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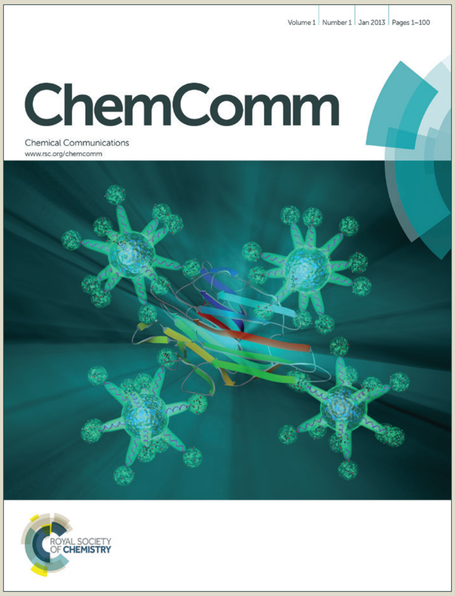

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A [2]Rota[2]catenane, Constructed from a Pillar[5]arene-Crown Ether Fused Double-Cavity Macrocycle: Synthesis and Structural Characterization

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A topologically novel [2]rota[2]catenane containing both a pillar[5]arene-imidazolium based [2]rotaxane subunit and a crown ether-tetracationic cyclophane [CBPQT]$^{4+}$ based [2]catenane one was designed and constructed. The structure of the [2]rota[2]catenane was thoroughly characterized by NMR spectra, MS spectrometry and single crystal X-ray diffraction analysis.

Mechanically interlocked molecules (MIMs), such as rotaxanes, catenanes and knots,[1] have attracted considerable interest in recent years in the fields of supramolecular chemistry, not only because of their intriguing topological appearance, but their potential applications in the field of molecular switches and machines,[2] drug delivery,[3] nanoscience,[4] and others.[5] [2]Pseudorotaxanes, characterized by a macrocyclic host component encircling a threading guest axle via non-covalent bonding interactions, such as π-π donor-acceptor, metal-ligand coordination and hydrogen bonding interactions, have been intensively used as precursors in the preparation of advanced MIMs. During recent years, many complicated and polymeric [*n*]pseudorotaxane MIMs, such as [c2]daisy chains,[6] polycatenanes,[7] hetero[*n*]rotaxanes,[8] and rotacatenane,[9] have been created using [*n*]pseudorotaxane-type complexes formed by macrocyclic host molecules along with their threading guests. Pillararenes, added to the list of macrocyclic hosts in 2008,[10] have been found to form threaded host-guest complexes with many guest species,[11] and used in the construction of MIMs in recent years.[12] Previously, we have developed a pillar[5]arene-crown ether fused bicyclic host molecule **1**, which can form [3]pseudorotaxane-type complexes by having two different guest species threading into its pillar[5]arene and crown ether cavity subunits.[13,14] Herein, we report our results on the use of **1** in the construction of topologically novel [2]rotaxane **2**, [2]catenane **3** and [2]rota[2]catenane **4** (Fig. 1).

The pillar[5]arene-crown ether fused bicyclic host **1** was synthesized by following a reported procedure.[13-15] Formation of a self-included pseudo[1]catenane[16] through rotation of the 1,4-hydroquinone unit was prevented due to the large size of the naphthalene unit in the polyether chain. Since host **1** could form a three component complex with an imidazolium ion and a bipyridinium ion, demonstrated in our previous report,[14] an attempt was thus made to synthesize topologically novel [2]rotaxane **2**, [2]catenane **3** and [2]rota[2]catenane **4** (Fig. 1) by utilizing the pillar[5]arene/imidazolium[17] and the crown ether/bipyridinium[18] host-guest motifs. In [2]rotaxane **2**, the pillar[5]arene cavity of **1** is threaded by an dumbbell-shaped imidazolium cationic thread **T**$^+$ (Fig.1) formed from **5** and **6** with its crown ether cavity unoccupied, while the crown ether ring of **1** is interlocked by a tetracationic cyclophane [**CBPQT**] $^{4+}$ ring (Fig.1) in [2]catenane **3**, leaving its pillar[5]arene cavity free. [2]Rota[2]catenane **4** is a hybrid of [2]rotaxane **2** and [2]catenane **3**. The synthesis of [2]rotaxane **2** was achieved in 50% yield by stirring a mixture of **1**, 1,3-bis(6-aminohexyl)-*1H*-imidazol-3-ium trifluoroacetate **5** and triethylamine in $CHCl_3$ at 5 °C for 30 min, followed by adding 1-naphthoyl chloride **6** to form the threading **T**$^+$ (Scheme 1). [2]Catenane **3** was synthesized in 70% yield by mixing of **1**, 1,1'-(1,4-phenylenebis(methylene))bis(4-phenylpyridin-1-ium)trifluoroacetate **7** and 1,4-di(bromomethyl)benzene **8** in DMF and then stirring for 3 days, during which the mechanically locked [**CBPQT**] $^{4+}$ ring was formed from **7** and **8** to encircle the crown ether ring of **1** (Scheme 1). Either [2]rotaxane **2** or [2]catenane **3** possesses one free macrocyclic subunit that could potentially be used to assemble one more mechanically interlocked linkage, so it's intriguing to see whether

