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Synthesis of Copillar[5]arenes and Their Host-Guest Complexation with Two Types of Guests

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A series of novel copillar[5]arenes **1a**—**1f** containing different substituents were synthesized. And their complexation with two types of guests was investigated. For symmetrical guests, 1,4-dibromobutane (DBB) could thread in the cavity of copillar[5]arenes to form inclusion complexes. But for the unsymmetrical guests, copillar[5]arene **1f** bearing 4-(naphthalen-1-yloxy)butoxy could not complex with *sec*-butyl iodide (SBI) and *sec*-butyl bromide (SBB) at all, while **1f** showed weak interaction with *sec*-butylamine•HCl (SBA) outside the cavity. These results indicated that the modified group of copillar[5]arene and the symmetry of guest played an important role in the complexation model and selectivity.

Keywords copillar[5]arenes, synthesis, complexation selectivity, host-guest chemistry

Introduction

Pillar[*n*]arenes, a new type of macrocyclic hosts, *para*-bridged analogues of calixarenes, have attracted considerable attention owing to their applications in host-guest chemistry,[1-4] self-assembly chemistry,[5-7] material chemistry,[8-11] photochemistry,[12-14] fluorescence chemistry,[15-21] and biochemistry.[22,23] As pillararenes show high symmetrical and rigid pillar-like structure and have rich electron density, they exhibit excellent host-guest binding properties for a variety of neutral and electron accepting molecules by weak noncovalent interactions (van der Waals, CH-π, cation-π, H-bonding, *etc*.).[24-26] Importantly, the complexation selectivity of pillararenes towards guest molecules can be tuned by varying the shape and size of the cavity or modifying the substituents on the upper and lower rims. A complete complexation selectivity was found between pillar[5]- and pillar[6]arenes with alkyl bromides due to their different size of cavity.[27] Considering the small size cavity of pillar[5]arene among other analogues, guest molecules have to be carefully selected for host-guest complexation study. Hou *et al*.[28] developed a class of artificial transmembrane channels from peptide-appended pillar[*n*]arenes (*n*＝5, 6), and some of them had chiral selectivity for amino acid enantiomers. Recently, we reported[29] the fully symmetrical pillar[5]-arenes with different alkyl chains, in which 1,4-bis-(methoxy)pillar[5]arene (MeP5, Figure 1) displayed strong complex with *sec*-butylamine•HCl, dodecylamine•HCl and octadecylamine•HCl, but 1,4-bis-(butoxy)pillar[5]arene did not form such complex at all. However, very little is known about the complexation selectivity for non-symmetrical pillar[5]arenes, especially the monofunctionalized copillar[5]arene,[30] although this type of structure has become the central topic of many studies because of their easy functionalization and different self-assembly behavior.[31,32]

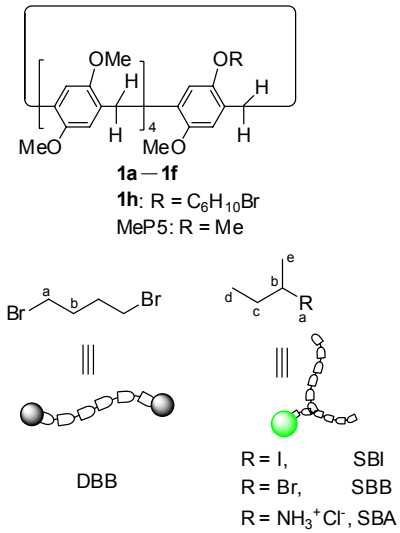

Figure 1 Structures of (co)pillar[5]arene hosts and two types of guests.

