

Synthesis

General methods

All the chemicals were obtained from Aldrich and were used without further purification unless specified otherwise. All the solvents used were HPLC grade. Dimethyl sulfoxide (DMSO) and chloroform used in reactions were obtained anhydrous in SureSeal bottles and used under nitrogen flow. Chloroform-d (CDCl_3 , 99.8%) and DMSO- d_6 (99.8%) were purchased from Cambridge Isotopes. 1,3-phenyl diisocyanate (PDI), 2,6-tolylene diisocyanate (TDI) and *o*-tolylene isocyanate (OTI) were stored at 4 °C and were warmed to room temperature before use.

Melting points (m.p.) were recorded with a capillary melting point apparatus (Thomas Hoover) and are uncorrected. The recorded R_f values were determined by a standard thin-layer chromatography (TLC) procedure: 0.25 mm silica gel plates (Aldrich, Z122785-25EA) eluting with the specified solvents. FTIR spectra were recorded with a Magna-IR Spectrometer 550 (Nicolet) in KBr pellets. 300 MHz ^1H NMR spectra and 75 MHz ^{13}C NMR spectra were recorded on a Varian 300 spectrometer. 500 MHz ^1H NMR were recorded on a Varian 500 spectrometer. The spectra were recorded at room temperature unless specified otherwise. The residual proton signals of the deuterated solvents were used as internal standards (DMSO- d_6 : δ (^1H) = 2.50 ppm, δ (^{13}C) = 39.51 ppm; CDCl_3 : δ (^1H) = 7.27 ppm, δ (^{13}C) = 77.23 ppm). The following notation is used for the ^1H NMR splitting patterns: singlet (s), doublet (d), triplet (t), quartet (q), doublet-doublet (d-d), triplet-doublet (t-d), multiplet (m) and broad signal (br). ^1H coupling constants are given in Hz and the values are for three-bond coupling protons unless specified otherwise. MALDI-TOF mass spectra were obtained using a Micromass BAA037 (Micromass, UK) time-of-flight mass spectrometer. A nitrogen laser (337 nm wavelength) was used to desorb the sample ions. The matrices used were dihydroxybenzoic acid (DHB), dithranol (DIT), or *trans*-indoacrylic acid (IAA) and the spectra were recorded in the positive mode. ESI MS spectra were recorded on a Micromass Q-TOF2 mass spectrometer. X-ray diffraction data for **2** was collected on a Bruker AXS Smart 2K/Platform diffractometer with a graphite monochromated $\text{CuK}\alpha$ radiation (1.54178 Å), θ scans at 100(2) K. For **3c** and **4** and **24**, the data was collected on a Bruker D8 X-ray diffractometer with a graphite monochromatized $\text{MoK}\alpha$ radiation (0.71073 Å), θ scans at 100(2) K.

N,N-bis-(4-decyl-phenyl)-pyridazine-3,6-diamine was synthesized using a similar procedure described by Kumagai.^[1] Bis-alkylation of 3,6-dichloropyridazine required the use of the hydrochloride salt of the corresponding amine.^[2] 2,7-diamino-1,8-naphthyridine was synthesized following literature procedures.^[3-5] During the preparation of this publication, a new and safer procedure was published by Zimmerman *et al.*^[6]

Experimental procedures

***N,N*-Bis-(4-decyl-phenyl)-pridazine-3,6-diamine** 3,6-dichloropyridazine (0.50 g, 3.36 mmol) and 4-decylaniline (1.84 g, 7.88 mmol) were reacted at 120 °C for 15 minutes (the mixture became very viscous). The crude product was dissolved in ethanol (10 mL) and precipitated by increasing the pH with concentrated aqueous sodium hydroxide. The product was filtered and the solid was redissolved in chloroform and extracted with water to remove the salt. The organic layer was dried with anhydrous sodium sulfate and filtered. The solvent was removed from the filtrate under vacuum to afford golden flake-like crystals (1.50 g, 82.6%). R_f = 0.57 (chloroform/acetone, 8/2); m.p. = 193.5-195.5 °C; ^1H NMR (300 MHz, DMSO- d_6 , 60 °C): δ = 8.61 (s, 2H), 7.58 (d, J = 8.4, 4H), 7.07 (d, J = 8.5, 4H), 7.04 (s, 2H), 3.19 (m, 4H), 1.56 (m, 4H), 1.26-1.30 (m, 28H), 0.86 (t, J = 6.6, 6H); ^{13}C NMR (75 MHz, CDCl_3): δ = 153.64, 138.24, 138.04, 129.54, 120.41, 117.80, 35.54; 32.13, 31.83, 29.94, 29.84, 29.75, 29.56, 29.52, 22.91, 14.34; ESI MS: m/z calcd for $[\text{M}+\text{H}]^+$: 543.443; found: 543.422.

