

Chapter 15

[3]Pseudorotaxanes Based on the Cryptand/Monopyridinium Recognition Motif

15.1. INTRODUCTION

The design and preparation of interlocked threaded structures, such as rotaxanes, catenanes, and polyrotaxanes, is a hot topic in chemistry due to not only their topological importance but also their potential applications.¹ The study of pseudorotaxanes, prepared by threading a linear species through the cavity of a cyclic species, is important because they are commonly used as intermediates in the fabrication of these interlocked structures.^{1a} Recently we reported some [2]pseudorotaxanes based on the new cryptand/monopyridinium salt recognition motif.² Herein, we report two [3]pseudorotaxanes which arise from self-assembly of a cryptand and bispyridinium salts.

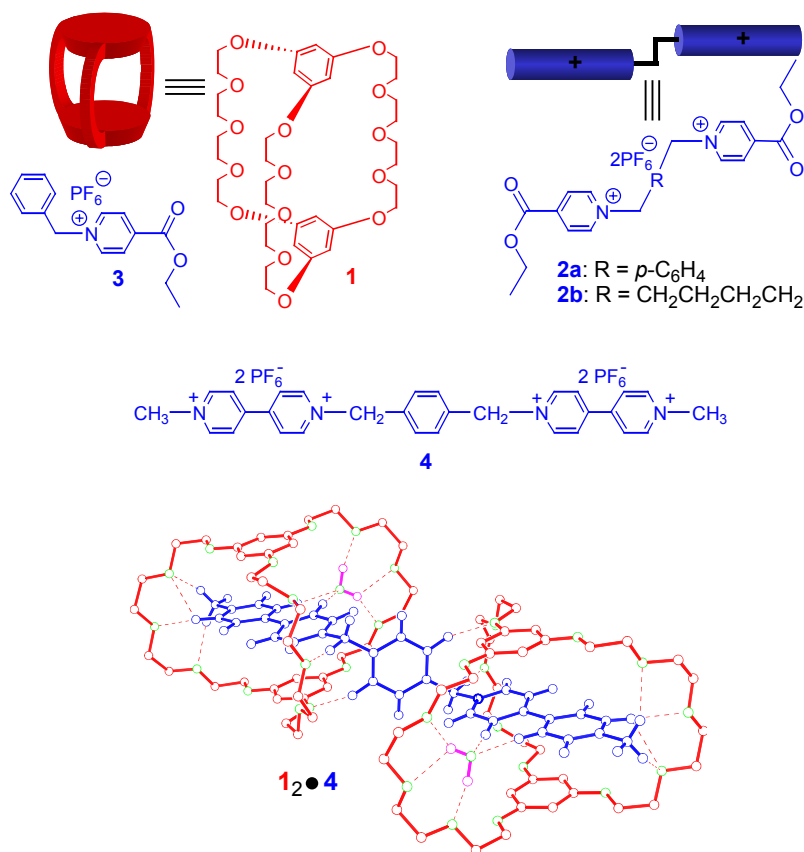


Figure 1. Compounds used in this study and the X-ray structure³ of **1**₂•**4** with PF₆ counterions removed.

15.2. RESULTS AND DISCUSSION

The yellow color of solutions of cryptand **1**⁴ and bispyridinium salts **2** is due to the charge transfer between the electron-rich phenylene rings of **1** and the electron-poor pyridinium rings of **2**. Partial proton NMR spectra of **1**, **2a**, and a mixture of **1** and **2a** are shown in Figure 2. Only one set of peaks was found in the proton NMR spectrum of the solution of **1** and **2a**, indicating fast-exchange complexation. After complexation, peaks corresponding to β-pyridinium protons H₆ on **2a** and aromatic protons H₁ of **1** moved upfield significantly. Furthermore, α-pyridinium protons H₇, phenylene protons H₉, and benzylic protons H₈, and ethyl ester methylene protons H₅ on **2a** and α- and β-ethyleneoxy protons (H₂ and H₄) of **1** also moved upfield, while γ-ethyleneoxy protons

H₃ of 1 moved downfield. The stoichiometries of the complexes between cryptand 1 and bispyridinium salt 2 were determined to be 2:1 in solution by a mole ratio plot⁵ using proton NMR data (Figure 3).