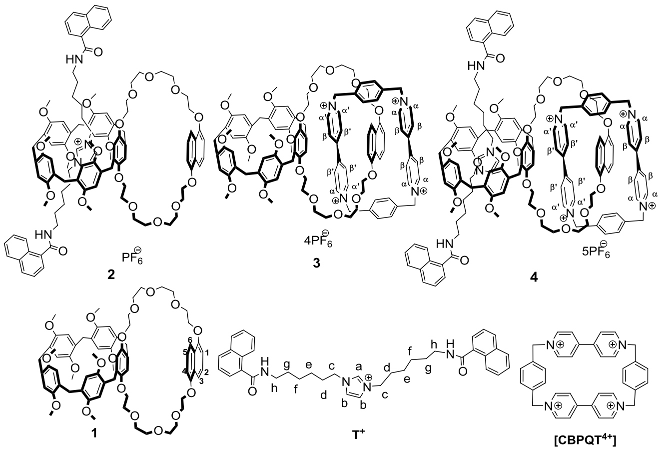
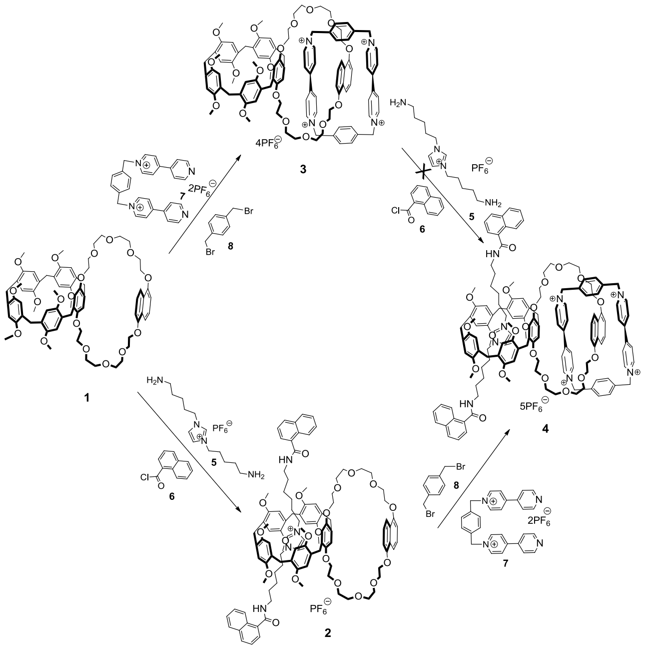


Fig. 1. The structures of **1**, [2] rotaxane **2**, [2]catenane **3**, [2]rota[2]catenane **4**, thread **T**$^+$ and tetracationic cyclophane [**CBPQT**] $^{4+}$ ring.

we can have both subunits interlocked in a hybrid structure [2]rota[2]catenane **4** (Fig. 1). It seemed to us that [2]rota[2]catenane **4** could be created by either threading **5** into the pillar[5]arene cavity of **3**, followed by capping with 1-naphthoyl chloride **6**, or encircling the crown ether ring of **2** with a **[CBPQT]$^{4+}$** ring (Scheme 1). However, our attempt to thread **5** into the pillar[5]arene cavity of **3** was not successful, which did not surprise us since we found in our previous work that a negative cooperativity existed between the two positively charged guests in their binding to the pillar[5]arene-crown ether fused bicyclic host **1**.[14] Thus, we postulate that there must be a strong negative cooperativity displayed by **[CBPQT]$^{4+}$** towards **5** which prevented its threading into the pillar[5]arene cavity of **3**. To our delight, [2]rota[2]catenane **4** was successfully constructed by following the other route - the binding of thread **7** by the crown ether cavity of [2]rotaxane **2**, followed by ring-closing *via* locking **7** with **8** to form the tetracationic cyclophane **[CBPQT]$^{4+}$** (Scheme 1). It took 20 days for **4** to be formed in 25% isolated yield, a very slow process compared to the formation of **3** in 70% isolated yield within only 3 days, because of the negative cooperativity which could be attributed to Coulombic repulsion of the positively charged dumbbell-shaped imidazolium thread **T$^+$** in [2]rotaxane **2** toward **7.**



Scheme 1. Synthesis of [2]rotaxane **2**, [2]catenane **3** and [2]rota[2]catenane **4**.

