
Electronic Supplementary Information for

Chelate Effects in Sulfate Binding by Amide/Urea-Based Ligands

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Contents

1. Synthesis and characterization of ligands 1-6

Synthesis and characterization of **1**

Ligand **1** was synthesized according to a previous literature[S1] and the identity was confirmed by ^{1}H NMR spectra (500 MHz, DMSO-d_6, ppm): δ 9.34 (t, J = 6.0 Hz, 2H, NH), 8.17 (m, 3H, H1 and H2), 3.41 (m, 4H, H3), 1.19 (t, J = 8.0 Hz, 6H, H4).

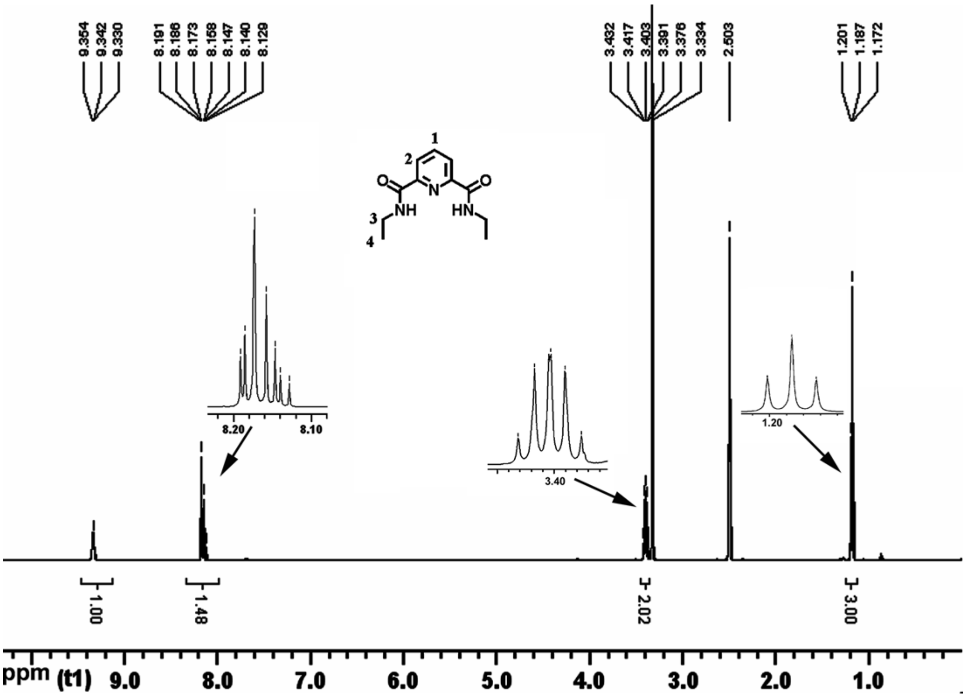
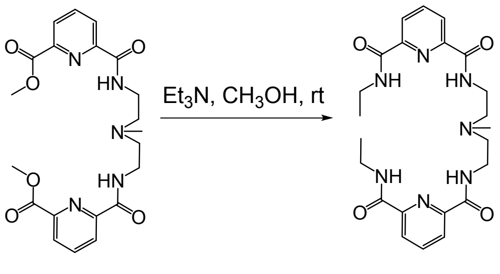


Figure S1. ^{1}H NMR (500 MHz, DMSO-d_6) of **1**.

Synthesis and characterization of **2**



2a **2**

To a 15 mL methanol solution of **2a**[S2] (0.22 g, 0.5 mmol), ethyl amine was bubbled for 3 minutes. After reacting for 24 h, the solvent was removed in vacuum which gave a light brown oil. The oil was redissolved in 25 mL MeCN and the solvent removed in vacuum to yield **2** (0.15 g, 65 %) as a sticky off-white solid. ^{1}H NMR (500 MHz, DMSO-d_6, ppm): δ 9.28 (m, 4H, NHa and NHb), 8.15 (m, 6H, H4, H5 and H6), 3.48 (dd, J = 6.0 Hz, 14 Hz, 4H, H3), 3.39 (m, 4H, H7), 2.64 (t, J = 7.0 Hz, 4H, H2), 2.35 (s, 3H, H1), 1.17 (t, J = 7.5 Hz, 6H, CH$_3$). ^{13}C NMR (125 MHz, DMSO-d_6), δ 163.1 (C=O), 162.7 (C=O), 148.7 (Ar), 148.6 (Ar), 139.3 (Ar), 124.0 (Ar), 56.4 (CH2), 42.2 (CH2), 37.1 (CH2), 33.6 (CH3), 15.7 (CH3). Exact mass for $C_{23}H_{31}N_7O_4 + Cl^-$ 504.2132, found (TOF-MS-ES$^-$) 504.2126.

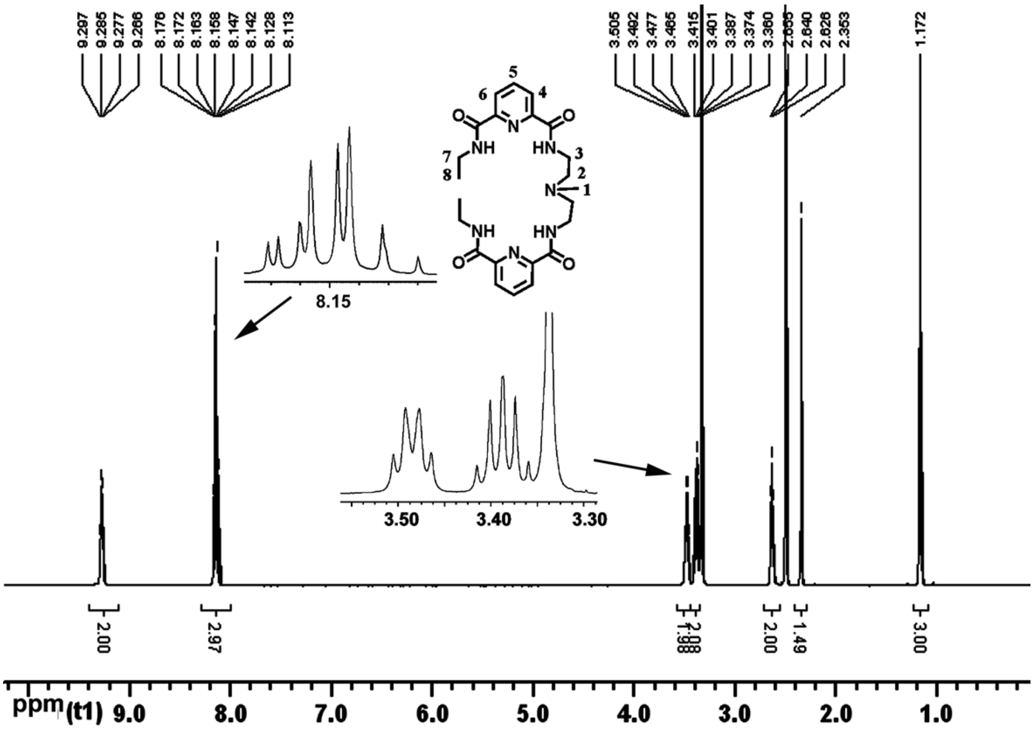


Figure S2. ^{1}H NMR (500 MHz, DMSO-d_6) of **2**.