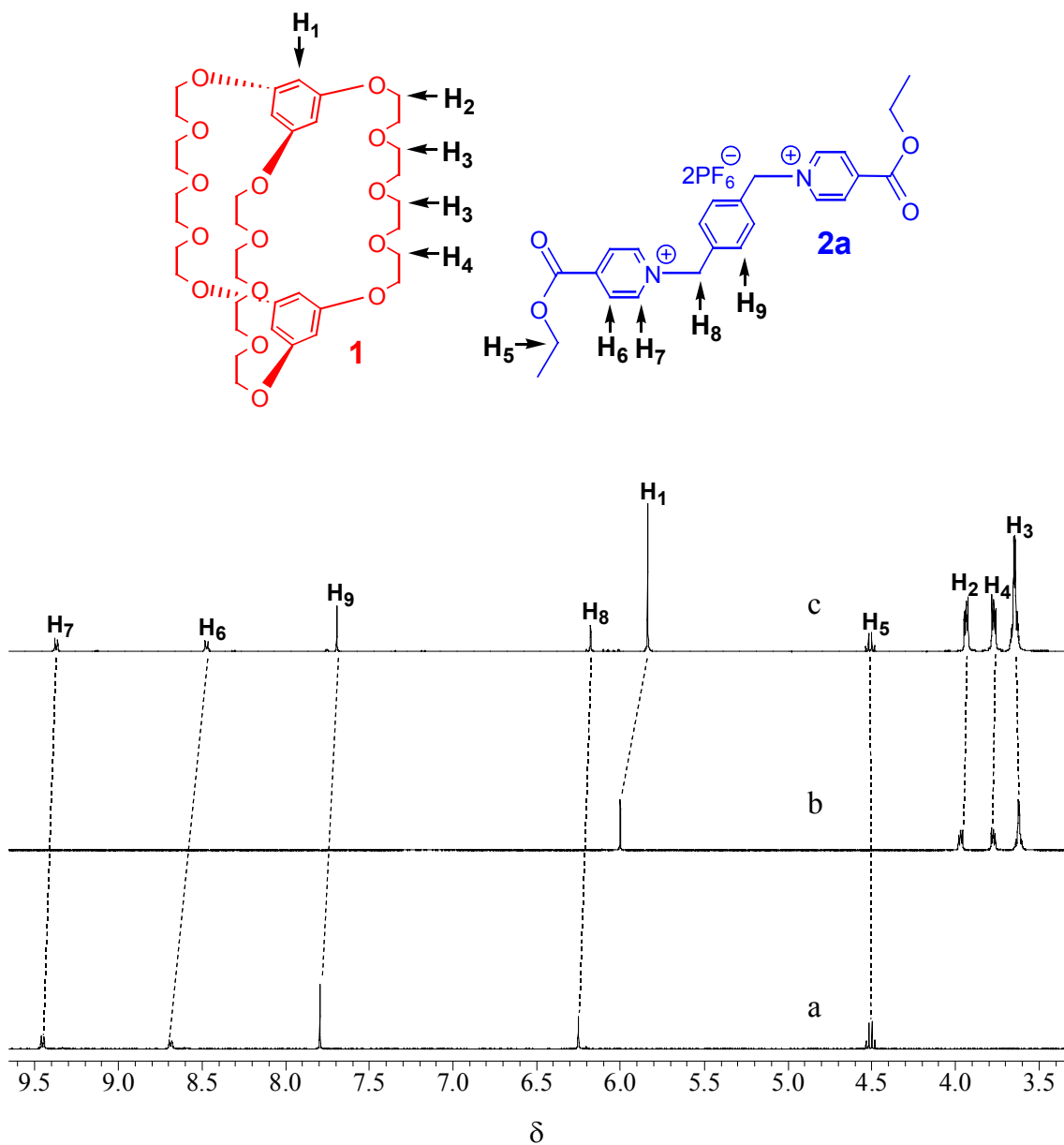


Figure 2. Partial proton NMR spectra (400 MHz, acetone-d₆, 22 °C) of bispyridinium salt 2a (a, bottom), cryptand 1 (b, middle), and 6.00 mM 1 and 3.00 mM 2a (c, top).

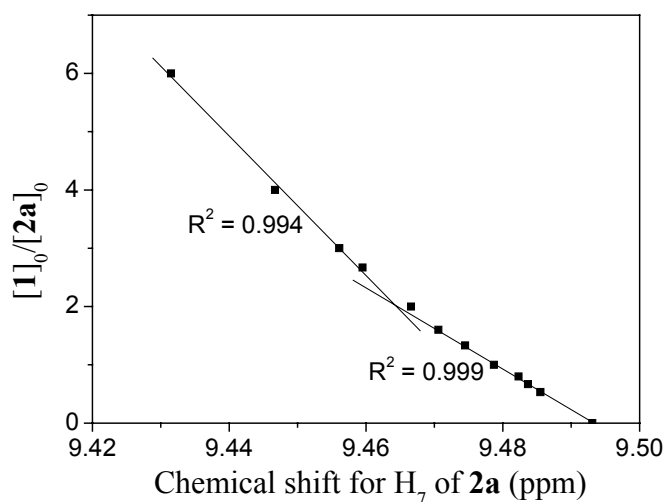


Figure 3. Mole ratio plot for **1** and **2a**, indicating 2:1 stoichiometry. The solvent is acetone-*d*₆. [2a]₀ = 0.250 mM.

Electrospray ionization mass spectra of solutions of **1** and **2** in 4:1 acetonitrile:chloroform confirmed the 2:1 stoichiometry of the complexes. For the mass spectrum of a solution of **1** and **2a**, the base peak was at *m/z* 566.6, corresponding to [1•2a – 2PF₆]²⁺. One more peak was found for 1•2a: *m/z* 1277.6 (13%) [1•2a – PF₆]⁺. Three peaks were found for 1₂•2a: *m/z* 929.9 (19%) [1₂•2a – 2PF₆]²⁺, 915.7 (12%) [1₂•2a – 2PF₆ – C₂H₄]²⁺, and 611.6 (6%) [1₂•2a – 2PF₆ – C₂H₅OH + Na]³⁺. For the mass spectrum of a solution of **1** and **2b**, the base peak was at *m/z* 556.6, corresponding to [1•2b – 2PF₆]²⁺. One more peak was found for 1•2b: *m/z* 1257.6 (15%) [1•2b – PF₆]⁺. Two peaks were observed for 1₂•2b: *m/z* 919.8 (21%) [1₂•2b – 2PF₆]²⁺ and 601.5 (7%) [1₂•2a – 2PF₆ – COOC₂H₅ + K]³⁺.

In order to study the relationship between the two monopyridinium binding sites of **2a** during its complexation with **1**, proton NMR characterizations were done on a series of acetone solutions for which the initial concentration of guest **2a** was kept constant at 0.250 mM while the initial concentration of host **1** was systematically varied. Based on these proton NMR data, the extent of complexation, *p*, of the monopyridinium units was determined⁶ and a Scatchard plot⁷ was made (Figure 4). The linear feature of this plot demonstrated that the two pyridinium binding sites are independent of each

other during the complexation;⁸ that is, the complexation between **1** and **2a** is statistical. From the intercept and the slope of the plot the average association constant⁹ (K_{av}) is $2.8 (\pm 0.3) \times 10^2 \text{ M}^{-1}$ for the complexation between **1** and **2a**. This average association constant is a little higher than the association constant, 182 M^{-1} for **1**•**3** in acetone.²

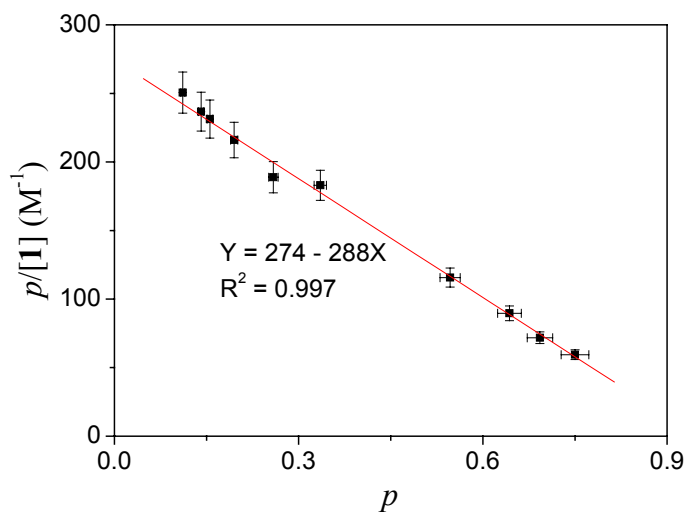


Figure 4. Scatchard plot for the complexation of cryptand host **1** with bispyridinium guest **2a** in acetone- d_6 at 22 °C. p = fraction of monopyridinium units bound. Error bars in p : ± 0.03 absolute; error bars in $p/[1]$: ± 0.06 relative.