3-Chloro-6-dodecyl-pyridazine 3,6-dichloropyridazine (4.00 g, 26.8 mmol) and 1-dodecylamine (15.0 g, 81.1 mmol) were reacted at 90-100 °C for 0.5 hours and then refluxed in ethanol (20 mL) for another 1 hour. The crude product was precipitated in a saturated aqueous solution of NaHCO_3 to yield a white powder (5.86 g, 73.2%). R_f = 0.67 (chloroform/acetone, 9.5/0.5); m.p = 85-86 °C; ^1H NMR (300 MHz, CDCl_3): δ = 7.15 (d, J = 9.3, 1H), 6.67 (d, J = 9.3, 1H), 5.02 (m, 1H), 3.36 (m, 2H), 1.64 (m, 2H), 1.25-1.37 (m, 18H), 0.88 (t, J = 6.9, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ = 158.83, 146.58, 129.11, 116.06, 42.49, 32.14, 29.87, 29.847, 29.81, 29.57, 29.51, 27.20, 22.91, 14.34; ESI MS: m/z calcd for $[\text{M}+\text{H}]^+$: 298.205; found: 298.206.

Dodecylamine hydrochloride Dodecylamine (~10 g) dissolved in ethanol (*ca.* 50 mL) and excess concentrated hydrochloride acid (37%) was mixed at room temperature. The salt solution was placed in shallow dishes and air-dried in the fume hood. The product was isolated as a white powder in quantitative yield. m.p. = 175-176 °C; ^1H NMR (300 MHz, CDCl_3): δ = 8.29 (s, 3H), 2.99 (m, 2H), 1.77 (m, 2H), 1.26 (m, 18H), 0.89 (t, J = 6.6, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ = 40.22, 32.13, 29.84, 29.83, 29.77, 29.61, 29.56, 29.20, 27.92, 26.72, 22.90, 14.32.

***N,N*-Didodecyl-pyridazine-3,6-diamine** 3-chloro-6-dodecyl-pyridazine (1.00 g, 3.35 mmol) and 1-dodecylamine chloride (1.49 g, 6.70 mmol) were mixed and purged with nitrogen for 15 minutes. The mixture was reacted at 170 °C for 3 hours. After cooling to room temperature, the black product mixture was dissolved in chloroform (20 mL) and washed with a saturated aqueous solution of NaHCO_3 (2 x 10 mL) and water (2 x 10 mL). The organic layer was dried over anhydrous sodium sulfate and concentrated to about 10 mL by vacuum evaporation. Silica gel chromatography (chloroform/methanol/acetone, 8.5/1.5/0.5) was used to isolate the desired product (0.64 g, 42.0%). R_f = 0.50 (chloroform/methanol/acetone, 8.5/1.0/0.5); m.p. = 124-125 °C; ^1H NMR (300 MHz, CDCl_3): δ = 6.57 (s, 2H), 4.13 (m, 2H), 3.31 (m, 4H), 1.60 (m, 4H), 1.25-1.36 (m, 36H), 0.88

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(t, J = 6.6, 3H); ^{13}C NMR (75 MHz, CDCl_3): δ = 154.57, 117.75, 42.91, 32.10, 29.92, 29.84, 29.80, 29.62, 29.53, 27.29, 22.87, 14.29; ESI MS: m/z calcd for $[\text{M}+\text{H}]^+$: 447.443; found: 447.421.

(6-Chloro-pyridazin-3-yl)-isobutyl-amine 3,6-dichloropyridazine (4.00 g, 26.8 mmol) and isobutylamine (9.82 g, 135 mmol) were dissolved in ethanol (15 mL) and reacted under reflux for 3 hours. Excess isobutylamine was removed by vacuum evaporation and the product was recrystallized from ethanol to yield yellow needle-like crystals (4.21 g, 78.5%). R_f = 0.39 (chloroform/acetone, 9/1); m.p. = 85-86 °C. ^1H NMR (300 MHz, CDCl_3): δ = 7.15 (d, J = 9.3, 1H), 6.67 (d, J = 9.3, 1H), 5.05 (s, 1H), 3.19 (m, 2H), 1.934 (m, H), 0.98 (d, J = 6.6, 3H). ^{13}C NMR (75 MHz, CDCl_3): δ = 158.95, 146.53, 129.10, 115.88, 49.93, 28.34, 20.42; ESI MS: m/z calcd for $[\text{M}+\text{H}]^+$: 186.080; found: 186.069.