The structures of **2**, **3** and **4** were characterized by NMR, mass spectrometry, and X-ray single crystal diffraction analysis. The ^{1}H NMR spectra of **1**, [2]rotaxane **2**, [2]catenane **3**, [2]rota[2]catenane **4** and thread **T$^+$** in CD_3CN are shown in Fig. 2. Due to shielding effect of the pillar[5]arene cavity, the imidazolium protons (H_a and H_b) and the methylene protons (H_f, H_e and H_d) on the axles of **2** (Fig.2b) and **4** (Fig.2d) were upfield shifted compared to those of the free thread **T$^+$**. The significant upfield shift of the $H_{4/8}$ protons of the 1,5-dioxynaphthalene (DNP) function of the crown ether subunit in **3** (Fig.2c) and **4** (Fig.2d), relative to the corresponding proton signals of **1** (Fig.2a), indicated the strong π-donor/π-acceptor interactions of the **CBPQT^{4+}** ring with the DNP unit. The 2D NMR spectra of **2**, **3** and **4** showed the corresponding correlation signals between the in space correlated protons (ESI†), which are consistent with the 1D ^{1}H NMR spectra. It is very interesting to note that the correlation A between the proton H_b of imidazolium thread **T$^+$** and the hydroquinone protons of the pillar[5]arene subunit observed in the 2D ROSEY spectrum of [2]rotaxane **2** (Fig. 3a) disappeared in the 2D ROSEY spectrum of [2]rota[2]catenane **4** (Fig. 3b). We postulated that the disappearance of such correlations might be caused by Coulombic repulsion of the positively charged **[CBPQT]$^{4+}$** ring toward **T$^+$**, which forced the positively charged

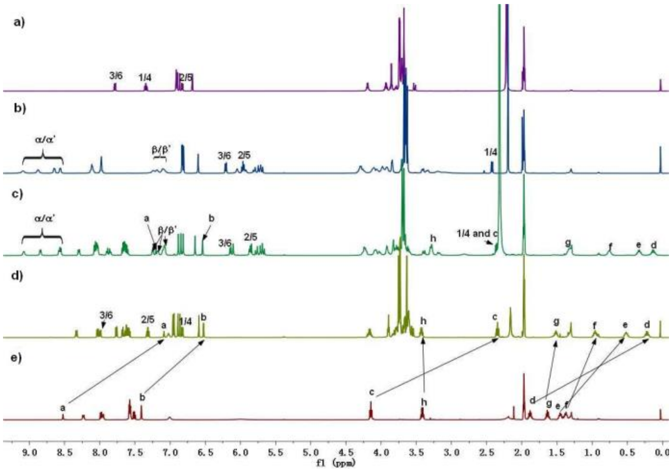
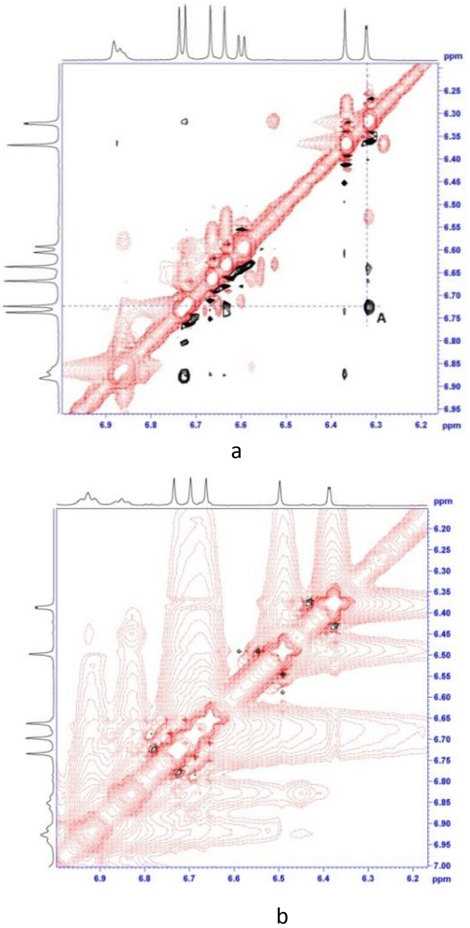


Fig. 2. ^{1}H NMR spectra of (a) bicyclic host **1**, (b) [2]catenane **3**, (c) [2]rota[2]catenane **4**, (d) [2]rotaxane **2**, and (e) thread **T** in CD_3CN.



