guest complexation behaviour of **DMP5** to ***trans*-G1**, **G2**, **G3**, **G4**, **G5** and **G6**, it can be seen that only ***trans*-G1** and **G2** which possess two pyridyl groups which are not directly linked can complex with **DMP5**. That is to say electron-deficient pyridyl group and the linker between the pyridyl groups are the key parts in the formation of the [2]pseudorotaxanes, **DMP5$\supset$*trans*-G1** and **DMP5$\supset$G2**.[49] The reason may be that the C–H⋯N or C–H⋯O hydrogen-bonding interactions and C–H⋯π interactions play important roles in the threaded structure since the pyridyl groups of ***trans*-G1** and **G2** located at the rim of the cavity of **DMP5**. Therefore, multiple weak C–H⋯N interactions between the methyl groups on **DMP5** and the nitrogen atoms of the pyridyl groups on ***trans*-G1** and **G2** are the dominant driving forces for complexation.[40]

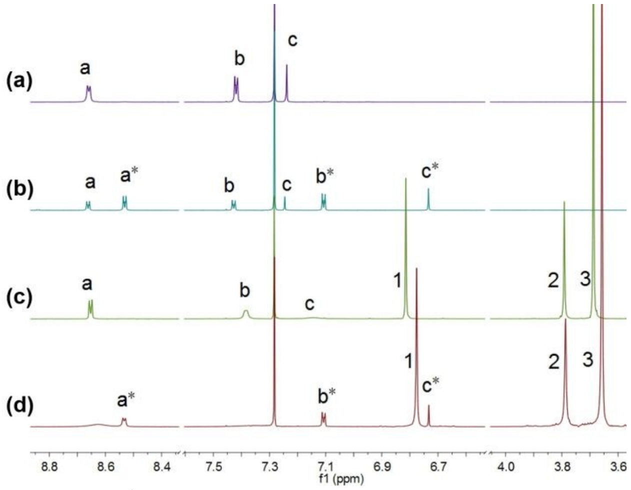

**Fig. 2** Partial $^1$H NMR spectra (600 MHz, CDCl$_3$, room temperature): (a) ***trans*-G1** (5.00 mM); (b) ***trans*-G1** (5.00 mM) after irradiation with UV light at 365 nm; (c) **DMP5$\supset$*trans*-G1** (5.00 mM); (e) **DMP5** + ***trans*-G1** (5.00 mM) after irradiation with UV light at 365 nm.

Next, the photo-responsive property of the host−guest complexation between **DMP5** and ***trans*-G1** which introduced by photo-sensitive property of ***trans*-G1** was investigated *via* $^1$H NMR spectroscopy. As shown in Fig. 2a and 2b, the peaks related to the protons H$_a$, H$_b$ and H$_c$ of ***trans*-G1** weakened accompanied by the enhancement of the peaks related to the protons H$_{a*}$, H$_{b*}$ and H$_{c*}$ of ***cis*-G1** upon irradiation by UV light at 365 nm, indicating that ***trans*-G1** photoisomerized from the *trans* state to the *cis* state and the *trans–cis* conversion was 65% at a photostationary state. In addition, after irradiating a solution of **DMP5$\supset$*trans*-G1** with UV light at 365 nm, the peaks related to H$_a$, H$_b$ and H$_c$ on ***trans*-G1** weakened and the signals for protons H$_{a*}$, H$_{b*}$ and H$_{c*}$ on free ***cis*-G1** were clearly observed (Fig. 2c and 2d), suggesting that the host–guest interactions between **DMP5** and ***trans*-G1** were destroyed and there was no complexation between **DMP5** and ***cis*-G1**. The reason is that the size of ***cis*-G1** is larger than the cavity of **DMP5**.[50]

In addition, the [2]pseudorotaxane formed by **DMP5** and ***trans*-G1** was used to fabricate a polypseudorotaxane linked by Cu(OAc)$_2$ (Cu(II)) coordination. As a comparison, the coordination between Cu(II) and ***trans*-G1** was investigated. As shown in Fig. 3a and 3b, upon addition of Cu(II) to the solution of ***trans*-G1**, the peaks related to H$_a$ and H$_b$ shifted downfield and all of the peaks became broad, indicating the formation of coordination polymer based on ***trans*-G1**. Meanwhile, upon addition of Cu(II) to the solution of **DMP5** + ***trans*-G1**, the peaks related to complexed H$_a$ and H$_b$ shifted downfield and all of the peaks became broad (Fig. 3c and 3d), suggesting the formation of the metallosupramolecular polypseudorotaxane based on **DMP5$\supset$*trans*-G1**. The result was then confirmed by 2D DOSY NMR spectroscopy experiments. As shown in Fig. 4 and Fig. S14, compared with the equimolar mixture of **DMP5** + ***trans*-G1** solution, the diffusion coefficient (*D*) decreased from $2.24 \times 10^{-9}$ to $1.00 \times 10^{-9}$ m$^2$s$^{-1}$ upon addition of Cu(II), indicating the formation of the polypseudorotaxane.

