

Interactions of α , ω -alkyldiammonium with inverted cucurbit[6]uril

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Abstract Molecular interactions between an inverted cucurbit[6]uril (*i*Q[6]) with α,ω -alkyldiammonium guests ($\text{H}_3\text{N}(\text{CH}_2)_x\text{NH}_3\text{Cl}_2$, $x = 2, 4, 6, 8, 10$, and 12 named **1–6**) were investigated in aqueous and acidic solutions by various techniques. ¹H NMR spectroscopy revealed that only compounds **3** and **4** underwent an equilibrium interaction with the *i*Q[6] host, with association constants in acidic solution of ~ 83 and ~ 41 L mol⁻¹, respectively, while the other guests showed no obvious interaction. Only two host–guest interaction systems gave rise to single crystals in the presence of polychloride transition metal anions as structure-directing agents. X-ray diffraction revealed that guests **1** and **3** interacted with *i*Q[6] through portal and cavity interactions, respectively.

Keywords Inverted cucurbit[6]uril · α,ω -alkyldiammonium · Host–guest interaction · Titration ¹H NMR experiments · X-ray diffraction analysis

Introduction

We recently found a convenient way to isolate an inverted cucurbit[6]uril (*i*Q[6]) from its normal cucurbit[6]uril (Q[6]) companion [1] and an inverted cucurbit[7]uril (*i*Q[7]) from

its water soluble mixture [2] using a simple Dowex (H⁺) column. The significant difference in retardation factors (Rf) of *i*Q[6] and *i*Q[7] from other common Q[*n*]s could be attributed to the subtle differences in the outer surface electropositive properties of Q[*n*] and *i*Q[*n*], facilitating the facile separation of inverted *i*Q[6] or *i*Q[7] from their companion compounds. A characteristic feature of inverted cucurbit[*n*]urils (*i*Q[*n*]s, $n = 6, 7$) is the movement of the two methine hydrogen atoms of the glycoluril unit into the cavity, as first identified by Isaacs and Kim [3]. However, progress on *i*Q[*n*]s has been minimal [4, 5], possibly due to the difficulty of isolating *i*Q[*n*]s from their common Q[*n*] companions. Gejji and co-workers [6, 7] recently employed density functional theory to derive the electronic structure and NMR chemical shifts of *i*Q[*n*] hosts and inclusion complexes with particular cationic guests, and predicted that inverted cucurbit[*n*]urils could exhibit distinct selectivity towards guests with the appropriate shape and dimensions for fitting the internal cavity. Indeed, our recent work confirmed that inverted cucurbit[*n*]urils possess different properties from those of their normal companions [2, 8]. For example, Isothermal titration calorimetry (ITC) experiments revealed that values of ΔS were unusually negative for *i*Q[7]-alkaline earth cation (AE²⁺) interaction systems, but were positive for Q[7]-AE²⁺ interaction systems in neutral aqueous solution. Moreover, titration ¹H NMR experiments revealed that interaction of 1,2-ethylenediamine with *i*Q[7] is mediated via a portal interaction [2], whereas interaction with Q[7] occurs in the cavity [8]. Similarly, *i*Q[6] differs in behaviour from Q[6]; coordination of *i*Q[6] with lanthanide cations (Ln³⁺) in the presence of the tetrachlorozincate anion [ZnCl₄]²⁻ as a structure-directing agent in aqueous acidic solution (3 M HCl) gave rise to different interaction products and isomorphous groups. Interactions were correlated with

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increasing atomic number as follows; interaction of *i*Q[6] with La³⁺ and Ce³⁺ immediately yielded precipitates; simple molecular bowl-like complexes were obtained with Pr³⁺ and Nd³⁺; coordination products of *i*Q[6] with Sm³⁺ and Eu³⁺ remained in solution; linear zigzag coordination polymers of alternating *i*Q[6] and lanthanide ions were obtained with Gd³⁺, Tb³⁺, Dy³⁺, and Ho³⁺; linear zigzag supramolecular chains containing alternating bowl-like and capsule-like complexes resulted from interaction with Er³⁺, Tm³⁺, Yb³⁺; Lu³⁺ produced linear zigzag supramolecular chains of Lu³⁺–*i*Q[6] pairs. In contrast, under the same conditions, Q[6] gave rise to only two groups: precipitates with La³⁺, Ce³⁺, Pr³⁺ and Nd³⁺, and linear zigzag coordination polymers with all other Ln³⁺ ions.