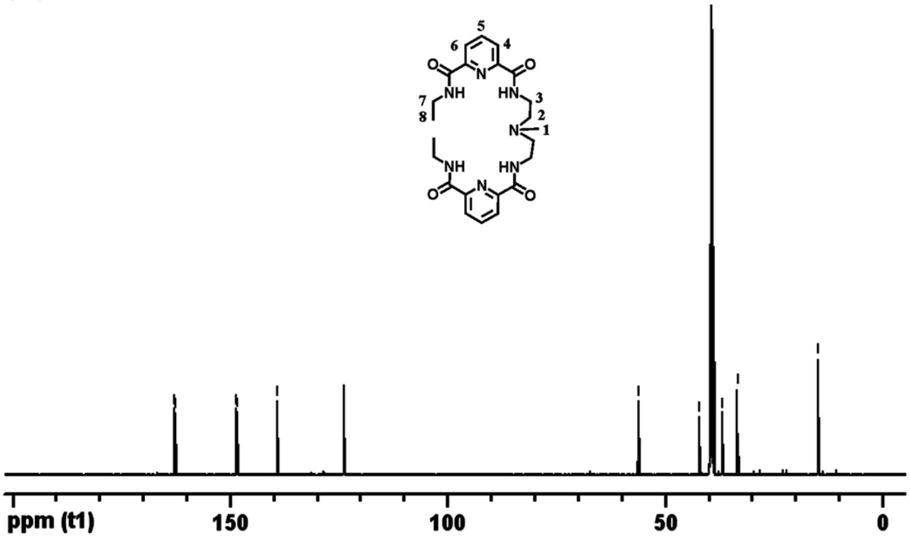


Figure S3. ^{13}C NMR (125 MHz, DMSO-d_6) of **2**.

Synthesis and characterization of **3**

Ligand **3** was synthesized according to a previous literature[S3] and the identity of the compound is confirmed by ^{1}H NMR spectra (400 MHz, DMSO-d_6, ppm): δ 5.72 (brs, 2H, NH), 2.98 (m, 4H, H1), 0.97 (t, J = 7.5 Hz, 6H, H2).

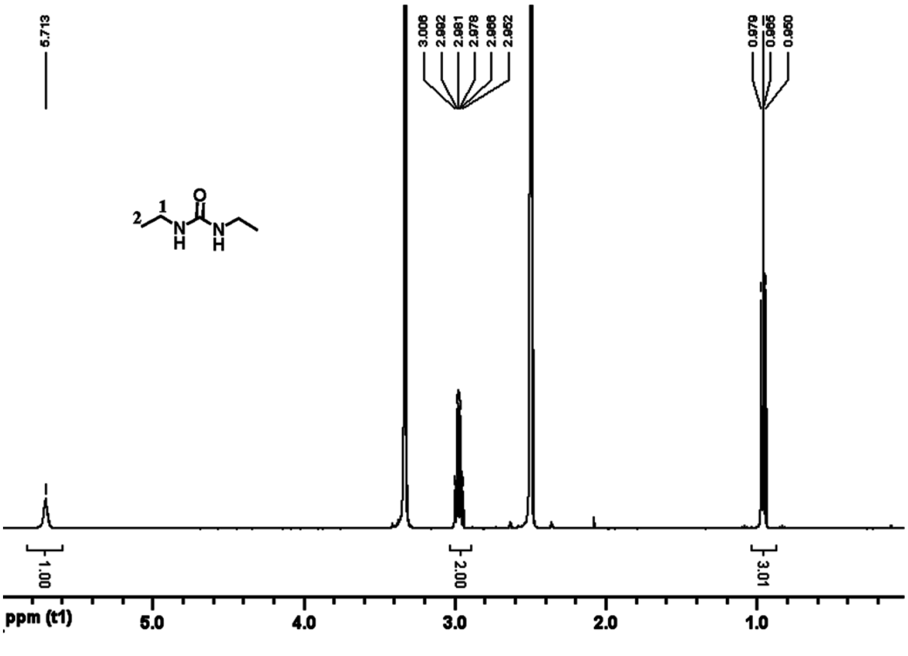


Figure S4. ^{1}H NMR (500 MHz, DMSO-d_6) of **3**.

Synthesis and characterization of **4**

0.23 g (2.0 mmol) 2,2'-diamino-N-methyldiethylamine in 20 mL of CH_2Cl_2 was added dropwise to a stirred $CHCl_2$ solution (30 mL) of ethyl isocyanate (0.57 g, 4.0 mmol). After reacting overnight under room temperature, the solvent was removed by rotary evaporation which gave pure **4** as a white solid (0.39 g, 75 %). ^{1}H NMR (500 MHz, DMSO-d_6, ppm): δ 5.87 (t, J = 5.5 Hz, 2H, NHa), 5.71 (t, J = 5.5 Hz, 2H, urea NHb), 3.04 (m, 4H, H3), 2.98 (m, 4H, H4), 2.33 (t, J = 6.5 Hz, 4H, H2), 2.16 (s, 3H, H1), 0.97 (t, J = 7.0 Hz, 6H, H5). ^{13}C NMR (125 MHz, DMSO-d_6), δ 157.9 (C=O), 57.5 (CH2), 42.0 (CH2), 37.1 (CH2), 34.0 (CH2), 15.7 (CH3). Exact mass for $C_{11}H_{25}N_5O_2 + Cl^-$ 294.1702, found (TOF-MS-ES$^-$) 294.1692.

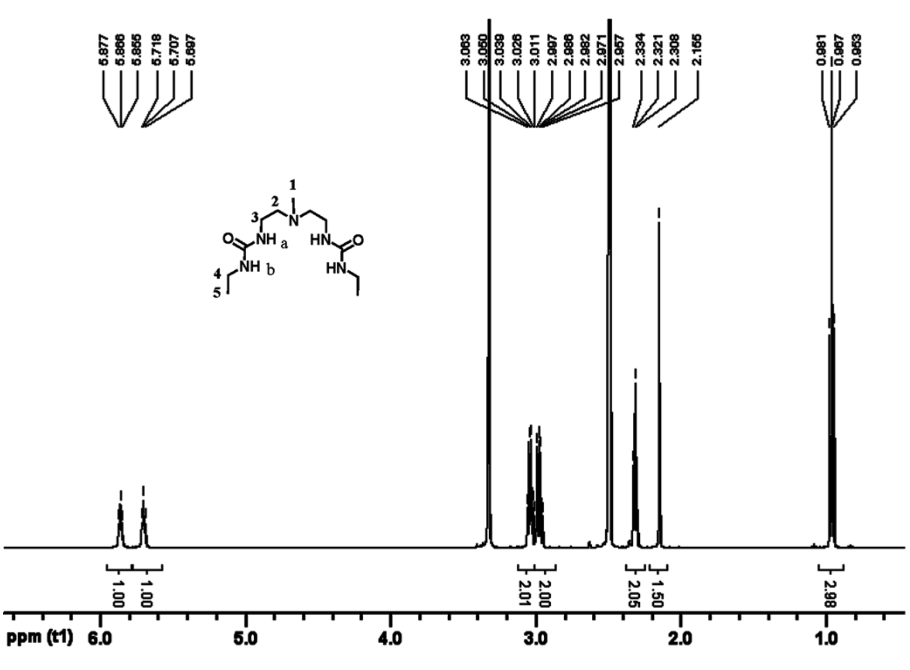


Figure S5. ^{1}H NMR (500 MHz, DMSO-d_6) of **4**.

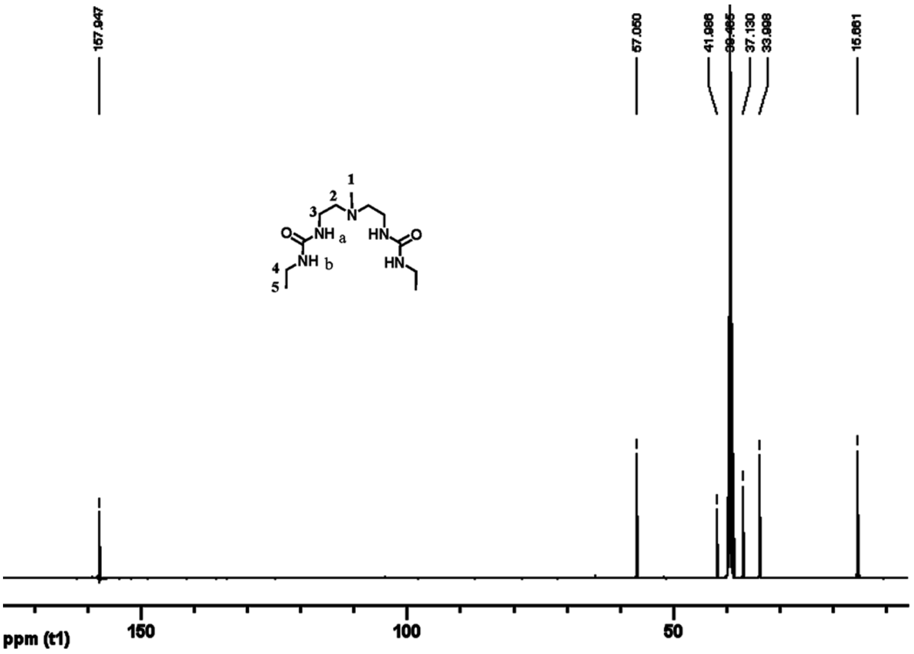


Figure S6. ^{13}C NMR (125 MHz, DMSO-d_6) of **4**.