Isobutylamine hydrochloride Isobutylamine (~10 g) diluted with ethanol (*ca.* 50 mL) and an excess of concentrated hydrochloric acid were mixed at room temperature. The salt solution was placed in shallow dishes and air-dried in the fume hood. The product was obtained as a white powder in quantitative yield. m.p. = 157-158 °C; ^1H NMR (300 MHz, CDCl_3): δ = 8.34 (s, 3H), 2.83 (t, 2H), 2.08 (m, 1H), 1.05 (d, J = 6.6, 6H); ^{13}C NMR (75 MHz, CDCl_3): δ = 47.06, 27.21, 20.16.

***N,N*-Diisobutyl-pyridazine-3,6-diamine** (6-Chloro-pyridazin-3-yl)-isobutylamine (1.30 g, 7.03 mmol) and isobutylamine hydrochloride (2.10 g, 19.5 mmol) were mixed and the flask was purged with nitrogen for 15 minutes. The mixture was heated at 140 °C for 3 hours. After cooling to room temperature, the black product mixture was dissolved in chloroform (20 mL) and washed with a saturated aqueous solution of NaHCO_3 (2 x 10 mL) and water (2 x 10 mL). The organic layer was dried over anhydrous sodium sulfate and concentrated to about 10 mL by vacuum evaporation. Silica gel column chromatography (chloroform/methanol/acetone, 8.5/1.0/0.5) was used to isolate the desired product (0.66 g, 42.3%). R_f = 0.48 (chloroform/methanol/acetone, 8.5/1.0/0.5); m.p. = 141.5-142.5 °C; ^1H NMR (300 MHz, CDCl_3): δ = 6.62 (s, 2H), 4.40 (s, br, 2H), 3.13 (m, 4H), 1.90 (m, 2H), 0.94 (d, J = 6.6, 12H); ^{13}C NMR (75 MHz, CDCl_3): δ = 154.49, 118.03, 50.40; 28.32, 20.49; ESI MS: m/z calcd for $[\text{M}+\text{H}]^+$: 223.192; found: 223.177.

Macrocycle 1: Cyclo-tris (2,7-(1,8-naphthyridyl)-urea) 2,7-diamino-1,8-naphthyridine (0.10g, 0.63 mmol) was dispersed in 1 mL DMSO under nitrogen to form a yellow suspension at 120 °C. 1,1'-carbonyldiimidazole (0.17 g, 0.94 mmol, 50% molar excess) was dissolved in DMSO (0.2 mL) at room temperature under nitrogen and then added dropwise into the suspension. The solid was completely dissolved resulting in a clear yellow solution. The solution gradually became cloudy over a period of 30 minutes. The reaction was continued at 120 °C under nitrogen for 24 hours and then cooled to room temperature. The reaction mixture was filtered and washed with methanol to yield a light yellow powder (0.075 g, 64%). ^1H NMR (300 MHz, $\text{DMSO}-d_6$): δ = 12.22 (s, 3H), 10.68 (s, 3H), 8.61 (d, J = 9.1, 3H), 8.37 (d, J = 9.1, 3H), 8.39 (d, J = 9.1, 3H), 7.26 (d, J = 9.1, 3H); IR (KBr, $\nu = \text{cm}^{-1}$): 3140, 3120, 1720; ESI MS: m/z calcd for $[\text{M} + \text{H}]^+$ 559.170; found: 559.170.

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Macrocycle 2: Cyclo-tris (2,7-(1,8-naphthrydyl) formamidine 2,7-diamino-1,8-naphththyridine (0.048 g, 0.30 mmol) was dissolved in DMSO (0.3 mL) at 120 °C to form a yellow suspension to which triethylorthoformate (0.089 g, 0.60 mmol) was added under nitrogen. The solution immediately turned bright yellow and turned red-brown in 2 hours. After cooling to room temperature, the product was precipitated with methanol. The solid was filtered and washed with methanol and vacuum dried at 60 °C for 24 hours to yield a bright yellow powder (0.038 g, 74%). ¹H NMR (300 MHz, DMSO-d₆): δ = 11.54 (s, 1 H), 10.68(s, 1 H), 8.32(d, J = 8.5, 2 H), δ 7.23 (d, J = 7.5, 2H); ¹³C (75 MHz, DMSO-d₆): δ = 154.53, 151.56, 139.03; IR (KBr, ν = cm⁻¹): 3375, 3202, 3315, 1654, 1594, 1121; ESI MS : *m/z* calcd for [M+H]⁺: 511.186; found: 511.191.