In previous work,[30,33-40] we have known that pil-

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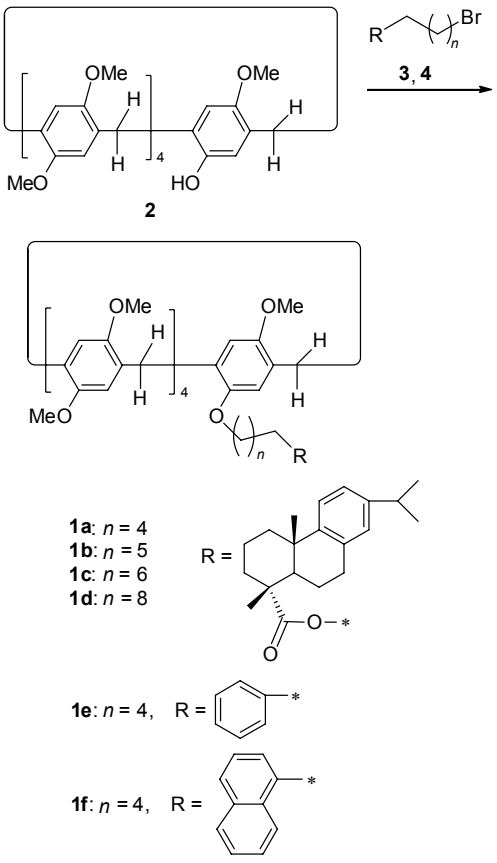
lar[5]arenes or biphen[*n*]arenes, which were first reported by Li *et al.*[41] in 2015, showed good binding properties to the symmetrical guests such as the linear *α*,*ω*-dihaloalkane, *α*,*ω*-dihydroxyalkane and *α*,*ω*-diaminoalkane by the two interactions of the cavity. In contrast, the unsymmetrical guests possessing one binding site always showed weak or no interaction. The different binding ability between these two types of guests would lead to their complexation selectivity or different complexation model. However, until now, this type of guests may not be feasible and have received less attention. Herein, we synthesized a series of novel copillar[5]arenes bearing different groups (Scheme 1) as model hosts and selected dibromoalkane (DBB), *sec*-butyl iodide (SBI), *sec*-butyl bromide (SBB) and *sec*-butylamine•HCl (SBA) (Scheme 1) as model guests to investigate their complex properties.

Scheme 1 Synthesis of novel copillar[5]arenes **1a**－**1f**



Experimental

Pillar[5]arene **2** and bromoalkane derivatives **3**, **4** were synthesized according to the literature procedure[42,43] and copillar[5]arene **1h** was previously synthesized. Solvents were either employed as purchased or dried according to the procedures described in the literature. ^{1}H NMR (600 MHz) and ^{13}C NMR (150 MHz) spectra were recorded on an Av-600 spectrometer (Brucker Co., Ltd., Switzerland) by using $CDCl_3$ as solvent and TMS as an internal standard. Mass spectra were performed on a TSQ Quantum Access MAX HPLC-MS instrument (Thermo Scientific Co., Ltd., USA).

General procedure for the preparation of copillar[5]arenes

To a solution of **2** (200 mg, 0.27 mmol) and K_2CO_3 (55 mg, 0.40 mmol) in DMF (5 mL), the corresponding bromoalkane derivative **3** or **4** (0.33 mmol) was added under a nitrogen atmosphere and the mixture was stirred at 90 ℃. After the completion of the reaction, water (50 mL) was added and the product was extracted with ethyl acetate (20 mL×3). The combined organic phase was dried over anhydrous Na_2SO_4. The crude mixture was chromatographed over silica gel column using a mixture of ethyl acetate and petroleum ether.

1a: white solid, in 81.1% yield (240 mg), m.p. 93－94 ℃; ^{1}H NMR (600 MHz, $CDCl_3$, 298 K) *δ*: 7.19 (d, *J*=8.4 Hz, 1H), 7.03 (d, *J*=7.8 Hz, 1H), 6.89 (s, 1H), 6.81－6.73 (m, 10H), 4.19－4.13 (m, 2H), 3.88 (t, *J*=6.0 Hz, 2H), 3.80－3.76 (m, 10H), 3.67－3.63 (m, 27H), 2.91－2.87 (m, 2H), 2.86－2.81 (m, 1H), 2.32 (d, *J*=12.0 Hz, 1H), 2.28 (dd, *J*=12.6, 1.8 Hz, 1H), 1.87－1.86 (m, 5H), 1.80－1.76 (m, 2H), 1.74－1.71 (m, 1H), 1.66 (d, *J*=9.6 Hz, 1H), 1.51－1.42 (m, 2H), 1.30 (s, 3H), 1.24 (s, 3H), 1.23 (d, *J*=3.0 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$, 298 K) *δ*: 178.74, 150.97, 150.92, 150.86, 149.97, 147.02, 145.90, 134.78, 128.59, 128.43, 128.42, 128.41, 128.37, 128.27, 127.09, 124.36, 124.13, 115.16, 114.30, 114.25, 114.22, 114.12, 114.03, 68.03, 64.47, 55.99, 55.93, 55.90, 55.88, 55.81, 53.12, 47.83, 45.00, 38.10, 37.13, 36.84, 33.61, 29.96, 29.75, 29.47, 26.64, 25.89, 25.40, 24.12, 21.94, 18.77, 16.69; ESI-MS *m/z*: 1091.88 ($[M+H]^+$), 1108.11 ($[M+NH_4]^+$), 1129.03 ($[M+K]^+$).