Synthesis and characterization of **5**

1,2-phenyldiamine (0.50 g, 9.2 mmol) and excessive ethyl isocyanate (2.63g, 37.0 mmol) in 70 mL THF/40 mL Toluene were reacted in room temperature overnight, and then the slurry thus obtained was filtrated which gave pure **5** as a white solid (0.62 g, 53 %). ^{1}H NMR (500 MHz, DMSO-d_6, ppm): δ 7.76 (s, 2H, NHa), 7.46 (m, 2H, H2), 6.96 (m, 2H, H1), 6.48 (t, J = 5.5 Hz, 2H, NHb), 3.10 (m, 2H, H3), 1.05 (t, J = 8.0 Hz, 2H, H4). ^{13}C NMR (125 MHz, DMSO-d_6), δ 155.8 (C=O), 131.5 (Ar), 123.4 (Ar), 123.1 (Ar), 34.2 (CH2), 15.4 (CH3). Exact mass for $C_{12}H_{18}N_4O_2$ + Cl^- 285.1124, found (HREIMS$^-$) 285.1116.

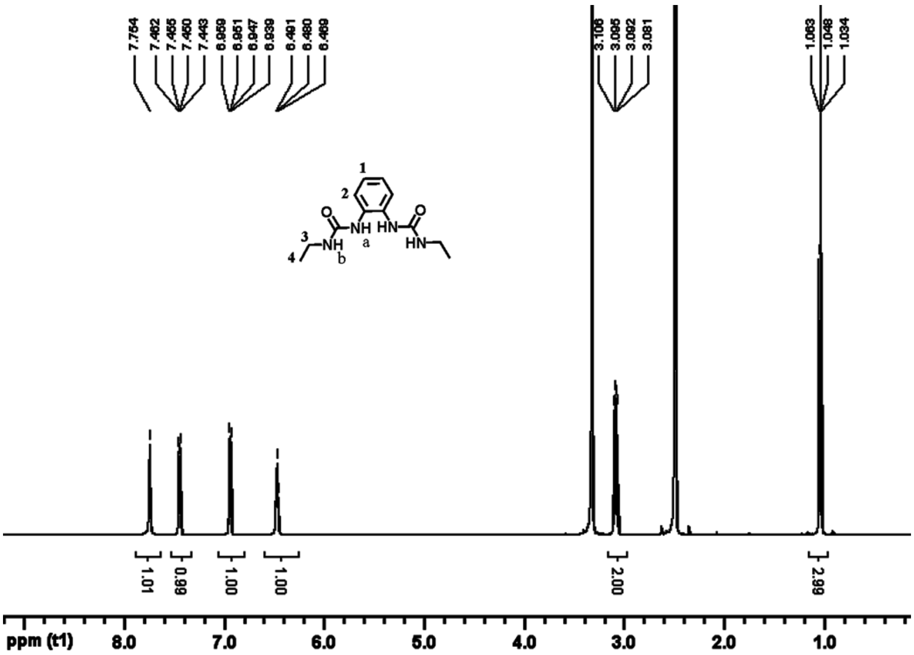


Figure S7. ^{1}H NMR (500 MHz, DMSO-d_6) of **5**.

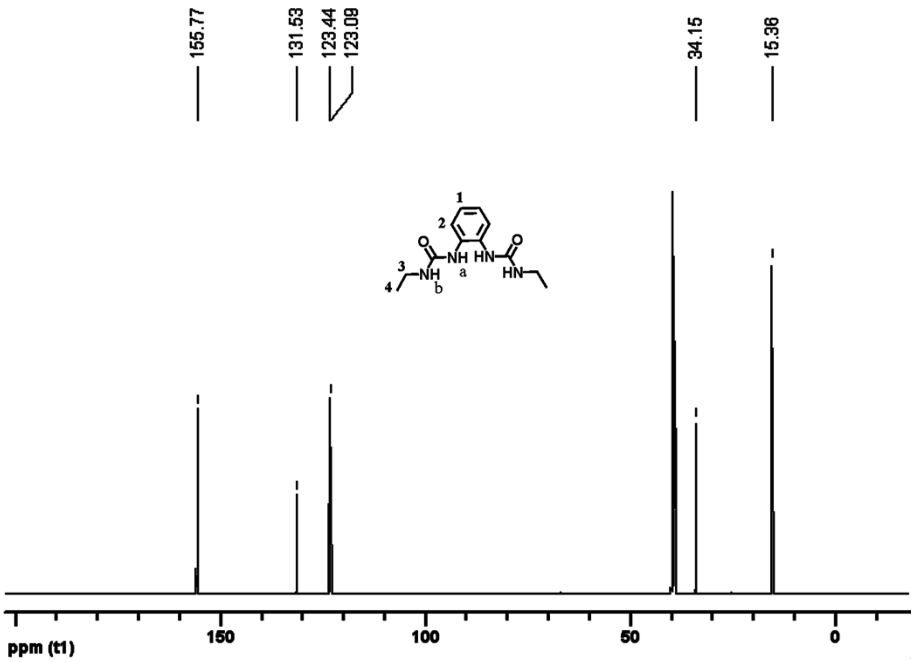
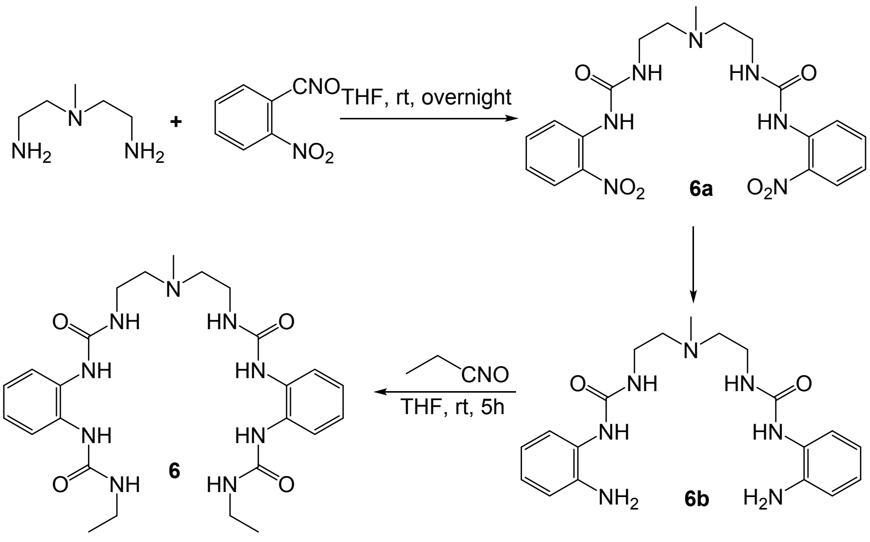


Figure S8. ^{13}C NMR (125 MHz, DMSO-d_6) of **5**.

