Supplementary Information

Synthesis and Complexation Study of Calixarene-like Azacyclophanes

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Contents:

- 1. General Methods
- 2. Selected ¹H and ¹³C NMR spectra

1. General methods

All experiments with moisture- or air-sensitive compounds were carried out in anhydrous solvents under Ar or N₂ atmosphere otherwise indicated. Organic solvents were evaporated under reduced pressure using a rotavapor. All synthetic products were dried overnight on vacuum pump, unless otherwise indicated. Flash chromatography was performed on silica gel, particle size 32-63 μ m, pore size 60 Å, Batch number 02826-25. Thin-layer chromatography was performed using precoated F-254-containing silica gel plates (Plastic Backed TLC, Hard Layer, Batch number 79011), layer thickness 200 μ m. All chemical reagents whose syntheses were not described herein were purchased from commercial sources. Solvents were dried and distilled according to standard procedures. Melting points (mp) are uncorrected. Unless otherwise indicated, ¹H spectra were recorded using CDCl₃ at either 500 or 300 MHz using TMS as the internal standard; ¹³C NMR spectra were recorded at 125 MHz. Data are presented as follows: chemical shift, multiplicity (s = singlet, br = broad, d = doublet, t = triplet, m = multiplet, sept = septet), coupling constant (*J*, Hz), integration (# of H), and assignment (when appropriate). Reported multiplicities are apparent. Chemical shifts in the ¹³C NMR are relative to solvent shifts.



































S 18

