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Host-guest complexation between simple pillar[5]arene and a new type of neutral guests

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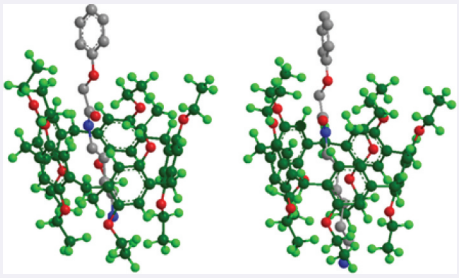
ABSTRACT

Pillar[5]arenes are a new type of supramolecular hosts constructed from hydroquinone and their derivatives linked by methylene units. Searching new host-guest interaction between pillar[5]arenes and neutral guests are thus great interesting. Here, four neutral guests (**AA0, AA2, AA4** and **AA6**) with both amino and amide groups were prepared from phenol by two steps. The host-guest interactions between perethylated pillar[5]arene (**EtP5**) and guest molecules were investigated in detail by various technologies, including ^{1}H NMR, ^{13}C NMR, 2D NOESY NMR, MS analysis and DFT calculation. We found that the guests (**AA4** and **AA6**) with longer alkyl chain can form a stable inclusion complex with **EtP5** through C–H···O, N–H···O, C–H···N and C–H···π interactions while shorter guests (**AA0** and **AA2**) could not.



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Pillar[5]arene; complex; supramolecular chemisty; host-guest interaction; neutral guests

Introduction

Pillar[5]arenes, which possess pillar-like structures, are the fifth classical macrocycles constructed from derivatives of hydroquinone connected by methylene bridges at their *2,5* positions[1]. Pillar[5]arenes can be easily functionalised with different groups, making them widely application in various areas, such as circular catalysis, drug delivery, molecular/ion detection, self-assembly materials, supramolecular gels, and so on[2]. The investigations of host-guest interactions of pillar[5]arenes are great important interest in supramolecular chemistry due to they are the main strategy and could make this area development quickly. Up to now, there are three types of guest molecules suitable for pillar[5]arenes: (I) Cationic guests, such as quaternary ammonium, pyridin-1-ium, ferrocenium, tropylium and imidazol-3-ium[3]. (II) Anionic guests, such as sulfonate and carboxylate guests[4]. (III) Neutral guests, such as nitrogen heterocyclics, nitriles, unsaturated aliphatic hydrocarbons and so on[5]. However, most of the host-guest studies of pillar[5]arenes are focus on cationic and anionic guests. The complexation of pillar[5]arenes with neutral guests in an organic solvent is very special, since other classical macrocycles such as calixarene, crown ethers generally no interact with neutral guests [5].[d-f] So the development of neutral guests is very important, according to these new-found recognition motifs, various of supramolecular structures and self-assemblies can be constructed [6,7].

Herein, we designed and prepared four neutral guests (**AA0, AA2, AA4** and **AA6**) with both amino and amide groups from phenol by two steps. The host-guest interactions between perethylated pillar[5]arene (**EtP5**) and guest molecules were investigated in detail by various technologies, including ^{1}H NMR, ^{13}C NMR, 2D NOESY NMR, MS analysis and DFT calculation. We found that the guests (**AA4** and **AA6**) with longer alkyl chain can form a stable

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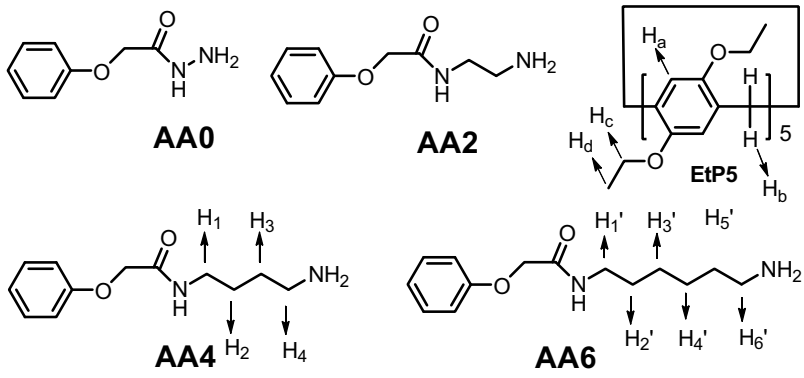

Scheme 1. Chemical structures of neutral guests (**AA0, AA2, AA4** and **AA6**) and perethylated pillar[5]arene (**EtP5**).