1b: white solid, in 84.3% yield (245 mg), m.p. 90－92 ℃; ^{1}H NMR (600 MHz, $CDCl_3$, 298 K) *δ*: 7.17 (d, *J*=8.4 Hz, 1H), 7.01 (dd, *J*=8.4, 1.2 Hz, 1H), 6.87 (d, *J*=1.2 Hz, 1H), 6.79－6.74 (m, 10H), 4.15－4.04 (m, 2H), 3.84 (t, *J*=6.0 Hz, 2H), 3.78－3.76 (m, 10H), 3.66－3.63 (m, 27H), 2.89－2.86 (m, 2H), 2.84－2.80 (m, 1H), 2.30 (d, *J*=12.0 Hz, 1H), 2.26 (dd, *J*=12.6, 2.4 Hz, 1H), 1.83－1.79 (m, 2H), 1.79－1.70 (m, 5H), 1.65－1.57 (m, 3H), 1.51－1.42 (m, 3H), 1.27 (s, 3H), 1.23 (s, 3H), 1.22 (s, 3H), 1.21 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$, 298 K) *δ*: 178.76, 151.97, 150.95, 150.94, 150.92, 150.90, 150.12, 147.05, 145.88, 134.81, 128.52, 128.42, 128.40, 128.36, 128.34, 128.28, 127.08, 124.35, 124.09, 115.08, 114.28, 114.24, 114.14, 114.10, 68.32, 64.58, 55.97, 55.89, 55.86, 55.81, 53.33, 47.79, 44.95, 38.09, 37.11, 36.81, 33.60, 30.29, 29.95, 29.87, 29.77, 29.60, 29.52, 28.73, 25.38, 24.14, 24.11, 23.01, 21.91, 18.75, 16.66; ESI-MS *m/z*: 1104.95 ($[M]^+$), 1122.12 ($[M+NH_4]^+$), 1142.90 ($[M+K]^+$).

1c: white solid, in 85.1% yield (250 mg), m.p. 88－89 ℃; ^{1}H NMR (600 MHz, $CDCl_3$, 298 K) *δ*: 7.19 (d, *J*=8.4 Hz, 1H), 7.02 (d, *J*=8.4 Hz, 1H), 6.89 (s, 1H), 6.81－6.77 (m, 10H), 4.10－4.05 (m, 2H), 3.84 (t, *J*=

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6.0 Hz, 2H), 3.80−3.78 (m, 10H), 3.67−3.65 (m, 27H), 2.89−2.88 (m, 2H), 2.85−2.81 (m, 1H), 2.32 (d, *J*=12.0 Hz, 1H), 2.27 (dd, *J*=12.6, 1.2 Hz, 1H), 1.88−1.77 (m, 5H), 1.75−1.70 (m, 1H), 1.68−1.65 (m, 3H), 1.56−1.51 (m, 2H), 1.46−1.39 (m, 4H), 1.29 (s, 3H), 1.24 (s, 3H), 1.23 (d, *J*=3.6 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$, 298 K) *δ*: 178.78, 150.96, 150.92, 150.91, 150.88, 150.87, 150.17, 147.08, 145.88, 134.83, 128.54, 128.50, 128.40, 128.38, 128.35, 128.33, 128.28, 127.09, 124.37, 124.10, 115.09, 114.27, 114.224, 114.21, 114.13, 114.09, 68.54, 64.69, 55.98, 55.92, 55.85, 55.82, 53.11, 47.80, 44.95, 38.12, 37.12, 36.81, 33.60, 30.30, 29.93, 29.86, 29.74, 29.50, 28.85, 26.20, 26.08, 25.39, 24.15, 24.13, 21.91, 18.77, 16.68; ESI-MS *m/z*: 1135.90 ($[M+NH_4]^+$), 1157.98 ($[M+K]^+$).