Synthesis and characterization of **6**



6a: 2,2'-diamino-N-methyldiethylamine (0.71 g, 6.0 mmol) and 2-nitrophenylisocyanate (2.00 g, 12.0 mmol) were mixed in 20 mL anhydrous THF in an ice-water bath. After reacting overnight, the solvent was removed by rotary evaporation in room temperature which gave pure **6a** (2.53 g, 94%) as a bright yellow solid. ^{1}H NMR (400 MHz, DMSO-d_6, ppm): δ 9.35 (s, 2H, NHb), 8.24 (d, J = 8.0 Hz, 2H, H7), 8.00 (dd, J = 8.0 Hz, 2H, H4), 7.59 (m, J = 8.0 Hz, 2H, H6), 7.43 (br, 2H, NHa), 7.10 (t, J = 8.0 Hz, 2H, H6), 3.21 (m, 4H, H3), 2.47 (t, J = 6.4 Hz, 4H, H2), 2.26 (s, 3H, H1). ^{13}C NMR (125 MHz, DMSO-d_6), δ 154.2 (C=O), 135.7 (Ar), 136.9 (Ar), 134.8 (Ar), 125.2 (Ar), 122.0 (Ar), 121.3 (Ar), 56.3 (CH2), 41.9 (CH2), 37.2 (CH3). Exact mass for $C_{11}H_{25}N_5O_2$ + Cl^- 480.1398, found (TOF-MS-ES$^-$) 480.1376.

6b: Hydrazine monohydrate (2.0 mL) was added dropwise to the suspension of compound **6a** (2.0 g 4.5 mmol) and Pd/C 10% (0.066 g, cat.) in 100 mL MeOH. After refluxing under intensive stirring for 3.5 hours, the black Pd/C was filtered off through Celite and the solvent in the filtrate was removed by rotary evaporation which gave a light brown oil. The oil was redissolved in acetone and white solid was precipitated by diethyl ether. After dried in vacuum, analytically pure **6b** was obtained as a white solid (1.20 g, 69 %). ^{1}H NMR (400 MHz, DMSO-d_6, ppm): δ 7.64 (s, 2H, NHb), 7.25 (d, J = 8.0 Hz, 2H, H4), 6.78 (t, J = 8.0 Hz, 2H, H6), 6.68 (d, J = 8.0 Hz, 2H, H7), 6.51 (t, J = 8.0 Hz, 2H, H5), 6.16 (t, J = 5.5 Hz, 2H, NHa), 4.71 (s, 4H, NHc), 3.18 (m, 4H, H3), 2.45 (t, J = 6.4 Hz, 4H, H2), 2.23 (s, 3H, H1). ^{13}C NMR (125 MHz, DMSO-d_6), δ 155.9 (C=O), 140.6 (Ar), 125.4 (Ar), 123.8 (Ar), 123.4 (Ar), 116.6 (Ar), 115.7 (Ar), 56.8 (CH2), 41.9 (CH2), 37.2 (CH3). Exact mass for $C_{11}H_{25}N_5O_2$ + Cl^- 420.1920, found

(TOF-MS-ES$^-$) 420.1917.

6: Compound **6b** (0.61 g, 1.6 mmol) and excessive ethyl isocyanate (0.45 g, 6.4 mmol) were suspended in 150 mL of THF. After reacting for 5 h in room temperature, the solvent was removed by rotary evaporation and the solid thus obtained was washed with diethyl ether and then dried over vacuum to yield ligand **6** as a white powder (0.51 g, 60%). ^{1}H NMR (400 MHz, DMSO-d_6, ppm): δ 7.91 (s, 2H, NHb), 7.77 (s, 2H,NHc), 7.49 (d, J = 8.0, 2H, H4), 7.43 (d, J = 8.0 Hz, 2H, H7), 6.95 (m, 4H, H5 and H6), 6.49 (m, 4H, NHa and NHd), 3.19 (m, 4H, H3), 3.07 (m, 4H, H8), 2.46 (t, J = 8.0 Hz, 4H, H2), 2.24 (s, 3H, H1), 1.03 (t, J = 8.0 Hz, 6H, H9). ^{13}C NMR (125 MHz, DMSO-d_6), δ 156.0 (C=O), 155.8 (C=O), 131.8 (Ar), 131.3 (Ar), 123.6 (Ar), 123.3 (Ar), 123.0 (Ar), 56.8 (CH2), 41.9 (CH2), 37.3 (CH2), 34.2 (CH3), 15.4 (CH3). Exact mass for $C_{25}H_{37}N_9O_4$ + Cl$^-$ 562.2663, found (HREIMS$^-$) 562.2657.

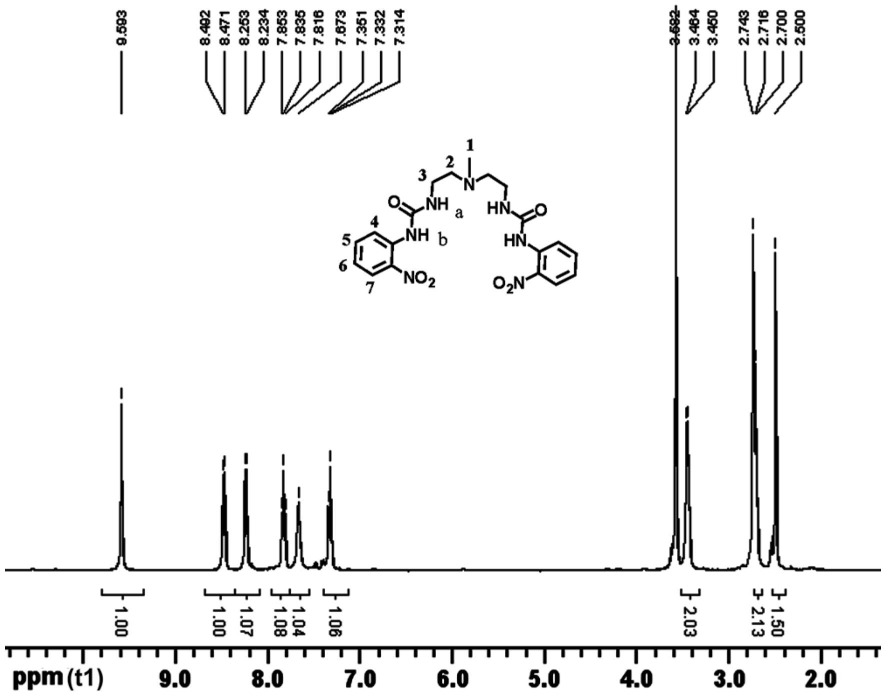


Figure S9. ^{1}H NMR (500 MHz, DMSO-d_6) of **6a**.

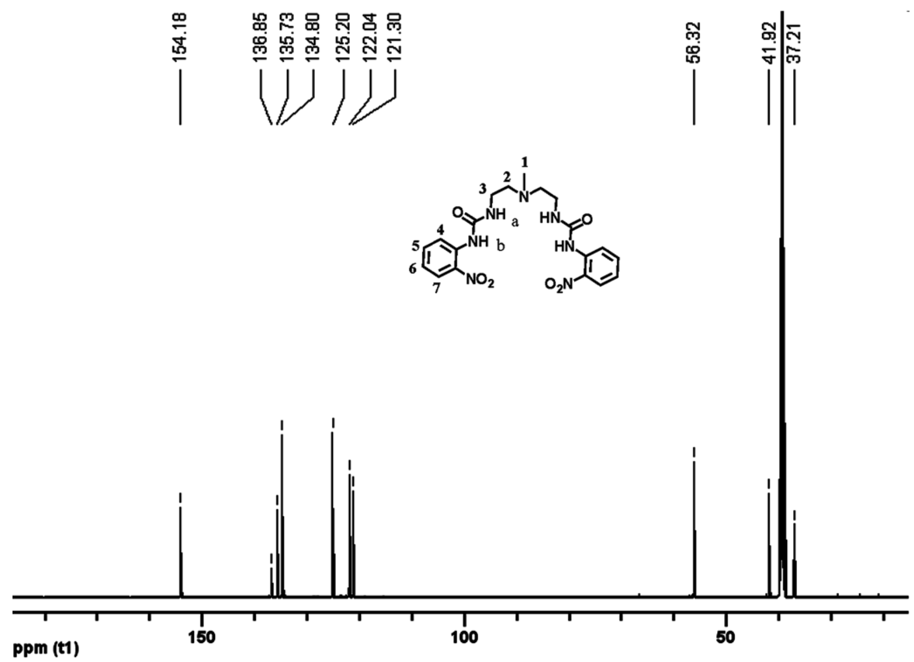


Figure S10. ^{13}C NMR (125 MHz, DMSO-d_6) of **6a**.