Ultimate proof of the formation of [3]pseudorotaxanes **1**₂•**2** based on the cryptand/monopyridinium recognition motif is from X-ray analysis (Figures 5 and 6). Φ X-ray quality yellow single crystals of **1**₂•**2a** were grown by vapor diffusion of pentane into an acetonitrile solution of **2a** with excess **1**. As in the 1:1 complex between cryptand **1** and **3**,² **1**₂•**2a** is stabilized by hydrogen bonding and face-to-face π -stacking interactions (Figure 5) and the ethyl ester methylene protons are involved in hydrogen bonding to ethyleneoxy oxygen atoms of cryptand **1** (**A** and **B** of Figure 5). An α -pyridinium proton of each monopyridinium unit is connected to an ethyleneoxy chain by a water hydrogen bonding bridge (**C**, **D**, and **E** of Figure 5). The left α -pyridinium proton of each

monopyridinium unit is directly hydrogen bonded to an ether oxygen (F of Figure 5). Also as in **1•3**² the distance between each pyridinium ring of **2a** and the phenylene rings of the cryptand host are about equal to each other, presumably in order to maximize face-to-face π -stacking between the electron-rich cryptand host and the electron-poor monopyridinium moieties. However, there is still an obvious difference; that is, a benzylic proton is hydrogen-bonded to an ethyleneoxy oxygen of the cryptand host **1** in **1•3**,² while none of xylyl methylene protons are involved in hydrogen bonding in **1₂•2a**. The centroid-centroid distance between the phenylene rings of the cryptand host in **1₂•2a** is 6.58 Å, while this distance is 6.88 Å in **1•3**,² indicating stronger charge transfer interactions between cryptand hosts and monopyridinium binding sites in **1₂•2a** due to the increase of the positive charge density on the pyridinium rings of bispyridinium salt **2a** compared with that of monopyridinium salt **3**.¹² This result is in agreement with the higher average association constant for the complexation between cryptand **1** and bispyridinium salt **2a** than between cryptand **1** and monopyridinium salt **3**.

The ¹H NMR chemical shift changes of host **1** upon complexation are consistent with a structure in solution similar to that of **1₂•2a** in the solid state (Figure 5). Protons H₁, H₂ and H₄ are in shielding regions of the aromatic guest, while H₃ resides in the deshielding environment of the central phenylene ring of the bispyridinium guest. The upfield shift of the ethyl ester methylene (H₅) and phenylene protons (H₉) of the guest **2a** is consistent with their positions in the shielding region of the aromatic moieties of the cryptand host **1**.

Previously we reported a [3]pseudorotaxane **1₂•4** from the cooperative complexation between cryptand host **1** and bisparaquat guest **4**.³ Based on X-ray structure of **1₂•4** (Figure 1), we claimed that one possible reason for this cooperative complexation was that the formation of the 1:1 complex effectively restricts rotation about the N-CH₂-C₆H₄ bonds of bisparaquat **4** because of the hydrogen bonding of both the xylyl methylene and ortho aromatic protons of **4** to cryptand host **1** (see Figure 1) and this conformational restriction facilitates complexation of the second paraquat site.³ Actually the observations here support this claim. From proton NMR characterization, we know that the complexation between **1** and **2a** is statistical. In the X-ray structure of

1₂•2a, none of xylyl methylene or central aromatic protons of **2a** are involved in hydrogen bonding to cryptand **1** (Figure 5).

Another difference between cryptand/bisparaquat [3]pseudorotaxane **1₂•4** and cryptand/bispyridinium [3]pseudorotaxane **1₂•2a** is that water molecules are hydrogen bonding bridges between β -pyridinium protons of the guest and cryptand hosts in **1₂•4**, while they are hydrogen bonding bridges between α -pyridinium protons and cryptand hosts in **1₂•2a**.

Further observation revealed that the guest **2a** is ion-paired¹⁴ in the X-ray structure of [3]pseudorotaxane **1₂•2a**; both PF₆ counterions are hydrogen bonded to β -pyridinium protons (see Electronic Supplementary Information for more details).

X-ray quality yellow single crystals of **1₂•2b** were grown by vapor diffusion of pentane into an acetonitrile solution of **2b** with excess **1**. The crystal structure of **1₂•2b** (Figure 6) is very similar to that of **1₂•2a** in terms of interactions, including hydrogen bonding and face-to-face π -stacking between the bispyridinium guest and the cryptand hosts. The guest **2b** is also ion-paired in the X-ray structure of [3]pseudorotaxane **1₂•2b**; both PF₆ counterions are hydrogen bonded to ester methyl protons (see Electronic Supplementary Information for more details).