a

b

Fig. 3. Part of 2D ^{1}H-^{1}H ROSEY NMR spectra of (a) [2]rotaxane **2**, and (b) [2]rota[2]catenane **4** in CD_3CN.

imidazolium subunit of $\mathbf{T}^+$ out of the pillar[5]arene cavity. ESI-MS results of **2**, **3** and **4** supported the structural assignment of [2]rotaxane **2**, [2]catenane **3**, [2]rota[2]catenane **4** by the presence of the peaks at *m/z* 887.4, assigned to $\mathbf{[M_2\text{-}PF_6^-{+}H^+]}$ for **2**; *m/z* 429.7, $\mathbf{[M_3\text{-}4PF_6^-]}$ for **3**; and *m/z* 458.8, 609.9, 862.1, $\mathbf{[M_4\text{-}5PF_6^-]^{5+}}$, $\mathbf{[M_4\text{-}4PF_6^-]^{4+}}$, $\mathbf{[M_4\text{-}3PF_6^-]^{3+}}$ for **4** (ESI†). Single crystals of [2]catenane **3** suitable for X-ray diffraction analysis were obtained by vapour diffusion of iso-propylether into an acetone solution of **3**. The crystal structure of **3** clearly revealed the interlocked nature of a **1**-$\mathbf{[CBPQT]^{4+}}$ based [2]catenane (Fig. 4), in which the naphtho moiety of crown ether subunit in **1** is encircled by the $\mathbf{[CBPQT]^{4+}}$ ring *via* mechanical linkage. The structure is stabilized by π-π stacking and hydrogen bonding interactions, which is consistent with the NMR analysis results obtained in solution. Single crystals of [2]rota[2]catenane **4** were harvested by vapour diffusion of THF into an acetone solution of **4**. The crystal structure of **4** clearly revealed that the dumbbell-shaped $\mathbf{T}^+$ was trapped in the pillar[5]arene cavity of **1**, while the $\mathbf{CBPQT^{4+}}$ ring encircled the naphtho moiety of crown ether subunit in **1** (Fig. 4). The imidazolium function in the thread $\mathbf{T}^+$ slipped out of the center cavity of the pillar[5]arene unit, which is consistent with the missing of correlations between the imidazolium proton of $\mathbf{T}^+$ and the hydroquinone protons of the pillar[5]arene subunit in its 2D ROSEY spectrum.

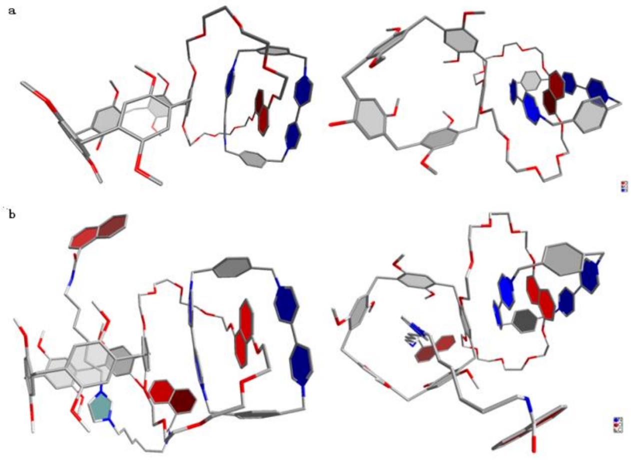


Fig. 4. Crystal structures of [2]catenane **3** (a) side view, (b) top view; and [2]rota[2]catenane **4** (c) side view, (d) top view. Color code: C (gray), N (blue), O (red). The hydrogen atoms and counter anions are omitted for clarity.

In summary, we have created three topologically novel MIMs, [2]rotaxane **2**, [2]catenane **3** and [2]rota[2]catenane **4**, using pillar[5]arene-crown ether fused bicycle **1** with either or both imidazolium thread $\mathbf{T}^+$ and tetracationic cyclophane $\mathbf{[CBPQT]^{4+}}$ ring. The synthesis of [2]rota[2]catenane **4** was significantly affected by the negative cooperativity displayed by the two threading partners towards each other in their binding to the host sites of the pillar[5]arene-crown ether fused bicycle. The structures of the three MIMs were established by 1D, 2D NMR, MS and single crystal X-ray diffraction studies.

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Notes and references

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†Electronic supplementary information (ESI) available: Full experimental details and characterization data including copies of ^{1}H NMR, ^{13}C NMR, 2D NOEY, 2D COSY and 2D ROSY spectra for **2**, **3** and **4**, X-ray crystallographic data of **3** and **4** (CIF) (CCDC 1047668 and 1047669).

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