1d: white solid, in 83.4% yield (252 mg), m.p. 70−71 ℃; ^{1}H NMR (600 MHz, $CDCl_3$, 298 K) *δ*: 7.20 (d, *J*=8.4 Hz, 1H), 7.03 (d, *J*=8.4 Hz, 1H), 6.91 (s, 1H), 6.80−6.77 (m, 10H), 4.11−4.02 (m, 2H), 3.85 (t, *J*=6.0 Hz, 2H), 3.81−3.78 (m, 10H), 3.67−3.65 (m, 27H), 2.90−2.89 (m, 2H), 2.87−2.84 (m, 1H), 2.33 (d, *J*=12.0 Hz, 1 H), 2.32 (d, *J*=12.0 Hz, 1 H), 1.89−1.84 (m, 1H), 1.80−1.77 (m, 4H), 1.73 (brs, 1H), 1.69−1.65 (m, 2H), 1.62−1.60 (m, 1H), 1.52−1.50 (m, 3H), 1.45−1.43 (m, 1H), 1.36−1.34 (m, 3H), 1.30 (brs, 8H), 1.25 (s, 3H), 1.24 (d, *J*=3.3 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$, 298 K) *δ*: 178.44, 151.62, 150.57, 150.49, 149.89, 146.74, 145.52, 134.50, 128.15, 128.12, 128.07, 128.05, 128.01, 127.97, 127.92, 126.73, 124.02, 123.74, 114.80, 113.94, 113.90, 113.83, 68.28, 64.48, 55.61, 55.57, 55.55, 55.49, 47.44, 44.60, 37.78, 36.77, 36.45, 33.25, 31.70, 29.95, 29.57, 29.50, 29.48, 29.45, 29.42, 29.24, 29.22, 29.18, 28.96, 28.46, 26.04, 25.69, 25.03, 23.78, 22.47, 21.55, 18.43, 16.32, 13.92; ESI-MS *m/z*: 1147.40 ($[M+H]^+$), 1164.10 ($[M+NH_4]^+$), 1168.98 ($[M+Na]^+$).

1e: white solid, in 89.4% yield (214 mg), m.p. 124−125 ℃; ^{1}H NMR (600 MHz, $CDCl_3$, room temperature) *δ*: 7.31−7.27 (m, 2H), 6.95 (t, *J*=7.2 Hz, 1H), 6.91 (d, *J*=7.8 Hz, 2H), 6.80−6.76 (m, 10H), 4.03 (t, *J*=6.0 Hz, 2H), 3.91 (t, *J*=6.0 Hz, 2H), 3.81−3.77 (m, 10H), 3.67−3.64 (m, 27H), 2.90−2.89 (m, 4H); ^{13}C NMR (150 MHz, $CDCl_3$, 298 K) *δ*: 160.40, 152.26, 152.23, 152.21, 151.42, 130.88, 129.72, 129.67, 129.64, 129.61, 129.59, 129.56, 122.05, 116.33, 115.84, 115.59, 115.56, 115.53, 115.49, 115.45, 115.43, 69.41, 68.78, 57.25, 57.19, 57.16, 57.15, 33.32, 32.86, 31.61, 31.15, 31.10, 31.05, 30.97, 30.80, 27.89, 27.71, 24.09, 15.54; ESI-MS *m/z*: 884.59 ($[M]^+$), 901.78 ($[M+NH_4]^+$), 906.72 ($[M+Na]^+$), 922.66 ($[M+K]^+$).

1f: white solid, in 95.8% yield (242 mg), m.p. 129−130 ℃; ^{1}H NMR (600 MHz, $CDCl_3$, 298 K) *δ*: 8.29 (d, *J*=8.4 Hz, 1H), 7.81 (d, *J*=7.8 Hz, 1H), 7.50−7.43 (m, 3H), 7.38 (t, *J*=8.4 Hz, 1H), 6.83−6.75 (m, 11H), 4.23 (t, *J*=6.0 Hz, 2H), 3.98 (t, *J*=6.0 Hz, 2H), 3.83−3.77 (m, 10H), 3.66−3.61 (m, 27H), 2.20−2.14 (m, 2H), 2.11−2.06 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$, 298 K) *δ*: 156.13, 152.25, 152.21, 152.19, 152.17, 151.42, 135.93, 129.76, 129.70, 129.65, 129.62, 129.57, 128.90, 127.82, 127.31, 127.08, 126.56, 123.38, 121.58, 116.37, 115.60, 115.53, 115.46, 115.45, 115.41, 105.92, 33.31, 31.19, 31.15, 31.10, 31.04, 30.97, 30.82, 28.15, 27.74, 24.09, 15.55; ESI-MS *m/z*: 934.57 ($[M]^+$), 951.64 ($[M+NH_4]^+$), 956.62 ($[M+Na]^+$).