In the present work, we focus on host–guest interactions between *i*Q[6] and a series of α,ω -alkyldiammonium guests (H₂N(CH₂)_xNH₂, x = 2, 4, 6, 8, 10, and 12, named 1–6 respectively) (Fig. 1). The *i*Q[6]-based compounds are even less soluble than those in Q[6] systems in neutral aqueous solutions, therefore experiments were performed in 4 M HCl solutions. ¹H NMR spectroscopy showed that *i*Q[6] only underwent specific interactions with guests 3 and 4. Interestingly, binding and dissociation of *i*Q[6] and guests was discernible in the ¹H NMR spectra, suggesting a weak interaction between *i*Q[6] and guests 3 or 4, consistent with their association constants, which were estimated to be ~83 and ~41 L mol⁻¹, respectively. In contrast, the Q[6] host displays a stronger interaction with all α,ω -alkyldiammonium guests [9, 10]. Crystal structures of *i*Q[6] with guests 1 and 3 were obtained with the assistance of polychloride transition metal anions as structure-directing agents. Similar to the Q[6] host, *i*Q[6] interacted with guest 1 via a portal interaction but interacted with guest 3 to form a cavity inclusion complex.

Experimental

Materials and methods

α,ω -alkyldiamanes (H₂N(CH₂)_xNH₂, x = 2, 4, 6, 8, 10, and 12) were purchased from Aldrich and used as supplied

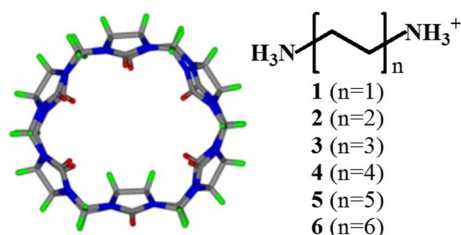


Fig. 1 Structures of the inverted cucurbit[6]uril (*i*Q[6]), α,ω -alkyldiammonium guests (H₂N(CH₂)_xNH₃Cl₂, x = 2, 4, 6, 8, 10, and 12) named 1–6

without further purification. α,ω -alkyldiammonium guests (H₃N(CH₂)_xNH₃Cl₂, x = 2, 4, 6, 8, 10, and 12 named 1–6) were prepared by protonation of the corresponding amines with concentrated HCl. *i*Q[6] was prepared according to a literature method [3], and separated according to the method as we recently reported [1].

Preparation of compounds A and B

*i*Q[6] (20.0 mg, 0.02 mmol) and ZnCl₂ (13.9 mg, 0.10 mmol) were dissolved in 1.0 mL of 6 M of HCl to prepare solution (V1), the guest 1 (18.9 mg, 0.14 mmol) was dissolved in deionized water to prepare solution (V2), which was then added with stirring to solution V1. X-ray quality crystals (compound A) could be obtained from the solution after a period of 10 days. *i*Q[6] (20.0 mg, 0.02 mmol) was dissolved in 1.0 mL 6 M of HCl to prepare solution (V3), Zn(NO₃)₂ (35.4 mg, 0.12 mmol) and guest 3 (19.8 mg, 0.17 mmol) were dissolved in 1.0 mL of water to prepare solution (V4), which was then added with stirring to solution V3. X-ray quality crystals (compound B) could be obtained from the solution after a period of 10 days. Crystal data and structure-refinement details for compounds A and B were given in table S1. Anal. calcd. for compound *i*Q[6][ZnCl₄]·0.5·1·H₃O·17H₂O (A), C₃₇H₇₇N₂₅O₃₀ZnCl₄ (%): C, 28.50; H, 4.98; N, 22.46, found: C, 28.44; H, 5.01; N, 22.51. Anal. calcd for compound {3@*i*Q[6]}·[ZnCl₄]²⁻·[Zn(H₃O)Cl₃]⁻·18H₂O (B), C₄₂H₉₂N₂₆O₃₁Zn₂Cl₇ (%): 27.47; H, 5.05; N, 19.83, found: C, 27.44; H, 5.09; N, 19.86.