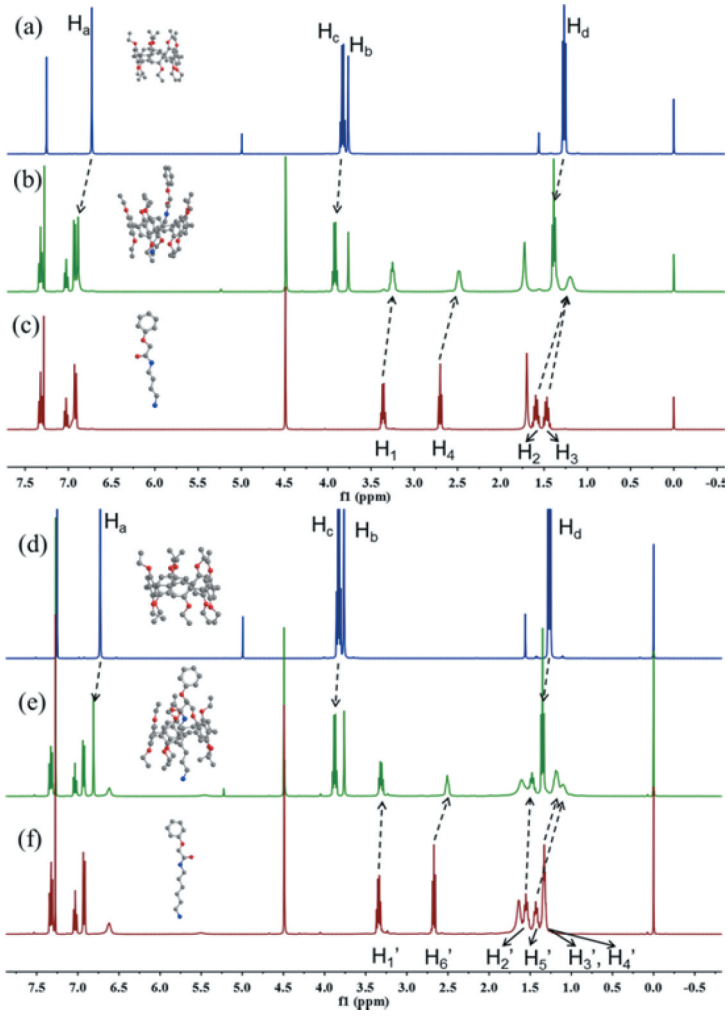

Figure 1. ^{1}H NMR spectra (400 MHz, 298 K, $CDCl_3$) of (a) **EtP5**, (b) **EtP5⊃AA4**, (c) **AA4**, (d) **EtP5**, (e) **EtP5⊃AA6**, and (f) **AA6**. [**EtP5**] = [AA4]= [AA6]= 5.00 mmol.

inclusion complex with **EtP5** via C–H···O, N–H···O and C–H···π interactions while shorter guests (**AA0** and **AA2**) could not.

Results and discussion

The neutral guests (**AA0, AA2, AA4** and **AA6**) were prepared from phenol by two steps as shown in scheme S1.