Results and Discussion

Figure 2 showed the ^{1}H NMR spectra of DBB in $CDCl_3$ in the absence and in the presence of approximately 1 equiv. of host **1a** (18.8 mmol•L^{-1}). The protons of DBB showed very substantial upfield shift ($\Delta\delta$=3.18 for H^b) as well as broadening effects in the presence of **1a** compared to that of free DBB. Meanwhile, the proton signals of aromatic region and methoxy of **1a** displayed downfield displacement. This result indicated that DBB was locked in the cavity. In contrast to **1a**, copillar[5]arene **1h** could form intramolecular or intermolecular self-assembled structures. On the basis of the understanding of the binding ability between *α*,*ω*-dihaloalkane and one haloalkane, our particular interest is to know whether **1h** could further complex with DBB. After complexing, the protons of DBB exhibited a larger upfield shift, while the alkyl bromide protons of **1h** shifted downfield (see Supporting Information Figure S31), which indicated that the protons of DBB were located in the deshielding region of the cyclic pillar structure, and the alkyl bromide protons of **1h** were squeezed outside the cavity.

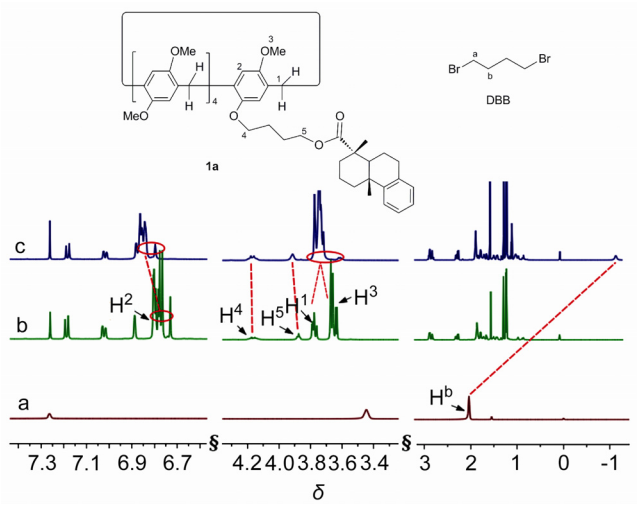

Figure 2 ^{1}H NMR spectra (600 MHz, $CDCl_3$, 298 K) of (a) DBB, (b) **1a**, and (c) an equimolar mixture of **1a** and DBB (18.8 mmol•L^{-1}).

The host-guest complexation of DBB with **1b**−**1f** containing different substituents, was also investigated. Table 1 showed the observed ^{1}H NMR up-field shifts $\Delta\delta$ of the guests in host-guest system. The large chemical shift $\Delta\delta$ of H^b indicated that all the designed copillar[5]arenes displayed excellent complex properties with

the symmetrical guest DBB. Compared with the other copillar[5]arenes, binding properties varied significantly by the type and side chain length of the substituent groups. Copillar[5]arenes possessing dehydroabietic acid group showed a better binding properties than those having phenyl or naphthyl group, and the copillar[5]arene **1a** bearing the dehydroabietic acid group with an alkyl chain having 4 carbon atoms showed the best binding property with DBB. In addition, in contrast to the full symmetrical pillar[5]arene MeP5 (its $\Delta\delta$ value was 2.5 in MeP5-DBB complex),[33] these hosts except **1f** had stronger binding abilities according to the $\Delta\delta$ values, which increased in the order of **1f**＜MeP5＜**1d**＜**1e**＝**1b**＜**1c**＜**1a**. This result suggested that the suitable substituent on the ring of pillar[5]arene could improve their binding properties in host-guest system, although the substituents were larger and longer.

To examine the influence of the different symmetrical guests on the binding interactions and avoid the interruption by the alkyl chain proton of host, we studied the complexation of **1f** with other guests SBI, SBB and SBA. Interestingly, their binding behaviors in host-guest system were very different. **1f** showed no complexation with SBI and SBB in $CDCl_3$, and its interaction with SBA was weaker than DBB in accordance to smaller upfield shifts (Table 1). The signals of the protons ($H^{1\text{-}4}$) of **1f** remained nearly unchanged (Figure 3), that is, **1f** did not form an inclusion complex with SBA. This result was further proved by the 2D NOESY analysis (see Supporting Information Figure S35). NOE correlations were not observed between the protons of **1f** and the alkyl protons of SBA, suggesting that the alkyl group was outside rather than into the cavity of **1f**.