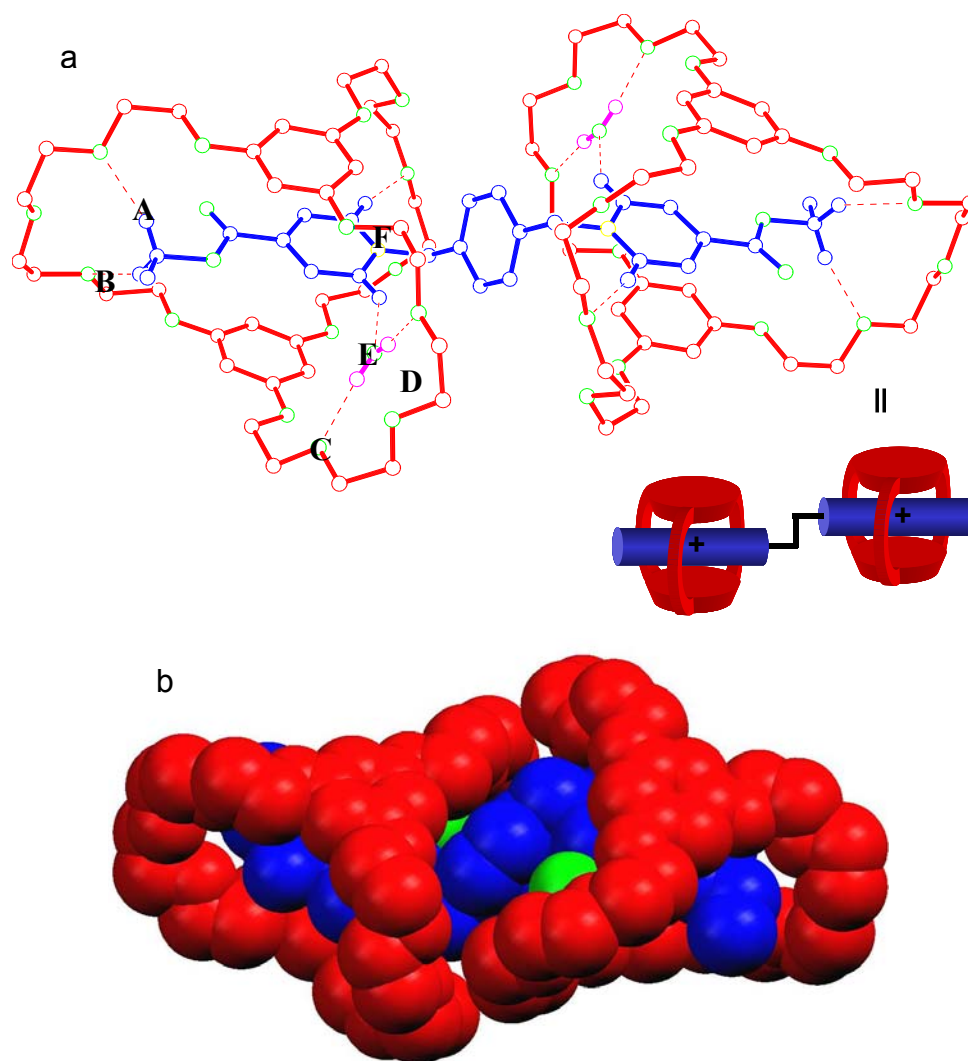


Figure 5. Ball-stick (a, top) and space-filling (b, bottom) views of the X-ray structure of $1_2 \cdot 2a$. (a) **1** molecules are red, **2a** is blue, water molecules are magenta, oxygens are green, and nitrogens are yellow. Two PF_6 counterions, other solvent molecules, and the protons except the ones involved in hydrogen bonding were omitted for clarity. Hydrogen-bond parameters: C(O) \cdots O distances (\AA), H \cdots O distances (\AA), C(O)-H \cdots O angles (deg) **A**, 3.42, 2.53, 150; **B**, 3.21, 2.26, 161; **C**, 2.99, 2.15, 171; **D**, 2.92, 2.08, 173; **E**, 3.06, 2.28, 140, **F**, 3.41, 2.47, 170. Face-to-face π -stacking parameters: centroid-centroid distances (\AA) 3.74, 3.66; ring plane/ring plane inclinations (deg): 9.1, 4.8. (b) All protons, two PF_6 counterions, and other solvent molecules were omitted. **1** molecules are red, **2a** is blue, and the water molecules are green.

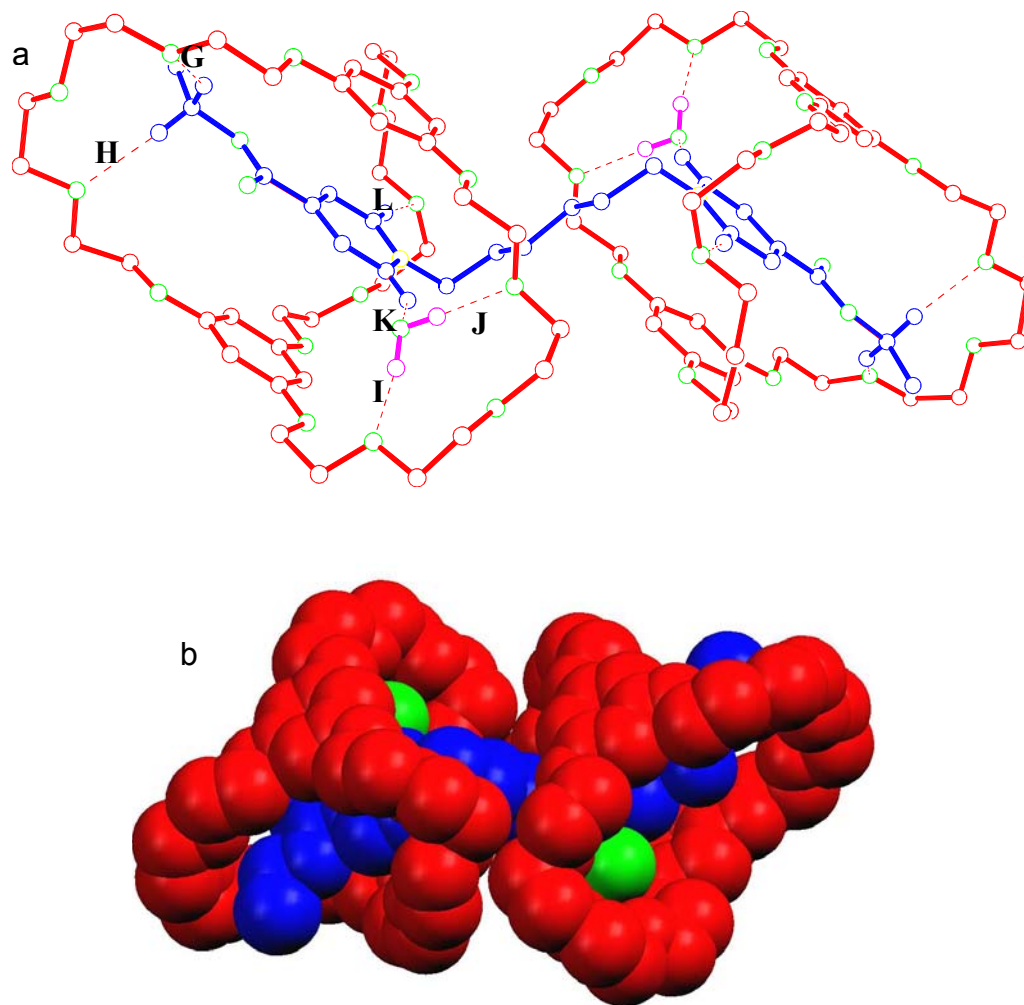


Figure 6. Ball-stick (a, top) and space-filling (b, bottom) views of the X-ray structure of $1_2 \cdot 2b$. (a) **1** molecules are red, **2b** is blue, water molecules are magenta, oxygens are green, and nitrogens are yellow. Two PF_6 counterions, other solvent molecules, and the protons except the ones involved in hydrogen bonding were omitted for clarity. Hydrogen-bond parameters: C(O) \cdots O distances (\AA), H \cdots O distances (\AA), C(O)-H \cdots O angles (deg) **G**, 3.31, 2.45, 144; **H**, 3.34, 2.35, 171; **I**, 2.93, 1.97, 168; **J**, 2.85, 1.92, 167; **K**, 3.04, 2.32, 129, **L**, 3.25, 2.26, 173. Face-to-face π -stacking parameters: centroid-centroid distances (\AA) 3.80, 3.74; ring plane/ring plane inclinations (deg): 6.4, 5.4. (b) All protons, the PF_6 counterions, and other solvent molecules were omitted. **1** molecules are red, **2b** is blue, and the water molecules are green.