Macrocycle 3a: To a flask with dried *N, N*-bis-(4-decyl-phenyl)-pyridazine-3,6-diamine (0.90 g, 1.66 mmol) was added a solution of tolylene-2,6-diisocyanate (0.35 g, 2.00 mmol) in anhydrous chloroform (50 mL). The resulting light brown solution was allowed to react at 50 °C under N₂ for 24 hours. Methanol (5 mL) was then added and the product mixture was stirred overnight at room temperature to neutralize unreacted isocyanate. Solvent was removed under vacuum and the product was redissolved in chloroform (30 mL) and filtered. The filtrate was concentrated to about half the volume and then separated by silica gel column chromatography (chloroform/acetone, 9.5/0.5) to yield 0.73 g (61.4 %) of the target product. *R_f* = 0.67 (chloroform/acetone, 9.5/0.5); m.p. =191-192 °C; ¹H NMR (300 MHz, CDCl₃): δ = 12.15 (s, 4H), 8.02 (d, J = 8.4, 4H), 7.31 (d, J = 8.4, 8H), 7.24 (d, J = 8.4, 8H), 7.10 (t, J = 8.4, 2H), 6.51 (s, 4H), 2.81 (s, 6H), 2.64 (m, 8H), 1.64 (m, 8H), 1.28-1.34 (m, 56H), 0.90 (t, J = 6.9Hz, 12H); ¹³C NMR (75 MHz, CDCl₃): δ=154.99, 152.82, 143.98, 137.56, 136.96, 130.50, 129.69, 126.58, 122.13, 117.13, 116.55, 35.85, 32.10, 31.53, 29.83, 29.78, 29.71, 29.61, 29.53, 22.88, 14.69, 14.32; IR (KBr, ν in cm⁻¹): 2923, 2852, 1697, 1597, 1547, 1475, 1448, 1250; ESI MS: *m/z* : calcd for [M+H]⁺ : 1433.96; found 1433.99.

Macrocycle 3b: To a flask with *N,N*-didodecyl-pyridazine-3,6-diamine (0.33 g, 0.74 mmol) was added a solution of tolylene-2,6-diisocyanate (0.16 g, 0.89 mmol) in anhydrous chloroform (15 mL). The resulting yellow solution was allowed to react at 50 °C under nitrogen for 24 hours. Methanol (4 mL) was then added and the reaction mixture was stirred overnight at room temperature to neutralize unreacted isocyanate. Solvent was removed under vacuum and the product was redissolved in chloroform (20 mL) and filtered. The filtrate was concentrated to about half the volume and then separated by silica gel column chromatography (chloroform/acetone, 9.5/0.5) to yield the target product (0.31g, 67%). *R_f* = 0.63 (chloroform/acetone, 9.5/0.5); m.p. = 194-195 °C; ¹H NMR (300 MHz, CDCl₃): δ = 11.69 (s, 4H), 7.80 (d, J = 8.1, 4H), 7.23 (s, 4H), 6.99 (t, J = 8.1, 2H), 3.88 (t, J = 6.3, 8H), 2.36 (s, 6H), 1.71 (m, 8H), 1.28-1.38 (m, 72H), 0.89 (t, J = 6.6, 12H); ¹³C NMR (75 MHz, CDCl₃): δ = 153.86, 152.37, 137.61, 126.12, 120.82, 116.18, 116.01, 46.25, 32.14, 29.91, 29.89, 29.86, 29.83, 29.66, 29.57, 28.47, 27.19, 22.90, 14.77, 14.33; IR (KBr, ν in cm⁻¹): 2925, 2854, 1679, 1599, 1545, 1454, 1232; ESI MS: *m/z*: calcd for [M+H]⁺: 1241.96; found: 1241.99.

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Macrocycle 3c: To a flask with of *N,N*-diisobutyl-pyridazine-3,6-diamine (0.20 g, 0.90 mmol) was added a solution of tolylene-2,6-diisocyanate (0.40 g, 0.91 mmol) in anhydrous chloroform (8 mL). The resulting light brown solution was allowed to react at 50 °C under nitrogen for 24 hours. Methanol (4 mL) was then added and the product mixture was stirred overnight at room temperature to neutralize unreacted isocyanate. Solvent was removed under vacuum and the product was redissolved in chloroform (15 mL) and filtered. The filtrate was concentrated to about half the volume and then separated by silica gel column chromatography (chloroform/acetone, 9/1) to yield of the target product (0.23g, 64%). R_f = 0.55 (chloroform/acetone, 9/1); m.p. = 210-211 °C; ^1H NMR (300 MHz, CDCl_3): δ = 11.99 (s, 4H), 7.84 (d, J = 8.4, 4H), 7.43 (s, 4H), 7.22 (t, J = 8.4, 2H), 4.00 (d, J = 7.5, 8H), 2.56 (s, 6H), 1.96 (m, 4H), 1.00 (d, J = 6.6, 24H); ^{13}C NMR (75 MHz, CDCl_3): δ = 154.23, 153.28, 137.59, 126.54, 121.50, 118.10, 117.78, 51.35, 27.67, 20.15, 14.77; IR (KBr, ν in cm^{-1}): 2960, 2875, 1685, 1592, 1546, 1477, 1454, 1218; ESI MS: m/z : calcd for $[\text{M}+\text{H}]^+$: 793.46; found: 793.47.