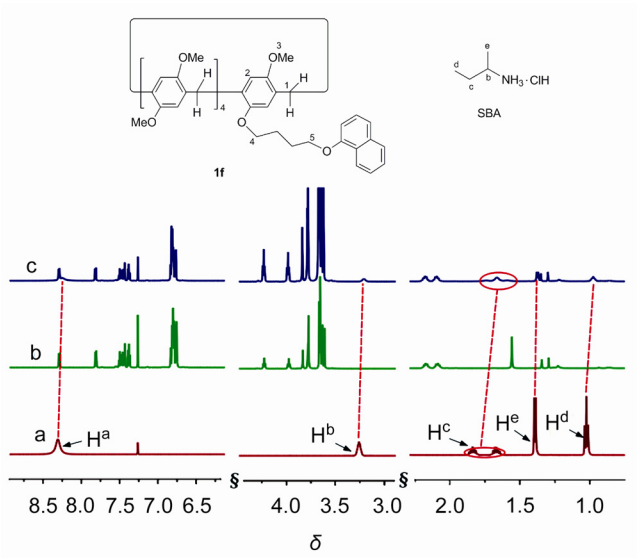

Figure 3 ^{1}H NMR spectra (600 MHz, $CDCl_3$, 298 K) of (a) SBA, (b) **1f**, and (c) an equimolar mixture of **1f** and SBA (18.8 mmol•L^{-1}).

Based on the information gained from the above results, the complexation models of copillar[5]arenes with guests were deduced as shown in Figure 4. It should be emphasized that pillar[5]arene could complex with terminal one haloalkane such as **1h**, which could form self-complex by its side chain and cavity. But for the nonterminal one haloalkane, copillar[5]arene did not form such complex, leading to the complexation selectivity between these two types of guests. On the other hand, the full symmetrical pillar[5]arene MeP5 could bind the guest SBA. However, when the alkyl substituent was bulkier than methyl group, such complex was not formed, even though the group was replaced by ethyl substituent. But for copillar[5]arene, the introduced larger and longer substituent such as 4-(naphthalen-1-yloxy)butyl group as side chain, could complex with SBA, indicating the good advantage of copillar[5]arene for their host-guest properties toward this type of guest.

Table 1 Upfield shifts of two types of guest molecules ([G]＝18.8 mmol•L^{-1}, $CDCl_3$, 298 K) in different host-guest complexation[a]

Guest	Proton	Δδ(**1a**)	Δδ(**1b**)	Δδ(**1c**)	Δδ(**1d**)	Δδ(**1e**)	Δδ(**1f**)
DBB	H^b	3.18	2.93	2.96	2.85	2.93	2.45
	H^a						0.05
	H^b						0.06
SBA	H^c	—	—	—	—	—	0—0.17
	H^d						0.05
	H^e						0.02
	H^b						0
SBI	H^c	—	—	—	—	—	0
	H^d						0
	H^b						0
SBB	H^c	—	—	—	—	—	0
	H^d						0

[a] —: The binding property between the host and guest was not tested.

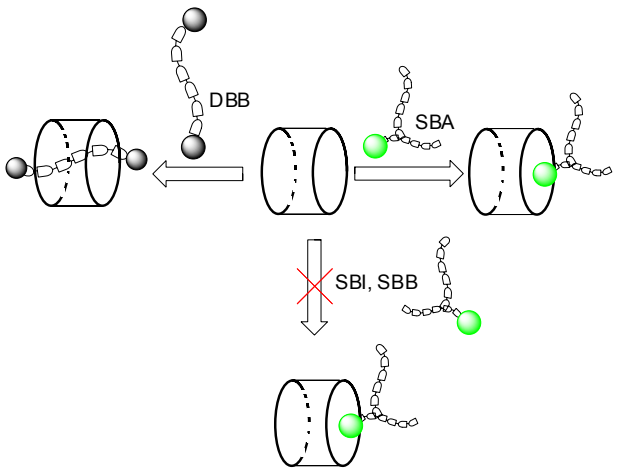

Figure 4 The complexation models of copillar[5]arenes with guests.

Conclusions

In summary, we synthesized a series of novel copillar[5]arenes bearing different groups and studied their complexation with two types of guests DBB, SBI, SBB and SBA. For symmetrical guests, DBB can thread in the cavity of copillar[5]arenes to form inclusion complexes. The binding ability of copillar[5]arenes increases in the order of **1f**＜**1d**＜**1e**＝**1b**＜**1c**＜**1a**. But for the unsymmetrical guests, **1f** can not complex SBI, SBB at all, and shows weak interaction with SBA. Furthermore, the binding models of copillar[5]arene between DBB and SBA are very different. Such complexation selectivity would lead to the application of copillar[5]arene for recognition and extraction of the symmetrical dibromoalkane in organic media.

Acknowledgement

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