X-ray crystallography

A suitable single crystal (~0.2 × 0.2 × 0.1 mm³) was embedded in paraffin oil and mounted on a Bruker SMART Apex II CCD diffractometer equipped with a graphite-monochromator Mo K α radiation source ($\lambda = 0.71073 \text{ \AA}$, $\mu = 0.828 \text{ mm}^{-1}$) that was operated in the ω -scan mode under a nitrogen stream (–50 °C). Data were corrected for Lorentz and polarization effects using the SAINT program, and semi-empirical absorption corrections based on equivalent reflections were also applied using SADABS. The structure was elucidated through direct methods and refined by the full-matrix least-squares method on F² values using SHELXS-97 and SHELXL-97, respectively [11, 12]. All non-hydrogen atoms were refined anisotropically, and carbon-bound hydrogen atoms were introduced at calculated positions and treated as riding atoms with an isotropic displacement parameter equal to 1.2 times that of the parent atom. Most of the water molecules in A and B were omitted using the SQUEEZE option in the PLATON program (17 and 18 water molecules for compounds A and B, respectively). Analytical expressions for neutral-

atom scattering factors were employed, and anomalous dispersion corrections were incorporated. Details of the crystal parameters, data collection conditions, and refinement statistics are summarized in Table S1, and crystallographic data have been deposited at the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-1422875 (A), 1422876 (B). These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/data_request/cif, by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK (fax: +44 1223 336033).

Results and discussion

Interactions between the common Q[6] hosts and alkyamine and alkyldiamine hydrochloride guests have been extensively investigated since Q[6] compounds were first reported [9, 13–15], and no obvious interaction between Q[6] with α,ω -alkyldiamines ($\text{H}_2\text{N}(\text{CH}_2)_x\text{NH}_2$, $x < 3$) has been observed in 50 % formic acid [14]. We recently showed that the interaction of a symmetrical tetramethylcucurbit[6]uril (TMeQ[6]) with ethylenediammonium dichloride in D_2O occurred at the portal position of TMeQ[6] [10], whereas *i*Q[6] incorporated the same guest in 4 M DCI/ D_2O solution in the present study, titration ^1H NMR spectra appeared to indicate no obvious changes for either host or guest proton signals (Fig. 2, left). However, a closer inspection revealed that the methylene proton resonance of guest **1** experienced a downfield shift with increasing amounts of guest (Fig. 2, right), and a similar result was observed in *i*Q[6]-5 and *i*Q[6]-6 host–guest interaction systems (Figure S1 and S2, Supporting Information), and there was almost no change in the *i*Q[6]-2 system (Figure S3, Supporting Information). The titration ^1H NMR spectra of all four host–guest interaction systems showed only one set of proton resonances for the guests, suggesting that (1) free and bound (portal and cavity) guest molecules have a high exchange ratio for binding and unbinding on the NMR time scale, and (2) portal interaction and cavity inclusion of the host and guest are competitive. The slightly upfield shift of the alkyl proton resonances indicates that the cavity inclusion dominates in the *i*Q[6]-1, *i*Q[6]-5 and *i*Q[6]-6 systems, while the free (portal) and cavity (inclusion) interactions are balanced in *i*Q[6]-2. This appears to be a common property of *i*Q[6] hosts, since the interaction of *i*Q[6] with *p*-phenylenediaminium guests gave rise to two interaction models in another of our recent studies that showed portal and cavity interactions in the crystal structure, but titration ^1H NMR spectra showed that *i*Q[6] preferred to include the *p*-phenylenediaminium guest [16].