Macrocycle 4: To a flask with 1,3-phenylene diisocyanate (0.12 g, 0.75 mmol) in anhydrous chloroform (5 mL) was added a solution of *N,N*-diisobutyl-pyridazine-3,6-diamine **21** (0.15 g, 0.68 mmol) in anhydrous chloroform (5 mL). The resulting light brown solution was allowed to react at 50 °C under nitrogen for 24 hours. Methanol (2 mL) was then added and the product mixture was stirred overnight at room temperature to neutralize unreacted isocyanate. Solvent was removed under vacuum and the product was redissolved in chloroform (15 mL) and filtered. The filtrate was concentrated to about half the volume and then separated by silica gel column chromatography (chloroform/acetone, 9/1) to yield the target product (0.12 g, 46%). R_f = 0.55 (chloroform/acetone, 9/1); m.p. = 212-213 °C; ^1H NMR (300 MHz, CDCl_3): δ = 12.00 (s, 4H), 7.90 (d-d, 3J = 8.4, 4J = 2.1, 4H), 7.44 (s, 4H), 7.35 (t, J = 8.4, 2H), 6.76 (t, 4J = 2.1, 2H), 3.96 (d, J = 7.5, 8H), 1.92 (m, J = 6.6, 4H), 0.97 (d, J = 6.6, 24H); ^{13}C NMR (75 MHz, CDCl_3): δ = 154.23, 153.00, 139.17, 130.12, 121.85, 116.03, 111.26, 51.27, 27.60, 20.04; IR (KBr, ν in cm^{-1}): 2962, 2877, 1680, 1610, 1570, 1549, 1495, 1454, 1338, 1232, 1209; ESI MS: m/z : calcd for $[\text{M}+\text{H}]^+$: 765.431; found: 765.432.