15.3. CONCLUSIONS

In summary, [3]pseudorotaxanes were successfully prepared by self-assembly between a cryptand host and bispyridinium guests based on the cryptand/monopyridinium salt recognition motif as confirmed by proton NMR characterization, electrospray ionization mass spectrometry, and X-ray analysis. Once appropriate stoppers are introduced at the ends of the bispyridinium guests, they can be converted into mechanically interlocked [3]rotaxanes. We are working on this project now.

15.4. ACKNOWLEDGEMENTS

This work was supported by the National Science Foundation (DMR0097126) and the Petroleum Research Fund (40223-AC7). The purchase of the diffractometer Xcalibur2 (VPI&SU) was also supported by the National Science Foundation (CHE-131128).

15.5. SUPPORTING INFORMATION

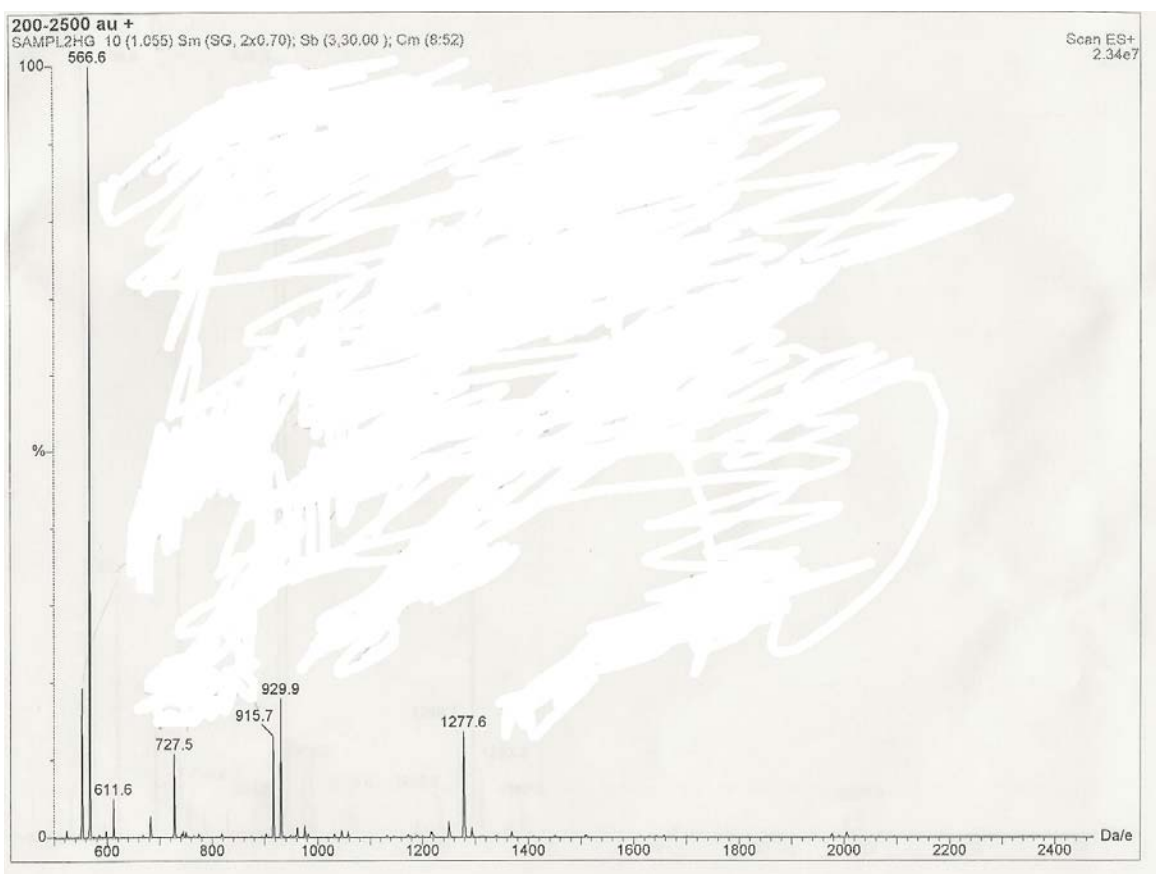
Syntheses of bispyridinium salts 2

2a: To a 50 mL three-necked round bottom flask equipped with a magnetic stirrer and a condenser were added 1.32 g (5.00 mmol) 1,4-di(bromomethyl)benzene and 10 mL acetonitrile. To this solution was added a solution of ethyl isonicotinate (15.1 g, 100 mmol) in 10 mL acetonitrile and the mixture was stirred at reflux for 48 hours. The reaction mixture was cooled to room temperature and the precipitate was filtered. The solid was boiled in CHCl₃ and filtered. This solid was dissolved in a minimum volume of deionized water. To this solution was added NH₄PF₆ until no further precipitation was observed. The precipitate was filtered and recrystallized from deionized water three times to afford **2a** as white crystals, 3.06 g (88%), m.p. 236.8-238.1 °C. ¹H NMR (400 MHz, CD₃COCD₃, 22 °C): δ = 1.44 (t, *J* = 7.2 Hz, 6H), 4.54 (q, *J* = 7.2 Hz, 4H), 6.29 (s, 4H),

8.73 (d, $J = 6.8$ Hz, 4H), 9.50 (d, $J = 6.8$ Hz, 4H); elemental analysis calcd for $C_{24}H_{26}O_4N_2P_2F_{12}$: C, 41.37; H, 3.76; N, 4.02; Found: C, 41.51; H, 3.74; N, 4.13.