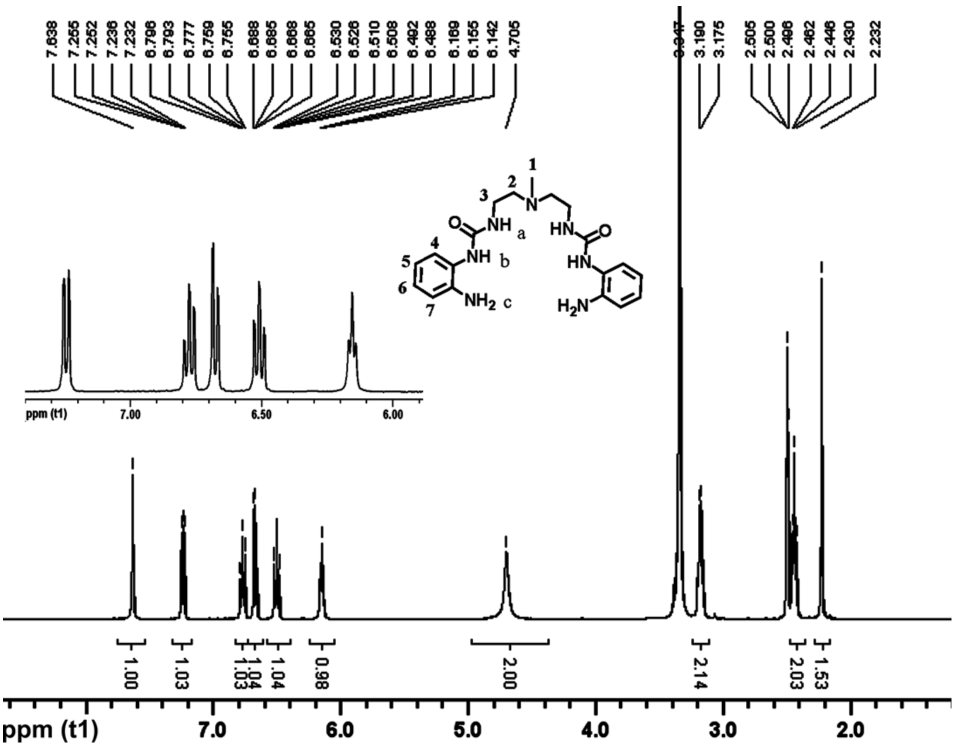


Figure S11. ^{1}H NMR (500 MHz, DMSO-d_6) of **6b**.

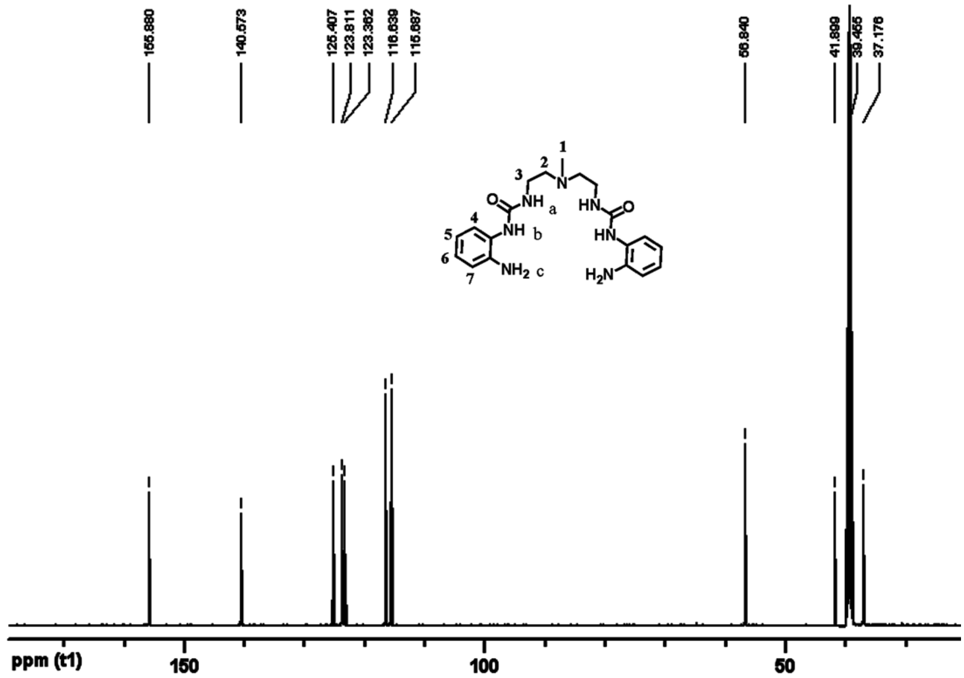


Figure S12. ^{13}C NMR (125 MHz, DMSO-d_6) of **6b**.

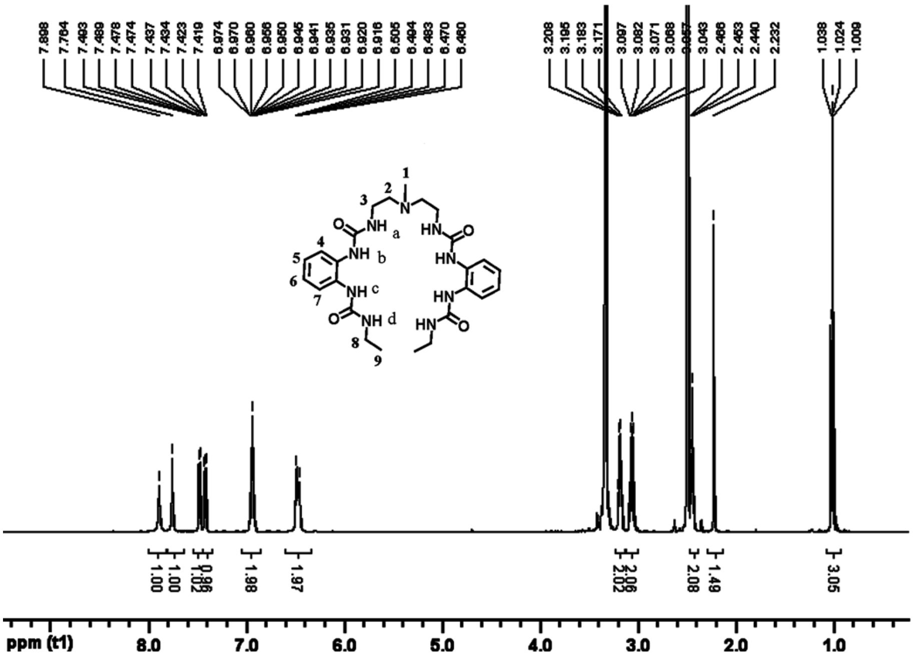


Figure S13. ^{1}H NMR (500 MHz, DMSO-d_6) of **6**.

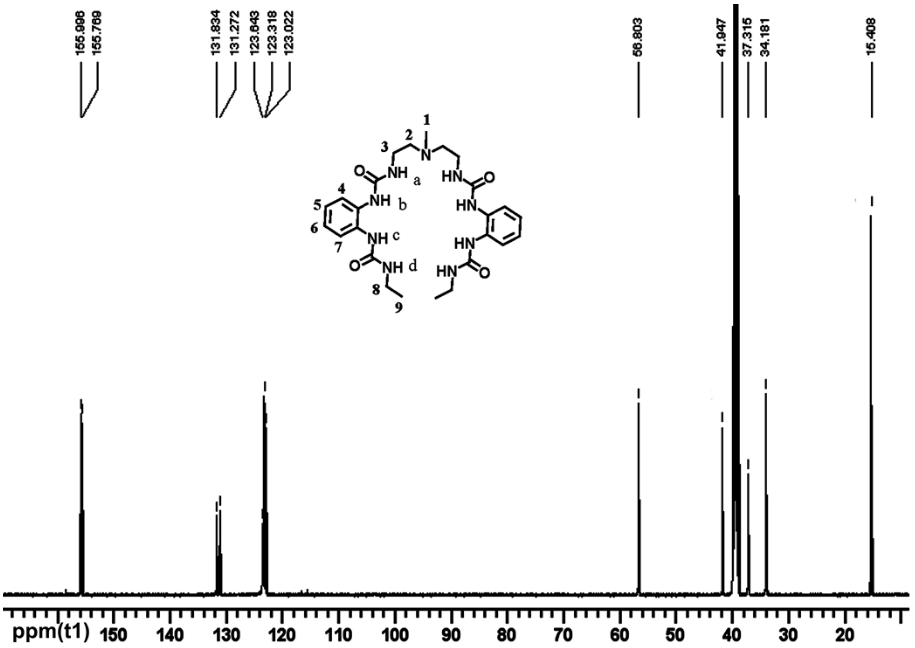


Figure S14. ^{13}C NMR (125 MHz, DMSO-*d₆*) of **6**.