Characterization

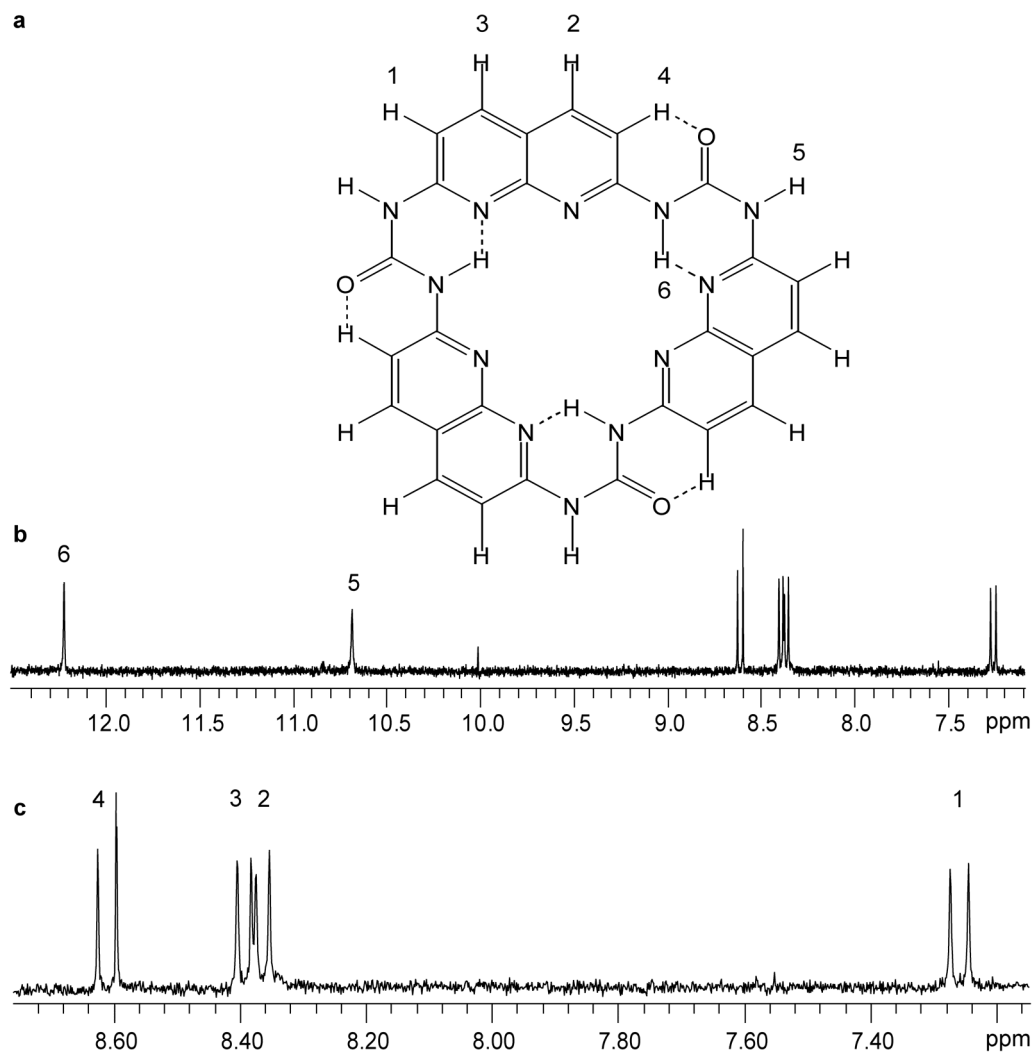


Figure S1. a) Structure of macrocycle **1** with hydrogen bonds represented by dashed lines; b) ^1H NMR spectrum of trimeric macrocycle **1** in DMSO-d_6 at $24.2\text{ }^\circ\text{C}$; c) expansion of the aromatic region of the NMR spectrum.

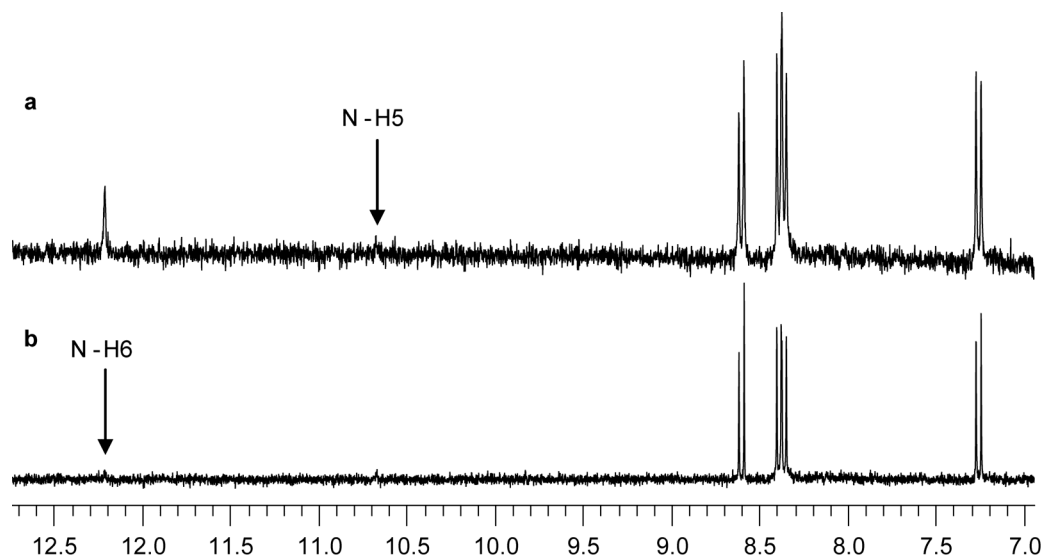


Figure S2. ^1H NMR spectra of macrocycle **1** after D_2O (5 μL) was added at room temperature (*ca.* 25 $^\circ\text{C}$): a) 5 minutes after addition; b) 30 minutes after addition. The arrows indicate the original positions of the urea protons.