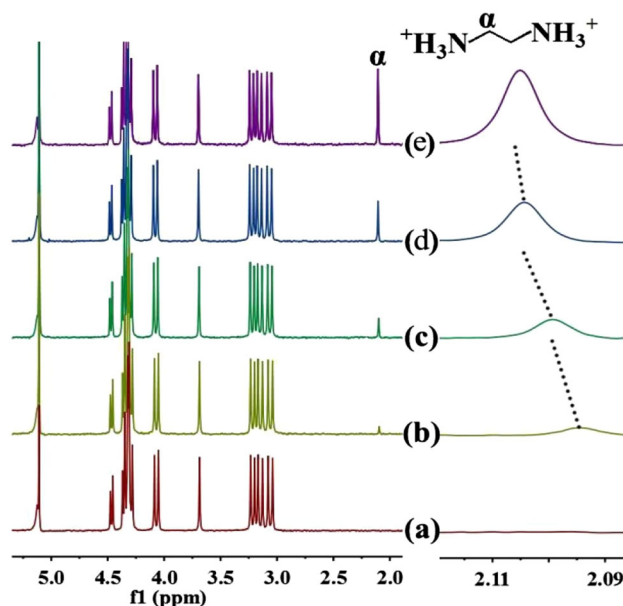


Fig. 2 Titration ^1H NMR spectra (400 MHz, 0.5 mL 4 M DCI/ D_2O) of 4 mg *i*Q[6] in the absence (a) and presence of (b) 0.04, (c) 0.11, (d) 0.25, (e) 1.0 equiv. of guest **1**

Figure 3 shows titration ^1H NMR spectra obtained using a fixed equivalent of *i*Q[6] and various equivalents of **3** in 4 M DCI/ D_2O . Unlike the aforementioned systems, two sets of proton signals from **3** were seen to represent the binding and unbinding. Meanwhile, two sets of proton signals from *i*Q[6] were observed to represent binding and unbinding in the corresponding ^1H NMR spectra, even when the host (Fig. 3b) or guest (Fig. 3d) was present in excess. This result suggests that the inclusion interaction of *i*Q[6] was not strong enough to completely bind guest **3**, and the resultant *i*Q[6]-**3** inclusion complex was of a lower

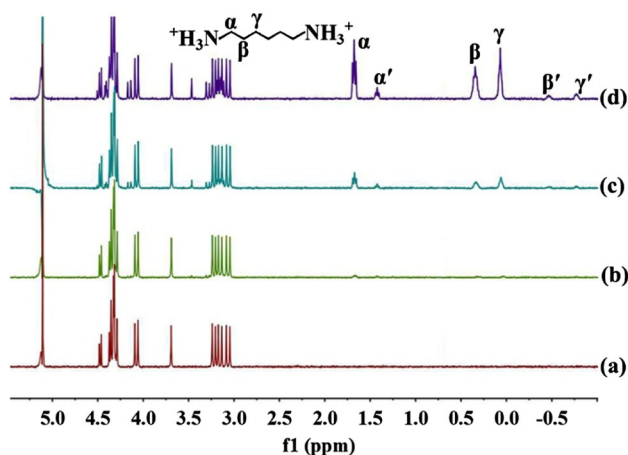


Fig. 3 Titration ^1H NMR spectra (400 MHz, 0.5 mL 4 mol/L DCI/ D_2O) of 4 mg *i*Q[6], in the absence of (a) and presence of (b) 0.10, (c) 0.56, (d) 1.96 equiv of **3**