2. Binding studies

All anions used in binding studies are tetrabutyl ammonium (TBA) salts. Since the $(TBA)_2SO_4$ was purchased in a 50% H_2O% solution (Sigma Aldrich) since sulfate dianion is not soluble in neat DMSO. Thus the TBA salt of necessity is generally purchased as the 50% H_2O solution when titrations involving the dianion are sought. Stock solutions were prepared in DMSO-d_6 with aliquots diluted with DMSO-d_6 according to desired water concentrations, and were corrected based on the integral of TBA^+/Ligand. The association constants are calculated by fitting the titration profiles to 1:1 binding model with EQNMR program.[S4] The 1:1 *solution* binding model in all cases was confirmed by Job's plot (Figure S16) (although as noted for compound **5** this was not the case in the crystallographic results). For ligands with more than one NH signals, calculations based on all NH groups' titration profiles are conducted and the ones show the lowest errors are chosen for using. Competition experiments are shown in **Figure S15**. In all titrations, DMSO-d_6-0.5% water (Sigma Aldrich) was used to prepare DMSO solutions with desired water concentration (v/v%).

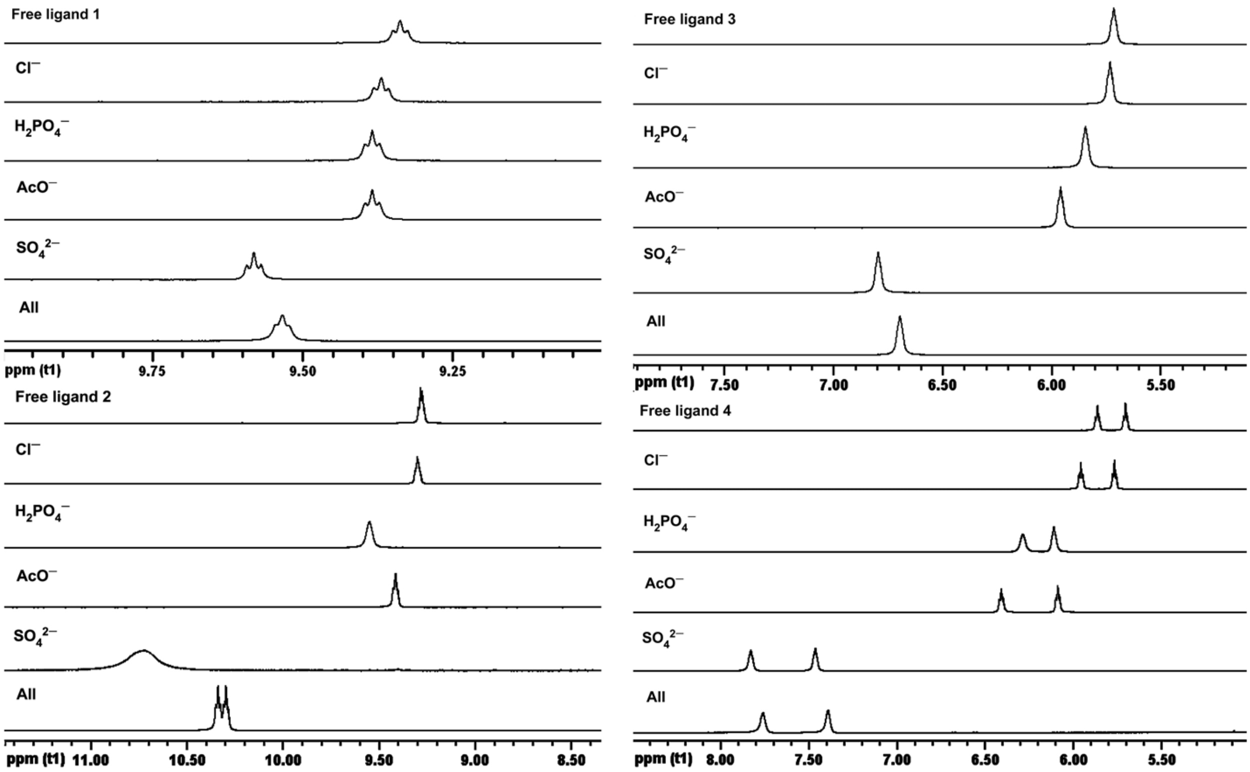


Figure S15. Partial ^{1}H NMR spectra (500 MHz, 293 K, DMSO-d_6) of ligand **1-4** showing the NH signals shifts in the presence of 1 equiv of various anions (all added as TBA salts) separately and together.

Job's plots were determined using ^{1}H NMR spectroscopy. Stock solutions of host (10.0 mM) and the guest anion (10.0 mM) in DMSO-d_6 (5.0 mL) were prepared in separate volumetric flasks. Ten 5 mm-o.d. NMR tubes were separately filled with a total of 500 μL solution of the host and guest in the following ratios (μL, host/guest) at 297 K: 10:0, 9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8, 1:9. The ^{1}H NMR spectra were obtained for each tube. $[HG] = [H]_t \times (\delta_{obsd} - \delta_{free})/(\delta_{com} - \delta_{free})$, where $[H]_t$ is the total concentration of the host, δ_{obsd} is the chemical shift observed on every point, δ_{free} and δ_{com} corresponds to the chemical shifts of the free ligand and the complex. This value was plotted against the mole fraction of the host (**Figure S16)**.

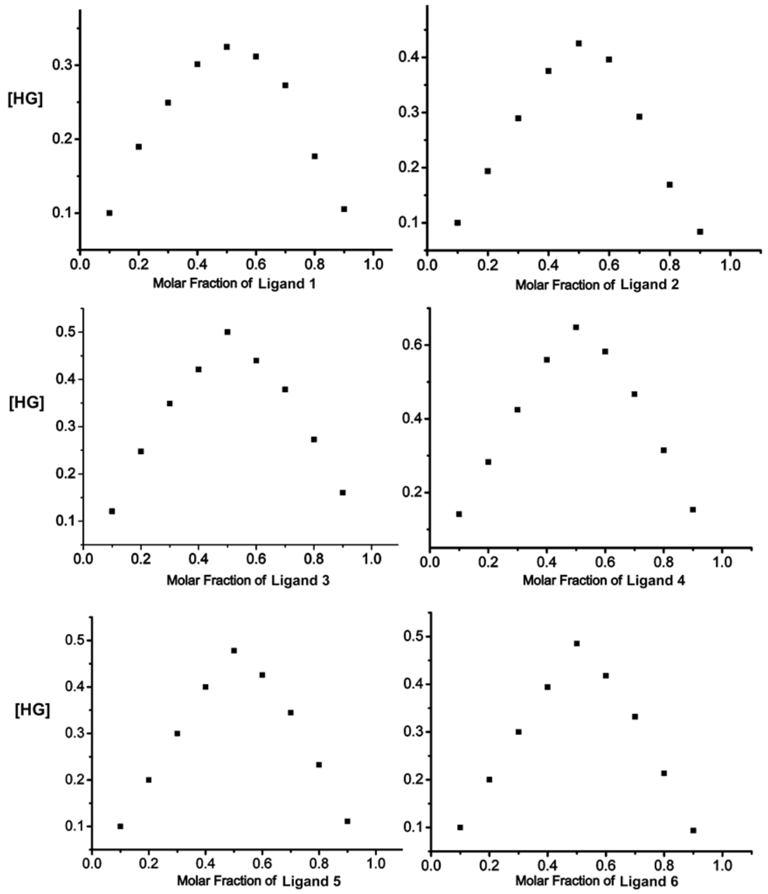


Figure S16. Job's plots between ligand **1-6** and SO_4^{2-}. Points were obtained based on changes of the signals of NH used for calculations.