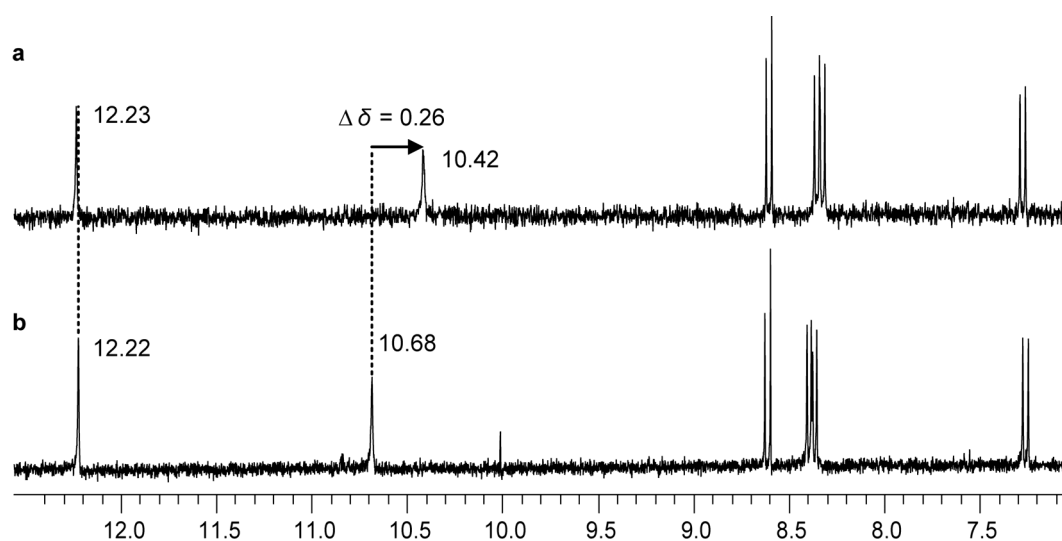


Figure S3. ^1H NMR spectra of macrocycle **1** at (a) 80 $^\circ\text{C}$ and (b) 24.2 $^\circ\text{C}$; with DMSO-d_6 as solvent.

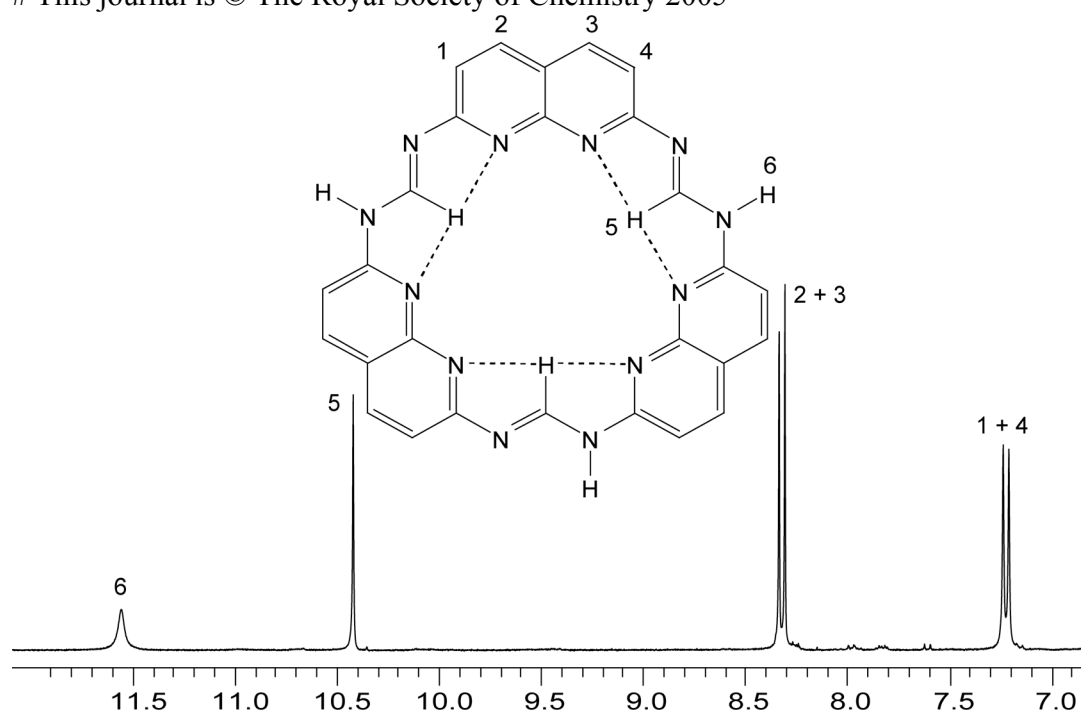


Figure S4. ^1H NMR spectrum of macrocycle **2** in DMSO-d_6 at room temperature.