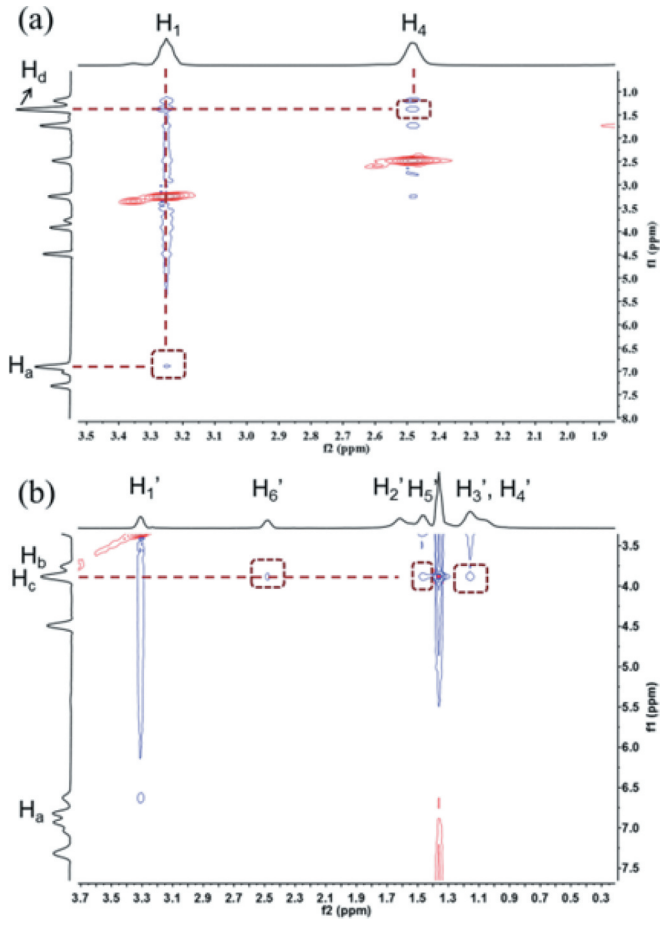

Figure 2. Partial NOESY spectra (400 MHz, 298 K, $CDCl_3$) of (a) **EtP5⊃AA4** and (b) **EtP5⊃AA6.**

Firstly, equal equivalent phenol and methyl 2-chloroacetate were reacted in acetone with KI as a catalyst under N_2 condition to afford methyl 2-phenoxyacetate (**1**). Then, compound **1** and diamine reflux in toluene to give neutral guests. It should be pointed that in the reaction, the amount of diamine is greatly excessive to confirm one amine group react with **1**. The successful preparation of neutral guests were confirmed by ^{1}H NMR (Figure S2), ^{13}C NMR (Figure S3) and MS analysis (Figure S4).

With the new type of neutral guests in hand, we then used [1]H NMR to investigate their host-guest interaction with **EtP5**. As shown in Figures S5 and S6, after addition of 1.00 equivalent of **EtP5** into the solution of guest **AA0** (or **AA2**), the signals of protons on **AA0** (or **AA2**) could not find any change, indicating that there is no obvious host-guest interaction between **EtP5** and **AA0** (or **AA2**). However, for guest **AA4** (Figure 1(a-c)), after addition of 1.00 equivalent of **EtP5**, the peaks for the methylene protons (H_1, H_2, H_3, and H_4) of **AA4** exhibit upfield shifts and broadening effects compared to the free guest (δ H_1: 3.35→3.24; H_4: 2.69→2.48; H_2: 1.60→1.20; H_3: 1.47→1.20, respectively). Similarity, after addition of 1.00 equivalent of **EtP5** into the solution of **AA6** (Figure 1(d-e)), the peaks for the methylene protons ($H_{1'}$, $H_{2'}$, $H_{3'}$, $H_{4'}$, $H_{5'}$, $H_{6'}$) of **AA6** also exhibit upfield shifts effects (δ $H_{1'}$: 3.36→3.30; $H_{6'}$: 2.65→2.50; $H_{2'}$: 1.55→1.47; $H_{3'}$: 1.43→1.10; $H_{4'}$, $H_{5'}$: 1.33→1.17, respectively). The above inclusion-induced shielding effects indicated that **EtP5** is threaded by guest **AA4** or **AA6**.

The host-guest interaction between **AA4** and **EtP5** was then studied by 2D NOESY analysis. As outlined in Figure 2(a), unequivocal correlation peaks between **AA4** protons H_1 and H_4 with the **EtP5**'s proton H_a and H_d could be observed, also revealing the formation of the inclusion complex. Similarity, as shown in Figure 2(b), NOE signal was observed between $H_{3'}$, $H_{5'}$, $H_{6'}$ of **AA6** and Hc of **EtP5**. The structure of the complex **EtP5⊃AA4** and **EtP5⊃AA6** were further confirmed by HRESIMS (ESI, Figures S7 and S8). HRESIMS revealed peaks at m/z 1113.6425, 1141.6747, corresponding to [**EtP5⊃AA4** + H]$^+$ and [**EtP5⊃AA6** + H]$^+$, indicating a 1:1 stoichiometry for the complexation.