3. Single crystal X-ray diffraction studies

Redundant sets of unique diffraction data were collected with 0.5°-wide ω- or ϕ-scans using the Bruker Apex2 Software Suite [S5-S6] at 100(2) K for a single-domain crystal of **A** and 2-domain non-merohedral twins of **B** and **C.** The measurements used monochromated CuKα radiation (λ= 1.54178 Å) on a Bruker Proteum Single Crystal Diffraction System equipped with Helios multilayer optics, an APEX II CCD detector and a Bruker MicroSTAR microfocus rotating anode x-ray source operating at 45kV and 60mA. The data were corrected empirically for variable absorption effects using equivalent reflections. The Bruker software package SHELXTL was used to solve all three structures using "direct methods" techniques. All stages of weighted full-matrix least-squares refinement were conducted using F_o^2 data with the SHELXTL Version 2013.10-0 software package[S7]. Final crystallographic details are summarized in Table S1. These structures have been deposited in the Cambridge Crystallographic Data Centre, CCDC 1051283-1051285.

The final structural models incorporated anisotropic thermal parameters for all ordered and major occupancy disordered nonhydrogen atoms. Isotropic thermal parameters were incorporated for the remaining nonhydrogen atoms and all included hydrogen atoms. Methyl groups for **A**, **B** and **C** were incorporated into the structural models as rigid groups (using idealized sp^3-hybridized geometry and a C-H bond length of 0.98 Å) that were either allowed to rotate about their C-C bonds during least squares refinement cycles (**A**) or placed at idealized "staggered" positions (**B** and **C**). The remaining hydrogen atoms for **A**, **B** and **C** were included in the structural model as idealized atoms (assuming sp^2- or sp^3-hybridization of the carbon or nitrogen atoms and C-H bond lengths of 0.95 or 0.99 Å and N-H bond lengths of 0.88 Å). The isotropic thermal parameters of all idealized hydrogen atoms were fixed at values 1.2 (nonmethyl) or 1.5 (methyl) times the equivalent isotropic thermal parameter of the carbon or nitrogen atom to which they are covalently bonded. Hydrogen atoms were not included for water molecules of crystallization in **C**.

Crystals of **B** appear metrically to utilize a C-centered monoclinic unit cell but are actually triclinic (primitive cell: a = 9.00 Å, b = 18.46 Å, c = 29.88 Å,α = 105.61°,β = 98.65° , γ = 90.05° and V = 4724 $Å^3$) and twinned to look monoclinic. The reflections used to determine a preliminary unit cell and orientation matrix for the crystal appeared to indicate a single-domain specimen: 95% of the reflections used to determine the preliminary orientation matrix indexed satisfactorily on the (reduced) primitive triclinic unit cell and indicated the lattice was probably C-centered monoclinic. The integrated intensities were consistent with monoclinic Laue symmetry (R_{sym} = 0.042) and the structure was originally solved (with difficulty) and refined in the monoclinic space group C2[S8]. Most, but not all, of the anticipated atoms were present and ordered. However, one of the acyclic hosts utilized a crystallographic two-fold axis to form a cyclic host with a (50/50) disordered amine nitrogen completing the ring. Interestingly, in this description, one of the sulfate sulfurs and one of the ammonium nitrogens each occupied different crystallographic C2 axes and generated ordered anions and cations, respectively. This structure gave R_1 = 0.108 with anisotropic nonhydrogen atoms for all ordered species. One cation was incomplete and severely disordered about another crystallographic C2 axis; its nonhydrogen atoms were included in the structural model with isotropic thermal parameters.

The space group symmetry of **B** was changed to noncentrosymmetric triclinic C1 [a nonstandard setting for P1 - C_1^1 (No. 1)] [S8] and the structure was then solved and refined with this lower symmetry. The previously cyclic host was now acyclic and the structure refined to R_1 = 0.079 with anisotropic nonhydrogen atoms and idealized hydrogen atoms. Introducing 54/46 two-domain 180° twinning about the (pseudo-monoclinic) b axis gave R_1 = 0.053. One methylene group (C7I) in a cation is 61/39 disordered in this structure. All other atoms in **B** are ordered.

The crystal of **C** was a 2-domain 55/45 nonmerohedral twin. The asymmetric unit contains four crystallographically- independent anion/cation pairs and two water molecules of crystallization. Three of the four sulfate anions are (83/17, 68/32 and 93/7) disordered, each with two orientations in the lattice. Anisotropic thermal parameters were used to model all full-occupancy nonhydrogen atoms in **C** and major-occupancy sulfate nonhydrogen atoms as well as the sulfur atom having an occupancy factor of 0.82. The remaining minor occupancy sulfate nonhydrogen atoms and all included hydrogen atoms were modeled with isotropic thermal parameters. The bond lengths and angles for the three minor occupancy sulfate groups were mildly restrained to have values similar to those of the full-occupancy sulfate. Hydrogen atoms were not included in the structural model for the two water molecules of crystallization in C.

The CIF files for q72c (compound B) and q73c (compound C) produced checkCIF A-Alerts. Two of the A-Alerts for q73c pertained to the nonstandard choice of space group that the software doesn't/can't recognize. As indicated above, the nonstandard C-centered triclinic space group, C1, was chosen for q72c (compound B) to show the pseudomerohedral twinning and how it related to the monoclinic symmetry elements which the structure appeared/tried to mimic. Both q72c (B) and q73c (C) were twinned and disordered. This can easily produce the remaining A-Alerts for these two compounds: anomalous C-C bond lengths (q72c; compound B) and a wide range of isotropic thermal parameters for nonsolvent hydrogen atoms (q72c, compound B and q73c, compound C).

Table S1. Crystal Data and Structure Refinement for $(TBA)_2[\mathbf{4}\cdot(SO_4)]$ (**A**), $(TBA)_2[\mathbf{5}\cdot(SO_4)]$ (**B**) and $(TBA)_2[\mathbf{6}\cdot(SO_4)]$ (**C**).

	A	**B**	**C**
Empirical formula	$C_{43}H_{97}N_7O_6S$	$C_{57}H_{109}N_{11}O_8S$	$C_{56}H_{108}N_{10}O_{8.50}S$
Formula weight	840.33	1108.61	1089.58
Temperature	100(2) K	100(2) K	100(2) K
Wavelength	1.54178 Å	1.54178 Å	1.54178 Å
Crystal system	Monoclinic	Triclinic	Triclinic
Space group	$P2_1$– C_2^2 (No. 4)[S4]	C1 – [nonstandard setting of C_1^1 (No. 1)] [S4]	P-1 – C_i^1 (No. 2) [S4]
a	9.1475(3) Å	59.086(2) Å	22.1618(9) Å
b	22.3413(7) Å	9.0002(4) Å	23.5118(9) Å
c	12.6753(4) Å	18.4644(6) Å	26.7043(11) Å
a	90°	90.050(2)°	95.763(2)°
β	100.559(1)°	105.800(2)°	102.319(2)°
γ	90°	89.986(2)°	108.227(2)°
Volume	2546.55(14) Å^3	9448.2(6) Å^3	12700(1) Å^3
Z	2	6	8
Density (calculated)	1.096 g/cm^3	1.169 g/cm^3	1.139 g/cm^3
Absorption coefficient	0.937 mm^{-1}	0.921 mm^{-1}	0.906 mm^{-1}
F(000)	936	3648	4784
Crystal size	0.40 x 0.22 x 0.07 mm^3	0.23 x 0.025 x 0.02 mm^3	0.25 x 0.20 x 0.15 mm^3
Theta range	3.55 to 69.40°	1.55 to 69.39°	2.01 to 70.28°
Index ranges	-9≤h≤10, -26≤k≤27, -12≤l≤15	-70≤h≤71, -9≤k≤10, -21≤l≤21	-27≤h≤25, -28≤k≤28, 0≤l≤32
Reflections collected	21070	61879	65391
Independent reflections	8045 [R_{int} = 0.020]	23820 [R_{int} = 0.050]	65391
Completeness to θ = 66.00°	98.5 %	99.0 %	97.7 %
Absorption correction	Multi-scan	Multi-scan	Multi-scan
Max. and min. transmission	1.000 and 0.853	1.000 and 0.860	1.000 and 0.804
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	8045 / 1 / 525	23820 / 27 / 2086	65391 / 30 / 2788
Goodness-of-fit on F^2	1.025	1.017	1.041
Final R indices [I>2σ(I)]	R_1 = 0.031, wR_2 = 0.081	R_1 = 0.053, wR_2 = 0.135	R_1 = 0.114, wR_2 = 0.316
R indices (all data)	R_1 = 0.031, wR_2 = 0.081	R_1 = 0.060, wR_2 = 0.142	R_1 = 0.143, wR_2 = 0.348
Largest diff. peak and hole	0.51 and -0.22 e^-/Å^3	0.57 and -0.24 e^-/Å^3	1.00 and -0.62 e^-/Å^3