stability. Three proton resonances corresponding to the binding of **3** were apparent, as were another three proton resonances corresponding to unbinding, and those corresponding to binding experienced an upfield shift of 0.25 ppm ($H\alpha$), 0.81 ppm ($H\beta$) and 0.84 ppm ($H\gamma$), respectively. Proton resonances—corresponding to binding and unbinding of the *i*Q[6] host—were also observed, including the inverted methine proton resonance that displayed a remarkable upfield shift of 0.22 ppm, whereas the other proton resonances experienced a downfield shift by 0.10 ppm. Although the *i*Q[6]-**3** interaction system is unusual in displaying resonances for both binding and unbinding of host and guest, indicating an unstable host-guest interaction, this provides a convenient way to estimate the association constant between *i*Q[6] and **3**, and a K_a was (83 L mol⁻¹ based on data in Fig. 3d. Similar titration ¹H NMR spectra were obtained for the *i*Q[6]-**4** interaction system (Fig. S4, Supporting Information), with both binding/unbinding resonances visible for both hosts and guests. For this system, the balance of binding and unbinding between host and guest appeared to favor unbinding, and the K_a of the *i*Q[6]-**4** complex (41 L mol⁻¹ based on data from Fig. S4e, Supporting Information) is smaller than that of the *i*Q[6]-**3** complex.

Despite significant effort, diffraction-quality crystals for studying the interaction of *i*Q[6] with α,ω -alkyldiammonium guests in acidic or salt solutions (described above) could not be obtained. Generally, α,ω -alkyldiammonium dichloride guests are considered to behave as typical salts in aqueous solution, and etheylenediammonium is considered a typical dictation. [CdCl₄]²⁻ or [ZnCl₄]²⁻ anions are known to function as effective structure directing agents in the construction of Q[*n*]-metal coordination polymers [16, 17], and introduction of these into *i*Q[6]- α,ω -alkyldiammonium guest systems resulted in suitable crystals for *i*Q[6][ZnCl₄] \cdot 0.5 \cdot **1** \cdot H₂O \cdot 17H₂O (**A**) and {**3**@*i*Q[6]}.[ZnCl₄]²⁻.[Zn(H₂O)Cl₃]⁻ \cdot 18H₂O (**B**). Figure 4a shows the overall assembly of *i*Q[6], [ZnCl₄]²⁻ anions and guest **1** observed in the structure of compound **A**. A ‘honeycomb’ structure built from a framework of [ZnCl₄]²⁻ anions can be observed (Fig. 4b), and closer inspection revealed that numerous [ZnCl₄]²⁻ anions (usually six) surround a *i*Q[6] molecule (Fig. 4c). The driving force is likely to be the electropositive outer surface of Q[*n*]s that attracts [ZnCl₄]²⁻ anions through ionic-dipole interactions, and we define this as the outer surface interaction [17, 18]. Similarly, the electronegative surface surrounding Q[*n*] (herein *i*Q[6]) attracts electropositive species such as metal ions and **1** molecules to form portal interaction products. Figure 4d shows an example of a portal interaction complex in which the protonated amine group (N25) interacts with portal carbonyl oxygens (O2 and O3) through hydrogen bonding and ionic-dipole interactions. The distances of N25-O2 and N25-O3 are 2.8 and 2.9 Å, respectively, and each **1** guest interacts with two *i*Q[6]