The ability of **EtP5** to form a 1:1 complex with **AA4** was assessed by ^{1}H NMR titration of **EtP5** into a 10.00 mM

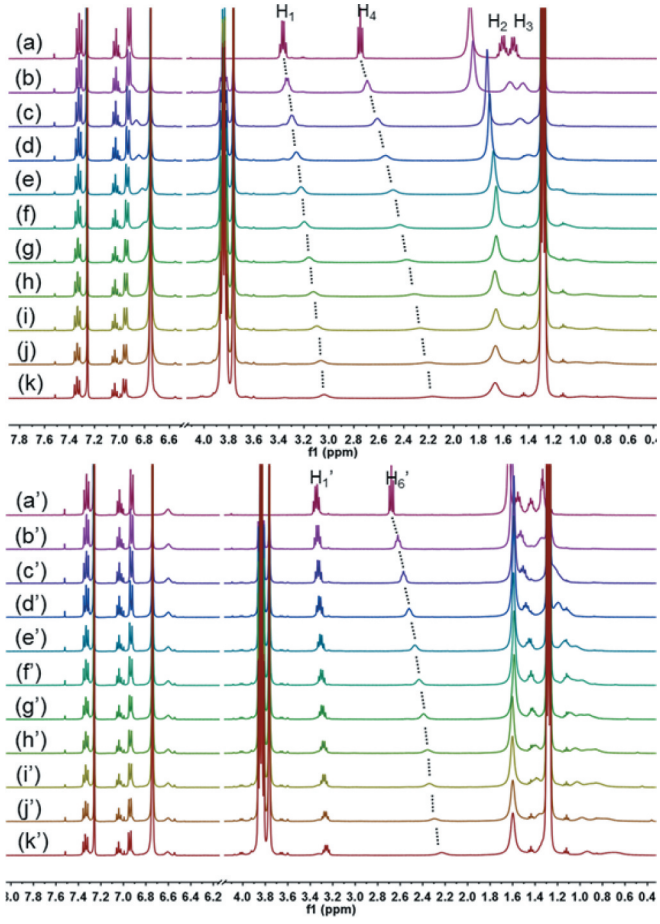

Figure 3. ^{1}H NMR spectra ($CDCl_3$, 293 K, 400 MHz) of **AA4** (upper spectra) or **AA6** (dower spectra) at a concentration of 10 mM upon different concentration of **EtP5**: (a) and (a') 0.00 mM, (b) and (b') 2.0 mM, (c) and (c') 4.0 mM, (d) and (d') 6.00 mM, (e) and (e') 8.0 mM, (f) and (f')10.0 mM, (g) and (g') 12.0 mM, (h) and (h') 14.0 mM, (i) and (I') 16.0 mM, (j) and (j') 18.0 mM, (k) and (k') 20.0 mM.

solution of **AA4** in $CDCl_3$ (Figure 3(a-k)). When the concentration of **EtP5**increased, proton NMR signals corresponding to H_1 and H_4 shifted upfield considerably. The same phenomenon also found in the ^{1}H NMR titration experiment of **EtP5** into guest **AA6** (Figures 3(a'-k')). A mole ratio plot for the complexation between **EtP5** and **AA4** (or **EtP5** and **AA6**) showed that the stoichiometry of the complex between **EtP5** and **AA4** (or **AA6**) is 1:1 (Figure S9, ESI), in accordance with the above-mentioned results of HRESIMS. The association constant (*Ka*) of **EtP5⊃AA4** and **EtP5⊃AA6** was calculated to be $(1.33 \pm 0.23)*10^3M^{-1}$ and $(2.33 \pm 0.23)*10^3M^{-1}$ in $CDCl_3$ using a nonlinear curve-fitting analysis according the equation '$\Delta\delta = (\Delta\delta_\infty/[G]_0)\ (0.5[H]_0 + 0.5([G]_0 + 1/Ka) - (0.5([H]_0^2 + (2\,[H]_0(1/Ka - [G]_0)) + (1/Ka + [G]_0)^2)^{\circ.5}))$', respectively,[8].

In order to further investigate the formation of the inclusion complex, DFT calculation of **EtP5⊃AA4** and **EtP5⊃AA6** (Figure 4) at the B3LYP/6-31 G (D) level using the PCM model in $CHCl_3$ was taken out. We can see that the guest **AA4** or **AA6** is included in the cavity of the **EtP5** host, which is consistent with the result from ^{1}H NMR and MS studies. The calculated structure of **EtP5⊃AA4** is shown in Figure 4(a-c). There exit weak C–H···O and amide N–H···O hydrogen bonds between the axle's CH_2 and the host's O atoms (Figure 4(a)), multiple C–H···π interactions between axle's CH_2 and host's dialkoxybenzene units (Figure 4 (b)), and C–H···O or C–H···N hydrogen bonds between the host's ethyls and the guest's N or O atoms (Figure 4(c)). Similarity, the weak hydrogen bonds and C–H···π interactions can be found in the calculated structure of **EtP5⊃AA6** (Figure 4(d-f)).