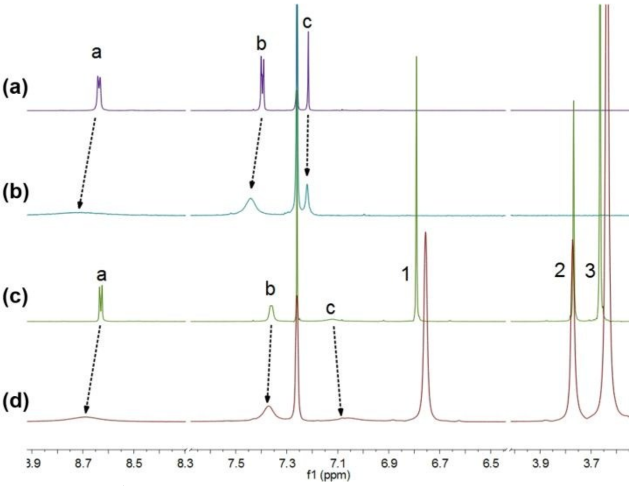

**Fig. 3** Partial $^1$H NMR spectra (600 MHz, CDCl$_3$, room temperature): (a) ***trans*-G1** (5.00 mM); (b) after addition of equimolar Cu(II) to a; (c) **DMP5** + ***trans*-G1** (5.00 mM); (d) after addition of equimolar Cu(II) to c.

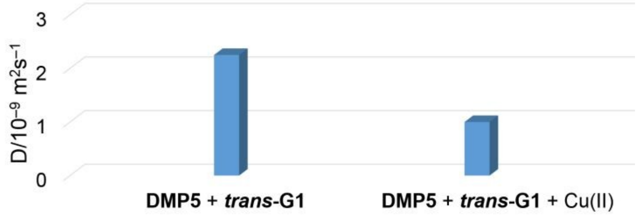

**Fig. 4** Diffusion coefficient *D* (600 MHz, CDCl$_3$, room temperature) of **DMP5** + ***trans*-G1** (5.00 mM) and after addition of equimolar Cu(II).

FT-IR spectroscopy was performed to investigate the solid structure of the polypseudorotaxane. As shown in Fig. S15, the main peaks related to **DMP5** + ***trans*-G1**, the polypseudorotaxane and Cu(II) were marked and assigned. The polypseudorotaxane showed strong absorbance bands including =C−H, C–H, C=C and C=N of the stretching vibrations at 2949, 2827, 1599 and 1598 $cm^{-1}$ and –$-CH_2-$ or $-CH_3$, C−O−C and =C−H of the bending vibration at 1448−1384, 1206 and 1041 $cm^{-1}$, respectively. In addition, these adsorption bands became broad compared to the mixture of **DMP5** and ***trans*-G1**. The powder X-ray diffraction spectrum (XRD) experiment was also carried out to investigate the solid state of the polypseudorotaxanes. As shown in Fig. S16, broad peaks appeared on the spectrum of the polypseudorotaxane, indicating that it was an amorphous material.[51] Scanning electron microscopy (SEM) were further utilized to investigate the micromorphology of the polypseudorotaxane. As shown in Fig. 5a, a rod-like fiber with the diameter about 5 μm were observed obtained from a high concentration solution of the polypseudorotaxane. In addition, as shown in Fig. 5b, the solid state of the polypseudorotaxane showed connected structures. Atomic Force microscopy (AFM) data confirmed the results (Fig. S17). These results suggested the formation of the polypseudorotaxane.

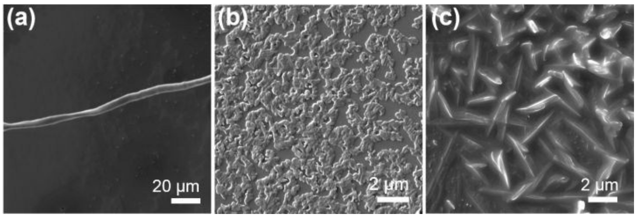

**Fig. 5** SEM images: (a) a fiber drawn from a high concentration solution of mixtures of **DMP5⊃*trans*-G1** and Cu(II) in the molar ratio of 1:1 in choloroform; (b) the powder obtained by drying of the polypseudorotaxane solution.

Furthermore, the photo-responsive property of the polypseudorotaxane was investigated by $^1H$ NMR spectroscopy. As shown in Fig. S18a–c, upon irradiation by UV light at 365 nm of ***trans*-G1**-based coordination polymer solution, new broad peaks appeared. These peaks were ascribed to the protons of $H_{a*}$, $H_{b*}$ and $H_{c*}$ of ***cis*-G1** after coordination with Cu(II). Meanwhile, the $^1H$ NMR spectrum of the polypseudorotaxane also displayed new broad peaks after irradition with UV light at 365 nm (Fig. S18e), which was due to the coordination between ***cis*-G1** and Cu(II). In addition, SEM and AFM experiments were used to study the morphology of the polypseudorotaxane after UV light irradiation. As shown in Fig. S19, the polypseudorotaxane displayed fiber-like structure after irradiation with UV light at 365 nm. These results suggested the metallosupramolecular polypseudorotaxane changed into ***cis*-G1**-based coordination polymer after irradiation by UV light at 365 nm.

## 4. Conclusion

In conclusion, a photo-responsive host–guest motif based the recognition of dimethoxylpillar[5]arene **DMP5** towards an azastilbene neutral guest ***trans*-G1** was invesitigated. A series of analogous neutral guest molecules were studied as comparisons to study the driving forces of the host–guest interaction between **DMP5** and ***trans*-G1**. Furthermore, the host–guest recognition system was used to construct a photo-responsive polypseudorotaxane by Cu(II) coordination. This host–guest recognition system based on **DMP5** and the neutral guest can be used as a new building block to fabricate supramolecular architechtures which can be applied in various fields such as sensors, supramolecular polymers and molecular machines.

## Acknowlegment

This work was supported by the National Science Foundation for Young Scientists of China (Grant 21704073) and the Natural Science Foundation for Young Scientists of Shanxi Province, China (Grant 201801D221078).

## Notes and references

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†Electronic Supplementary Information (ESI) available: $^1H$ NMR spectra, 2D NOESY NMR spectra, 2D DOSY NMR spectra, FT-IR data, XRD data and other materials. See DOI: 10.1039/c0xx00000x.

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