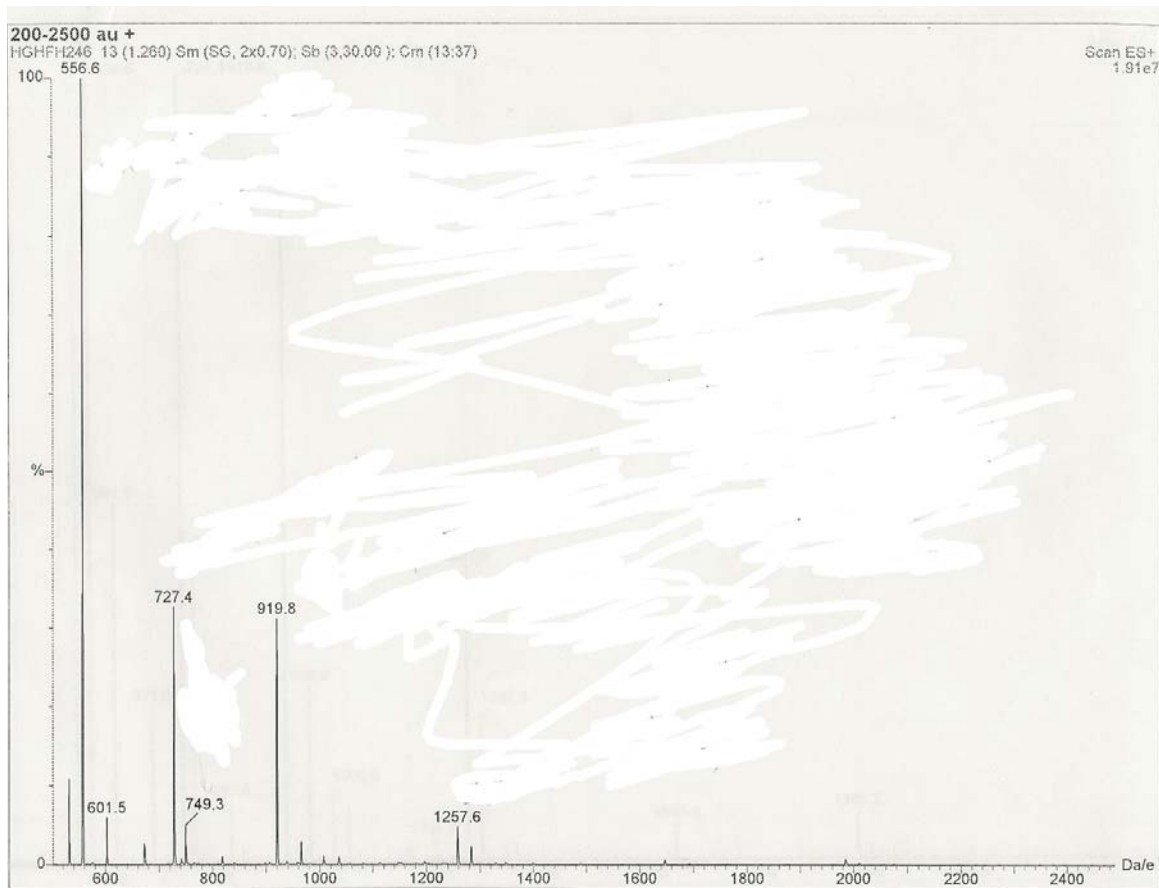
2b: 2b, prepared in a similar way as **2a**, was obtained also as white crystals (85%), m.p. 179.8-180.8 °C. 1H NMR (400 MHz, CD_3COCD_3 , 22 °C): $\delta = 1.45$ (t, $J = 7.2$ Hz, 6H), 1.66 (m, 4H), 2.29 (m, 4H), 4.55 (q, $J = 7.2$ Hz, 4H), 5.03 (t, $J = 7.7$ Hz, 4H), 8.71 (d, $J = 6.8$ Hz, 4H), 9.43 (d, $J = 6.8$ Hz, 4H); LRFABMS (matrix, NBA) m/z 531.7 $[M - PF_6]^+$, 386.6 $[M - 2PF_6]^+$; HRFABMS (matrix, NBA/PEG) m/z calcd for $[M - PF_6]^+$ $C_{22}H_{30}O_4N_2PF_6$, 531.1847, found 531.1846, error 0.2 ppm

Electrospray mass spectrum of a solution of 1 and 2a in a mixture of acetonitrile and chloroform (4:1)



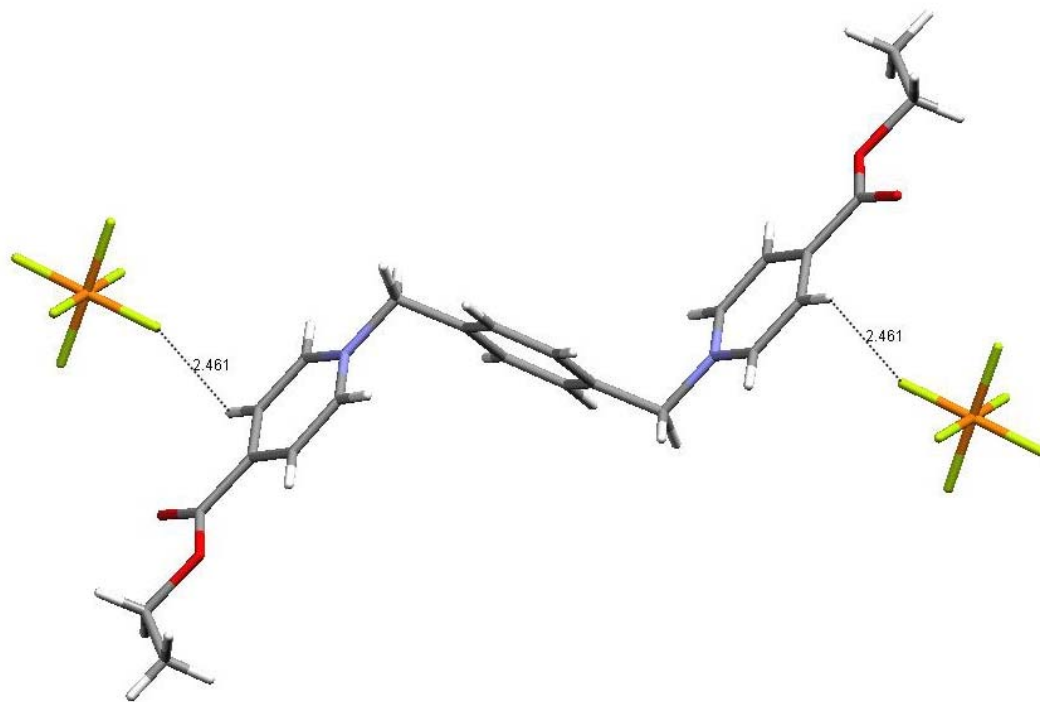
Assignments of main peaks: m/z 1277.6 $[1 \cdot 2a - PF_6]^+$, 929.9 $[1_2 \cdot 2a - 2PF_6]^{2+}$, 915.7 $[1_2 \cdot 2a - 2PF_6 - C_2H_4]^{2+}$, 727.5 $[1 + H]^+$, 611.6 $[1_2 \cdot 2a - 2PF_6 - C_2H_5OH + Na]^{3+}$, and 566.6 $[1 \cdot 2a - 2PF_6]^{2+}$.