Table S2. Hydrogen bonds [Å and °] in the three crystal structures of (TBA)$_2$[**4**·(SO$_4$)] (**A**), (TBA)$_2$[**5**·(SO$_4$)] (**B**) and (TBA)$_2$[**6**·(SO$_4$)] (**C**). (#1: Symmetry transformations used to generate equivalent atoms: #1 -x,y-1/2,-z)

Complex **A**, (TBA)$_2$[**4**·(SO$_4$)]

D–H···*A*	*d*(*D*–H)	*d*(H···*A*)	*d*(D···*A*)	∠(*D*H*A*)
N(4)-H(4N)...O(11)	0.88	2.03	2.826(2)	149.6
N(6)-H(6N)...O(12)	0.88	1.95	2.799(2)	160.3
N(11)-H(11N)...O(12)#1	0.88	1.94	2.791(2)	160.9
N(13)-H(13N)...O(14)#1	0.88	2.00	2.871(2)	168.0

Complex **B**, (TBA)$_2$[**5**·(SO$_4$)]

D–H···*A*	*d*(*D*–H)	*d*(H···*A*)	*d*(D···*A*)	∠(*D*H*A*)
N(3A)-H(3AA)...O(11)	0.88	2.39	3.239(10)	161.7
N(3A)-H(3AA)...O(41)	0.88	2.59	3.298(9)	137.7
N(5A)-H(5AA)...O(41)	0.88	2.00	2.813(8)	153.2
N(12A)-H(12A)...O(41)	0.88	1.93	2.789(8)	166.6
N(14A)-H(14A)...O(31)	0.88	2.30	3.071(8)	146.7
N(20A)-H(20A)...O(31)	0.88	2.12	2.987(9)	169.7
N(22A)-H(22A)...O(21)	0.88	1.95	2.820(9)	167.3
N(29A)-H(29A)...O(21)	0.88	1.89	2.742(8)	162.6
N(31A)-H(31A)...O(12)	0.88	2.29	3.129(10)	158.9
N(3B)-H(3BA)...O(12)	0.88	2.08	2.937(11)	162.9
N(5B)-H(5BA)...O(22)	0.88	1.92	2.800(10)	174.8
N(12B)-H(12B)...O(22)	0.88	1.84	2.714(9)	170.6
N(14B)-H(14B)...O(22)	0.88	2.57	3.245(11)	134.5
N(14B)-H(14B)...O(42)	0.88	2.45	3.227(12)	146.9
N(20B)-H(20B)...O(42)	0.88	2.13	2.989(11)	165.3
N(22B)-H(22B)...O(32)	0.88	1.87	2.747(9)	176.0
N(29B)-H(29B)...O(32)	0.88	1.91	2.785(9)	177.0
N(31B)-H(31B)...O(12)	0.88	2.38	3.146(13)	146.1
N(3C)-H(3CA)...O(13)	0.88	2.34	3.202(9)	165.1
N(3C)-H(3CA)...O(43)	0.88	2.64	3.334(9)	136.0
N(5C)-H(5CA)...O(43)	0.88	1.97	2.804(9)	157.6
N(12C)-H(12C)...O(43)	0.88	2.02	2.871(9)	161.8
N(14C)-H(14C)...O(33)	0.88	2.32	3.028(9)	137.6
N(20C)-H(20C)...O(33)	0.88	2.14	3.010(9)	169.0
N(22C)-H(22C)...O(23)	0.88	1.95	2.809(9)	165.4
N(29C)-H(29C)...O(23)	0.88	1.82	2.696(9)	173.2
N(31C)-H(31C)...O(13)	0.88	2.29	3.153(10)	167.5

Complex **C**, (TBA)$_2$[**6**·(SO$_4$)]

D–H···*A*	*d*(*D*–H)	*d*(H···*A*)	*d*(D···*A*)	∠(*D*H*A*)
N(3)-H(3N)...O(14A)	0.88	2.04	2.887(8)	160.0
N(5)-H(5N)...O(14A)	0.88	2.35	3.137(8)	148.5
N(12)-H(12N)...O(11A)	0.88	1.97	2.811(7)	160.4
N(14)-H(14N)...O(11A)	0.88	2.47	3.203(7)	141.1
N(14)-H(14N)...O(13A)	0.88	2.18	2.978(7)	151.0
N(23)-H(23N)...O(12A)	0.88	2.09	2.897(11)	152.7
N(25)-H(25N)...O(12A)	0.88	2.08	2.900(10)	155.4
N(32)-H(32N)...O(13A)	0.88	2.08	2.921(7)	158.7
N(34)-H(34N)...O(13A)	0.88	2.25	2.997(8)	143.2
N(43)-H(43N)...O(41A)	0.88	2.15	2.991(7)	160.3
N(45)-H(45N)...O(41A)	0.88	2.46	3.200(6)	141.4
N(45)-H(45N)...O(43A)	0.88	2.22	2.992(7)	146.9
N(52)-H(52N)...O(43A)	0.88	1.99	2.811(7)	155.5
N(54)-H(54N)...O(43A)	0.88	2.16	2.937(8)	146.9
N(63)-H(63N)...O(24A)	0.88	2.41	3.186(10)	147.5
N(65)-H(65N)...O(24A)	0.88	2.16	2.997(8)	159.0
N(72)-H(72N)...O(23A)	0.88	2.04	2.895(7)	163.3
N(74)-H(74N)...O(22A)	0.88	2.47	3.094(10)	128.6
N(74)-H(74N)...O(23A)	0.88	2.34	3.120(8)	148.5
N(83)-H(83N)...O(41A)	0.88	2.06	2.926(7)	169.3
N(92)-H(92N)...O(42A)	0.88	1.94	2.815(6)	170.3
N(94)-H(94N)...O(44A)	0.88	2.12	2.971(6)	163.9
N(103)-H(103)...O(34A)	0.88	2.09	2.968(8)	172.0
N(105)-H(105)...O(32A)	0.88	2.17	2.915(8)	141.7
N(112)-H(112)...O(32A)	0.88	1.94	2.813(6)	170.1
N(114)-H(114)...O(33A)	0.88	2.16	2.928(7)	145.0
N(123)-H(123)...O(22A)	0.88	2.02	2.890(8)	172.3
N(125)-H(125)...O(21A)	0.88	2.34	3.101(9)	145.6
N(132)-H(132)...O(21A)	0.88	1.94	2.812(8)	170.0
N(134)-H(134)...O(21A)	0.88	2.64	3.349(9)	138.0
N(134)-H(134)...O(24A)	0.88	2.15	2.986(10)	159.5
N(143)-H(143)...O(31A)	0.88	2.35	3.069(8)	139.0
N(145)-H(145)...O(31A)	0.88	2.01	2.802(7)	149.2
N(152)-H(152)...O(31A)	0.88	1.99	2.846(8)	165.0
N(154)-H(154)...O(31A)	0.88	2.54	3.277(8)	141.3
N(154)-H(154)...O(33A)	0.88	2.28	2.980(7)	136.4

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