Conclusions

In summary, four new neutral guests (**AA0, AA2, AA4** and **AA6**) with both amino and amide groups were prepared from phenol by two steps. The host-guest interactions between perethylated pillar[5]arene (**EtP5**) and guest molecules were then investigated

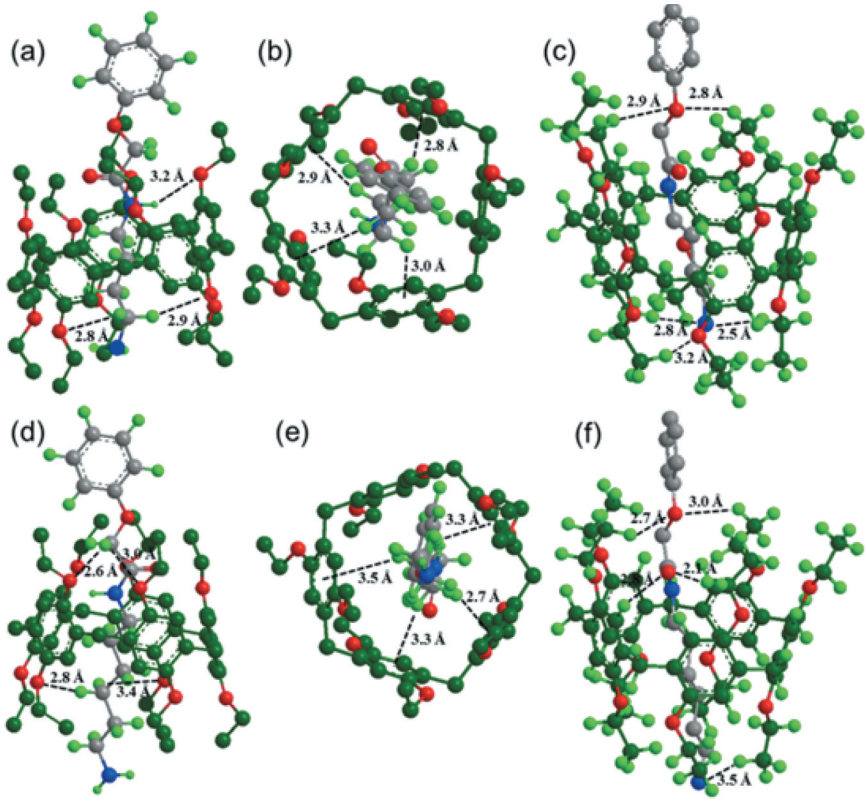

Figure 4. The optimised geometry of **EtP5⊃AA4** (a-c) and **EtP5⊃AA6** (d-f) at the B3LYP/6-31 G (D) level using the PCM model in $CHCl_3$, where only the hydrogens in question are given for clarity. (a) C–H□O hydrogen-bond, H□O distances, 2.8 Å, 2.9 Å (H on **AA4**); amide N–H□O hydrogen-bond, H□O distance, 3.2 Å. (b) C–H□π interactions, H□ring centre distances, 2.8 Å, 2.9 Å, 3.0 Å, 3.3 Å. (c) C–H□O hydrogen-bond, H□O distances, 2.8 Å, 2.9 Å, 3.2 Å (H on **EtP5**); C–H□N hydrogen-bond, H□O distances, 2.5 Å, 2.8 Å. (d) C–H□O hydrogen-bond, H□O distances, 2.6 Å, 2.8 Å, 3.0 Å, 3.4 Å (H on **AA6**) (e) C–H□π interactions, H□ring centre distances, 2.7 Å, 3.3 Å, 3.3 Å, 3.5 Å. (f) C–H□O hydrogen-bond, H□O distances, 2.7 Å, 2.7 Å, 2.8 Å, 3.0 Å (H on **EtP5**); C–H□N hydrogen-bond, H□O distances, 3.5 Å.

in detail by various methods, including ^{1}H NMR, ^{13}C NMR, 2D NOESY NMR, MS analysis and DFT calculation. We found that the neutral guests (**AA4** and **AA6**) with longer alkyl chain can form a stable inclusion complex with **EtP5** through combined C–H···O, amide N–H···O, C–H···π and C–H···N interactions while shorter neutral guests (**AA0** and **AA2**) could not. We believe the host-guest motifs developed in this work can be applied in the construction of more smart supramolecular assemblies.