Electrospray mass spectrum of a solution of **1** and **2b** in a mixture of acetonitrile and chloroform (4:1)

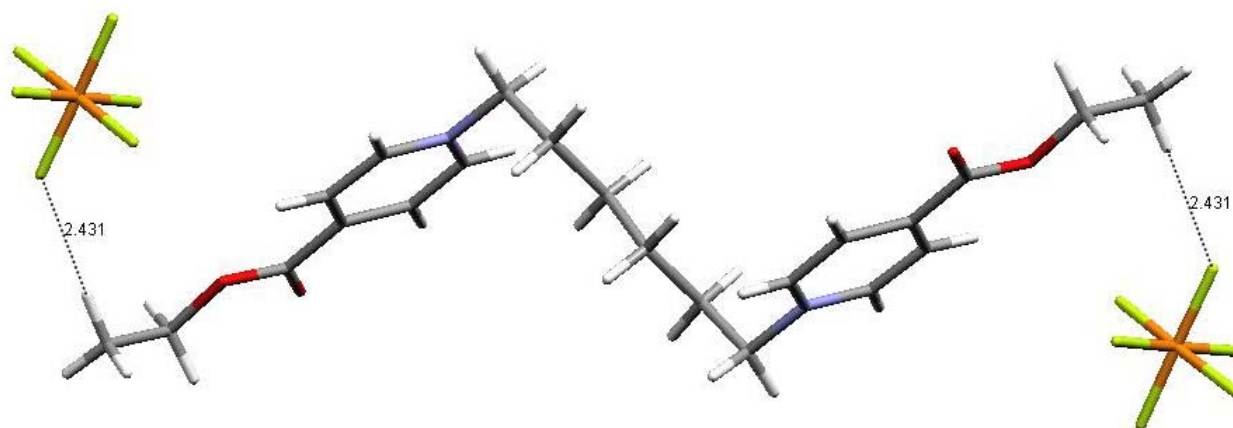


Assignments of main peaks: m/z 1257.6 [**1**•**2b** - PF₆]⁺, 919.8 [**1**₂•**2b** - 2PF₆]²⁺, 749.3 [**1** + Na]⁺, 727.4 [**1** + H]⁺, 601.5 [**1**₂•**2b** - 2PF₆ - COOC₂H₅ + K]³⁺, and 556.6 [**1**•**2b** - 2PF₆]²⁺.

*A view of the guest **2a** in the X-ray structure of [3]pseudorotaxane **1**₂•**2a** showing ion-pairing of the guest*



*A view of the guest **2b** in the X-ray structure of [3]pseudorotaxane **1**₂•**2b** showing ion-pairing of the guest*



REFERENCES AND NOTES

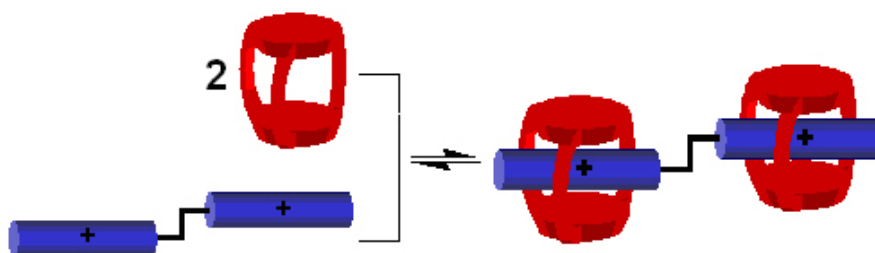
Φ Crystal data of **1₂•2a**: plate, yellow, 0.38 × 0.23 × 0.06 mm, C₁₀₄H₁₅₀O₃₆N₆P₂F₁₂, *FW* 2350.24, Triclinic, space group *P*-1, *a* = 10.5597(13), *b* = 16.0015(16), *c* = 18.397(2) Å; α = 105.583(9)°, β = 105.919(10)°, γ = 91.528(9)°; *V* = 2862.6(6) Å³, *Z* = 1, *D_c* = 1.363 g cm⁻³, *T* = 100 K, μ = 1.40 cm⁻¹, 13273 measured reflections, 10109 independent reflections, 802 parameters, *F*(000) = 1242, *R*_{int} = 0.0250, *R*₁ = 0.0519, *wR*₂ = 0.1247 [*I* > 2σ(*I*)], maximum residual density 1.231 e•Å⁻³, and GooF(*F*²) = 1.014. Crystal data of **1₂•2b**: plate, yellow, 0.33 × 0.20 × 0.09 mm, C₄₇H₇₁O₁₈N₁P₁F₆, *FW* 1083.02, Triclinic, space group *P*-1, *a* = 10.2668(10), *b* = 14.8351(16), *c* = 19.322(2) Å; α = 97.430(9)°, β = 101.347(9)°, γ = 95.682(9)°; *V* = 2837.6(5) Å³, *Z* = 2, *D_c* = 1.268 g cm⁻³, *T* = 100 K, μ = 1.35 cm⁻¹, 13982 measured reflections, 10042 independent reflections, 672 parameters, *F*(000) = 1146, *R*_{int} = 0.0459, *R*₁ = 0.0612, *wR*₂ = 0.1617 [*I* > 2σ(*I*)], maximum residual density 0.0601 e•Å⁻³, and GooF(*F*²) = 1.026. The structure was solved by SHELXS-97¹⁰ and refined by SHELXL-97.¹¹ CCDC 259498 and 259499. See <http://www.rsc.org/suppdata/cc/...> for crystallographic data in .cif or other electronic format.