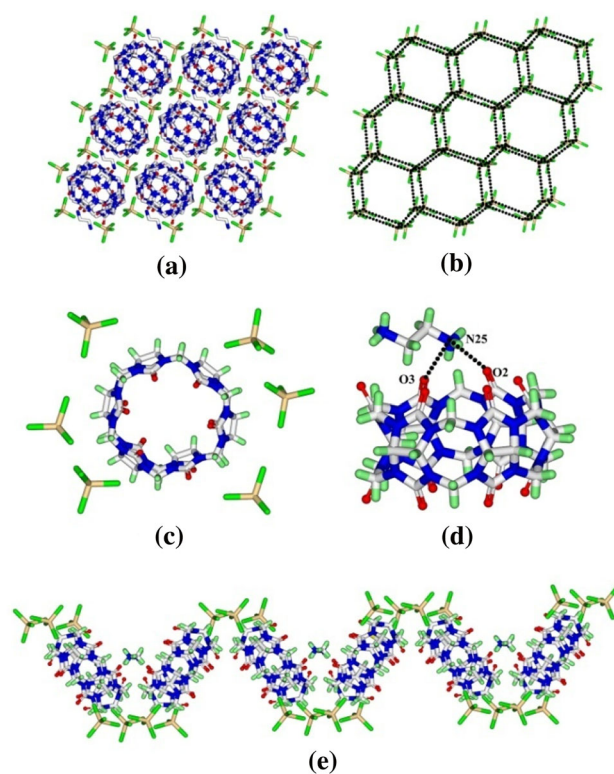


Fig. 4 Crystal structure of compound **A**: **a** overall view of the supramolecular assembly formed from *i*Q[6] molecules, guest **1** and [ZnCl₄]²⁻ anions; **b** [ZnCl₄]²⁻-based honeycomb-like framework; **c** interaction between *i*Q[6] and [ZnCl₄]²⁻ anions; **d** interaction between *i*Q[6] and guest **1**; **e** the *i*Q[6]-**1**-[ZnCl₄]²⁻ ternary supramolecular chain

molecules to form a ‘V’ type pair, with pairs linked by [ZnCl₄]²⁻ anions through the so-called outer surface interaction of Q[*n*]s. Thus, an *i*Q[6]-**1**-[ZnCl₄]²⁻ ternary supramolecular chain is formed (Fig. 4e).

A similar supramolecular assembly was observed in the crystal structure of compound **B** (Fig. 5a) in which [ZnCl₄]²⁻ and [Zn(H₂O)Cl₃]⁻ anions form a honeycomb-like framework (Fig. 5b), with *i*Q[6] molecules occupying the cavities of the framework. Each *i*Q[6] molecule is surrounded by three [ZnCl₄]²⁻ and four [Zn(H₂O)Cl₃]⁻ anions associated through outer surface interactions, water molecule (O1 W) is bound through a hydrogen bonding interaction, and [Zn(H₂O)Cl₃]⁻ anions are coordinated through the portal carbonyl oxygen (O4) of *i*Q[6] (red dashed line, 2.818 Å). There is an additional interaction between portal carbonyl carbon C19 and the chloride (Cl1) from the [Zn(H₂O)Cl₃]⁻ anion (blue dashed line, 3.448 Å in Fig. 5c). Unlike the interaction of *i*Q[6] with guest **1**, interaction with guest **3** results in a pseudorotaxane conformation (Fig. 5d). As with most threaded linear alkyldiammonium guests, the two protonated amine groups (N25 and N26) of guest **3** interact with two opening portal carbonyl oxygens (O5, O6, and O8, O9) of the *i*Q[6] host