Experimental section

Phenol, methyl 2-chloroacetate, K_2CO_3, KI, acetone, hydrazine hydrate, ethylenediamine, butyl diamine, and hexamethylenediamine were reagent grade and used as received. Solvents were either employed as purchased or dried according to procedures described in the literature. **EtP5** and compound **1** were synthesised according to previous reports[9]. The detail methods can be found in the supporting information part.

Syntheses of neutral guests

Take guest **AA0** as an example, in a 50 mL round-bottomed flask **1** (0.1 mmol), hydrazine hydrate (2.0 mmol) was refluxed in 25 mL CH_3CH_2OH for 24 h. The reaction mixture was then washed with water 3 times and recrystallised in methanol to afford **AA0** (yield: 67.5%) of a white power product.

AA0: white solid, yield: 67.5%, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (s, 1H, C-NH), 7.33 (t, *J* = 8.0 Hz, 2 H, ArH), 7.04 (t, *J* = 8.0 Hz, 2 H, ArH), 6.91 (t, *J* = 8.0 Hz, 1H, ArH), 4.58 (s, 2 H, -CH_2-), 3.91 (s, 2 H, -NH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.11, 158.23, 129.90, 121.53, 115.07, 66.60. MS (m/z): HRMS (ESI) Calcd. for $C_8H_{11}N_2O_2^+$ ([Ma + H]$^+$): 167.0821, found: 167.0028.

AA2: white solid, yield: 54.6%, ^{1}H NMR (400 MHz, $CDCl_3$) δ7.32 (t, *J* = 8.0 Hz, 2 H, ArH), 7.03 (t, *J* = 8.0 Hz, 1H, ArH), 6.93 (t, *J* = 8.0 Hz, 2 H, ArH), 4.51 (s, 2 H, -CH_2-),

3.43–3.38 (m, 2 H, N-CH_2-), 2.86 (t, J = 8.0 Hz, 2 H,-CH_2-NH_2). ^{13}C NMR (101 MHz, $CDCl_3$) δ 169.43, 168.67, 157.17, 129.80, 122.10, 114.65, 67.31, 41.56, 41.25. MS (m/z): HRMS (ESI) Calcd. for $C_{10}H_{15}N_2O_2^+$ ([Mb + H]$^+$): 195.1134, found: 195.1055.

AA4: white solid, yield: 83.4%; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (t, J = 8.0 Hz, 2 H, ArH), 7.02 (t, J = 6.0 Hz, 1H, ArH), 6.93 (t, J = 8.0 Hz, 2 H, ArH), 4.49 (s, 2 H, -CH_2-), 3.38–3.34 (m, 2 H, N-CH_2-), 2.76 (t, J = 8.0 Hz, 2 H,-CH_2-NH_2), 1.70–1.56 (m, 2 H, -CH_2-), 1.50–1.43 (m, 2 H, -CH_2-). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.85, 157.25, 129.74, 121.97, 114.72, 67.19, 40.17, 38.52, 26.60, 26.58. MS (m/z): HRMS (ESI) Calcd. for $C_{12}H_{19}N_2O_2^+$ ([Mc + H]$^+$): 223.1447, found: 223.1368.

AA6: white solid, yield: 64.3%, ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (t, J = 8.0 Hz, 2H, ArH), 7.03 (t, J = 6.0 Hz, 1H, ArH), 6.92 (d, J = 8.0 Hz, 2H, ArH), 4.49 (s, 2H, -CH_2-), 3.37–3.30 (m, 2H, N-CH_2-), 2.67 (t, J = 8.0 Hz, 2H,-CH_2-NH_2), 1.55–1.53 (m, 4 H, -CH_2-), 1.43–1.41 (m, 2H, -CH_2-), 1.34–1.31 (m, 4 H, -CH_2-).^{13}C NMR (101 MHz, $CDCl_3$) δ 168.19, 168.17, 157.18, 129.79, 122.10, 114.64, 67.32, 67.31, 41.87, 38.93, 33.14, 29.49, 29.42, 26.61, 26.44, 26.31. MS (m/z): HRMS (ESI) Calcd. for $C_{14}H_{23}N_2O_2^+$ ([M + H]$^+$): 251.1760, found: 251.1681.

Disclosure statement

No potential conflict of interest was reported by the authors.

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