1. (a) Reviews on threaded structures: Gong, C.; Gibson, H. W. *Curr. Opin. Solid St. Mater. Sci.* **1997**, *2*, 647-652. Gibson, H. W. In *Large Ring Molecules*; Semlyen, J. A. Ed.; John Wiley & Sons: New York, **1996**; Chapter 6, pp 191-262. Raymo, F. M.; Stoddart, J. F. *Chem. Rev.* **1999**, *99*, 1643-1664. *Catenanes, Rotaxanes and Knots*, (Eds.: J.-P. Sauvage, C. O. Dietrich-Buchecker), Wiley-VCH, Weinheim, 1999. Mahan, E.; Gibson, H. W. In *Cyclic Polymers*, 2nd ed.; Semlyen, A. J. Ed.; Kluwer Publishers: Dordrecht, **2000**; pp 415-560. Hubin, T. J.; Busch, D. H. *Coord. Chem. Rev.* **2000**, *200-202*, 5-52. Pease, A. R.; Jeppesen, J. O.; Stoddart, J. F.; Luo, Y.; Collier, C. P.; Heath, J. R. *Acc. Chem. Res.* **2001**, *34*, 433-444. Ballardini, R.; Balzani, V.; Credi, A.; Gandolfi, M. T.; Venturi, M. *Acc. Chem. Res.* **2001**, *34*, 445-455. Harada, A. *Acc. Chem. Res.* **2001**, *34*, 456-464. Panova, I. G.; Topchieva, I. N. *Russian Chem. Rev.* **2001**, *70*, 23-44. Kim, K. *Chem. Soc.*

- Rev.* **2002**, *31*, 96-107. Carlucci, L.; Ciani, G.; Proserpio, D. M. *Coordin. Chem. Rev.* **2003**, *246*, 247-289. (b) Recent publications on threaded structures: Leigh, D. A.; Wong, J. K. Y.; Dehez, F.; Zerbetto, F. *Nature* **2003**, *424*, 174-179. Huang, F.; Zakharov, L. N.; Rheingold, A. L.; Jones, J. W.; Gibson, H. W. *Chem. Commun.* **2003**, 2122-2123. Huang, F.; Gibson, H. W.; Bryant, W. S.; Nagvekar, D. S.; Fronczek, F. R. *J. Am. Chem. Soc.* **2003**, *125*, 9367-9371. Schalley, C. A.; Reckien, W.; Peyerimhoff, S.; Baytekin, B.; Vögtle, F. *Chem. Eur. J.* **2004**, *10*, 4777-4789. Hernández, J. V.; Kay, E. R.; Leigh, D. A. *Science* **2004**, *306*, 1532-1537. Huang, F.; Gibson, H. W. *J. Am. Chem. Soc.* **2004**, *126*, 14738-14739. Huang, F.; Zhou, L.; Jones, J. W.; Gibson, H. W.; Ashraf-Khorassani, M.; *Chem. Commun.* **2004**, 2670-2671. Huang, F.; Gibson, H. W. *Chem. Commun.* **2005**, Advance Article, Feb. 5th.
2. Huang, F.; Slebodnick, C.; Rheingold, A. L.; Ohs, A.; Switek, K. A.; Gibson, H. W. *J. Am. Chem. Soc.* **2005**, Submitted.
 3. Huang, F.; Fronczek, F. R.; Gibson, H. W. *J. Am. Chem. Soc.* **2003**, *125*, 9272-9273.
 4. Bryant, W. S.; Jones, J. W.; Mason, P. E.; Guzei, I. A.; Rheingold, A. L.; Nagvekar, D. S.; Gibson, H. W. *Org. Lett.* **1999**, *1*, 1001-1004.
 5. Tsukube, H.; Furuta, H.; Odani, A.; Takeda, Y.; Kudo, Y.; Inoue, Y.; Liu, Y.; Sakamoto, H.; Kimura, K. In *Comprehensive Supramolecular Chemistry*; Atwood, J. L., Davies, J. E. D., MacNicol, D. D., Vogtle, F., Lehn, J.-M., Eds.; Elsevier: New York, **1996**; Vol. 8, p 425.
 6. Δ_0 , the difference in δ values for H₇ of **2a** in the uncomplexed and fully complexed species, was determined as the y-intercept of a plot of $\Delta = \delta - \delta_0$ vs. $1/[1]_0$ in the high initial concentration range of **1**. Then $p = \Delta/\Delta_0$; Δ = observed chemical shift change relative to uncomplexed species.
 7. Marshall, A. G. *Biophysical Chemistry*; J. Wiley and Sons: New York, **1978**; pp 70-77. Freifelder, D. M. *Physical Biochemistry*; W. H. Freeman and Co.: New York, **1982**; pp 659-660. Connors, K. A. *Binding Constants*; J. Wiley and Sons: New York, **1987**; pp 78-86.

8. Other examples of statistical complexation: Gibson, H. W.; Yamaguchi, N.; Hamilton, L.; Jones, J. W. *J. Am. Chem. Soc.* **2002**, *124*, 4653-4665. Huang, F.; Nagvekar, D. S.; Sleboznick, C.; Gibson, H. W. *J. Am. Chem. Soc.* **2005**, *127*, 484-485. Huang, F.; Zakharov, L. N.; Rheingold, A. L.; Ashraf-Khorassani, M.; Gibson, H. W. *J. Org. Chem.* **2005**, *70*, 809-813.
9. $K_1 = [\mathbf{1} \cdot \mathbf{2a}] / \{[\mathbf{1}][\mathbf{2a}]\}$ and $K_2 = [\mathbf{1}_2 \cdot \mathbf{2a}] / \{[\mathbf{1} \cdot \mathbf{2a}][\mathbf{1}]\}$. $K_{av} = (K_1 + K_2)/2$. The value of K_{av} is equal to the y-intercept and the absolute slope of the best fit line in Figure 4. Since $K_1:K_2 = 4:1$ for statistical systems,⁷ K_1 and K_2 were calculated to be 112 and 448 M⁻¹.
10. Sheldrick, G. M. SHELXS-97, Program for the Solution of Crystal Structures, University of Göttingen, Göttingen (Germany), 1990.
11. Sheldrick, G. M. SHELXL-97, Program for the Refinement of Crystal Structures, University of Göttingen, Göttingen (Germany), 1997.
12. The benzyl group is an electron-releasing group¹³ so it can reduce the positive charge density on the pyridinium rings of monopyridinium salt **3** and bispyridinium salt **2**. However, two pyridinium rings share one xylyl group in bispyridinium salt **2** so this effect is weaker in **2** than **3**. This makes the positive charge density on pyridinium rings of bispyridinium salt **2** higher than that of monopyridinium salt **3**.
13. Hansch, C.; Leo, A.; Taft, R. W. *Chem. Rev.* **1991**, *91*, 165-195.
14. Ion-pairing of the guest leads to the concentration dependence of the apparent association constant if the complex is not ion-paired. See: Jones, J. W.; Gibson, H. W. *J. Am. Chem. Soc.* **2003**, *125*, 7001-7004. Huang, F.; Jones, J. W.; Sleboznick, C.; Gibson, H. W. *J. Am. Chem. Soc.* **2003**, *125*, 14458-14464.

TOC Graphic:



Abstract: [3]Pseudorotaxanes were prepared from a cryptand host and bispyridinium guests based on the cryptand/monopyridinium salt recognition motif as confirmed by proton NMR characterization, electrospray ionization mass spectrometry, and X-ray analysis.