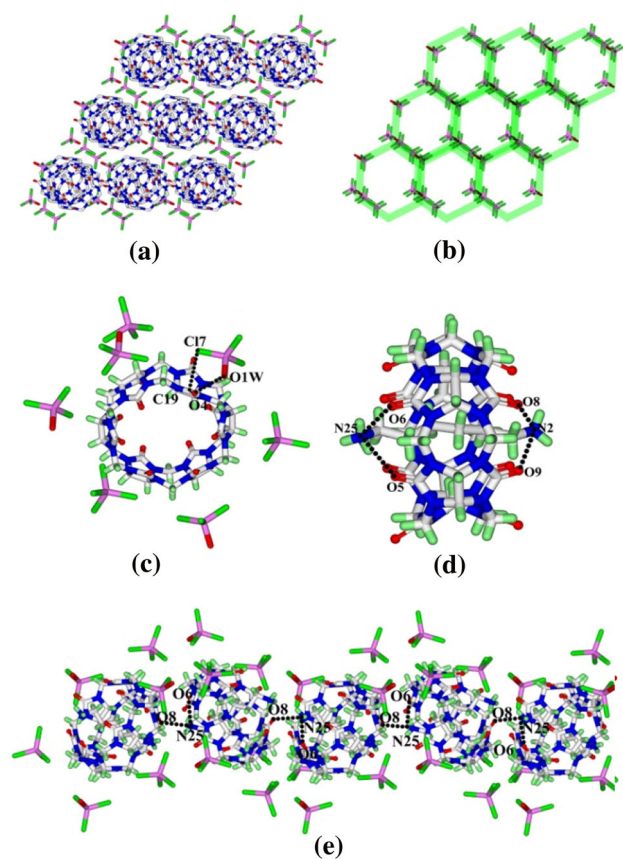


Fig. 5 Crystal structure of compound **B**: **a** overall view of the supramolecular assembly formed from *i*Q[6], guest **3**, $[\text{ZnCl}_4]^{2-}$ and $[\text{Zn}(\text{H}_2\text{O})\text{Cl}_3]^-$ anions; **b** honeycomb-like framework constructed of $[\text{ZnCl}_4]^{2-}$ and $[\text{Zn}(\text{H}_2\text{O})\text{Cl}_3]^-$ anions; **c** interaction of *i*Q[6] with $[\text{ZnCl}_4]^{2-}$ and $[\text{Zn}(\text{H}_2\text{O})\text{Cl}_3]^-$ anions; **d** interaction between *i*Q[6] and guest **3**; **e** supramolecular chain constructed of *i*Q[6], guest **3**, $[\text{ZnCl}_4]^{2-}$ and $[\text{Zn}(\text{H}_2\text{O})\text{Cl}_3]^-$ anions

through hydrogen bonding and ionic-dipole interactions, with $N_{\text{amine}}\text{-}O_{\text{carbonyl}}$ distances in the range of 2.978–3.322 Å. Moreover, the protonated amine groups (N25) of the included guest **3** interact with carbonyl oxygens (O8) of neighbouring *i*Q[6] molecules through hydrogen bonding and ionic-dipole interactions, with N25–O8 distances of 3.051 Å. Thus, the combination of these *i*Q[6]-**3**-based pseudorotaxane and the outer surface interactions between *i*Q[6] with $[\text{ZnCl}_4]^{2-}$ and $[\text{Zn}(\text{H}_2\text{O})\text{Cl}_3]^-$ anions results in the formation of an *i*Q[6]-**3** pseudorotaxane-based ‘pupae’ within the cells of the $[\text{ZnCl}_4]^{2-}$ and $[\text{Zn}(\text{H}_2\text{O})\text{Cl}_3]^-$ -based honeycomb (Fig.s 5a and 5e).

Conclusion

We present the first systematic investigation of the interaction of the *i*Q[6] host with α,ω -alkyldiammonium guests (**1–6**) in acidic (4 M HCl) aqueous solution using ^1H NMR spectroscopy. The results revealed that only *i*Q[6]-**3** and

*i*Q[6]-**4** interaction systems exhibit a lower exchange ratio for binding and unbinding on the NMR time scale. Moreover, both binding and unbinding of host and guest were discernible in the titration ^1H NMR spectra, indicating an unstable host–guest interaction, and this provided a convenient way to estimate the association constant between *i*Q[6] and **3** or **4**. The other guests showed no obvious interaction with *i*Q[6]. X-ray diffraction analysis revealed that guest **1** interacted with *i*Q[6] through a portal interaction, whereas guest **3** interacted with *i*Q[6] through a cavity interaction. Interestingly, titration ^1H NMR spectra displayed a slight upfield shift of proton signals corresponding to guest **1**, indicating that of the two different interactions present in this system, the inclusion interaction dominated. These results also suggest that different interaction types may be present in other *i*Q[6]-guest systems.

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