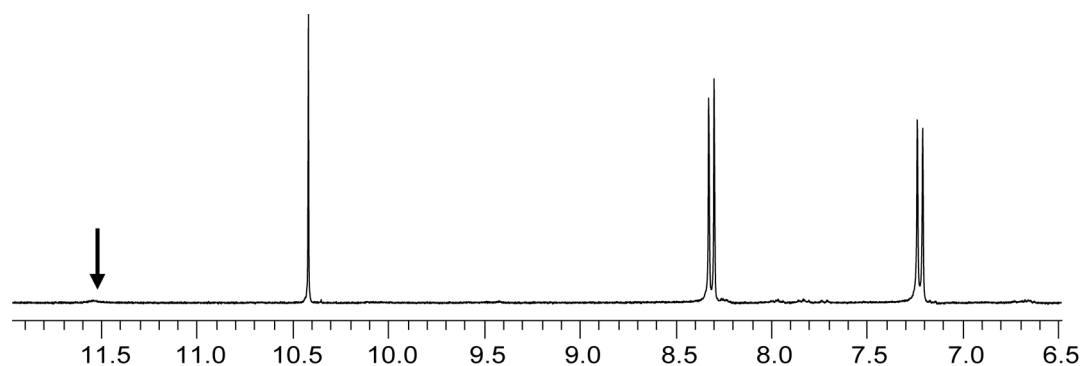


Figure S5. D/H exchange ^1H NMR experiment of macrocycle **2** in DMSO-d_6 with methanol-d_4 (room temperature). The spectrum was obtained 3 days after one drop of D_2O was added to the NMR tube. The arrow points to the residual signal after deuterium exchange.

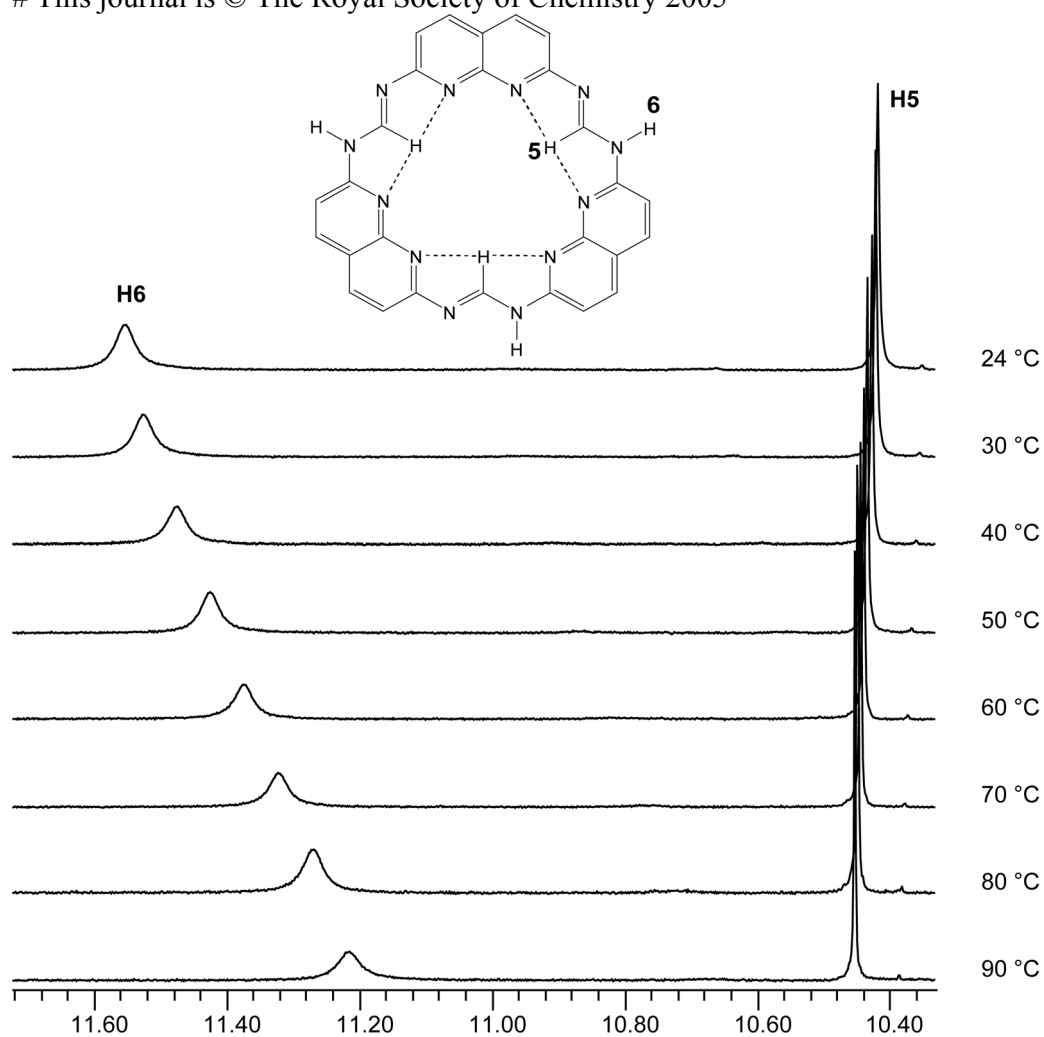


Figure S6. Variable temperature ^1H NMR spectra of macrocycle **2** in DMSO-d_6 .

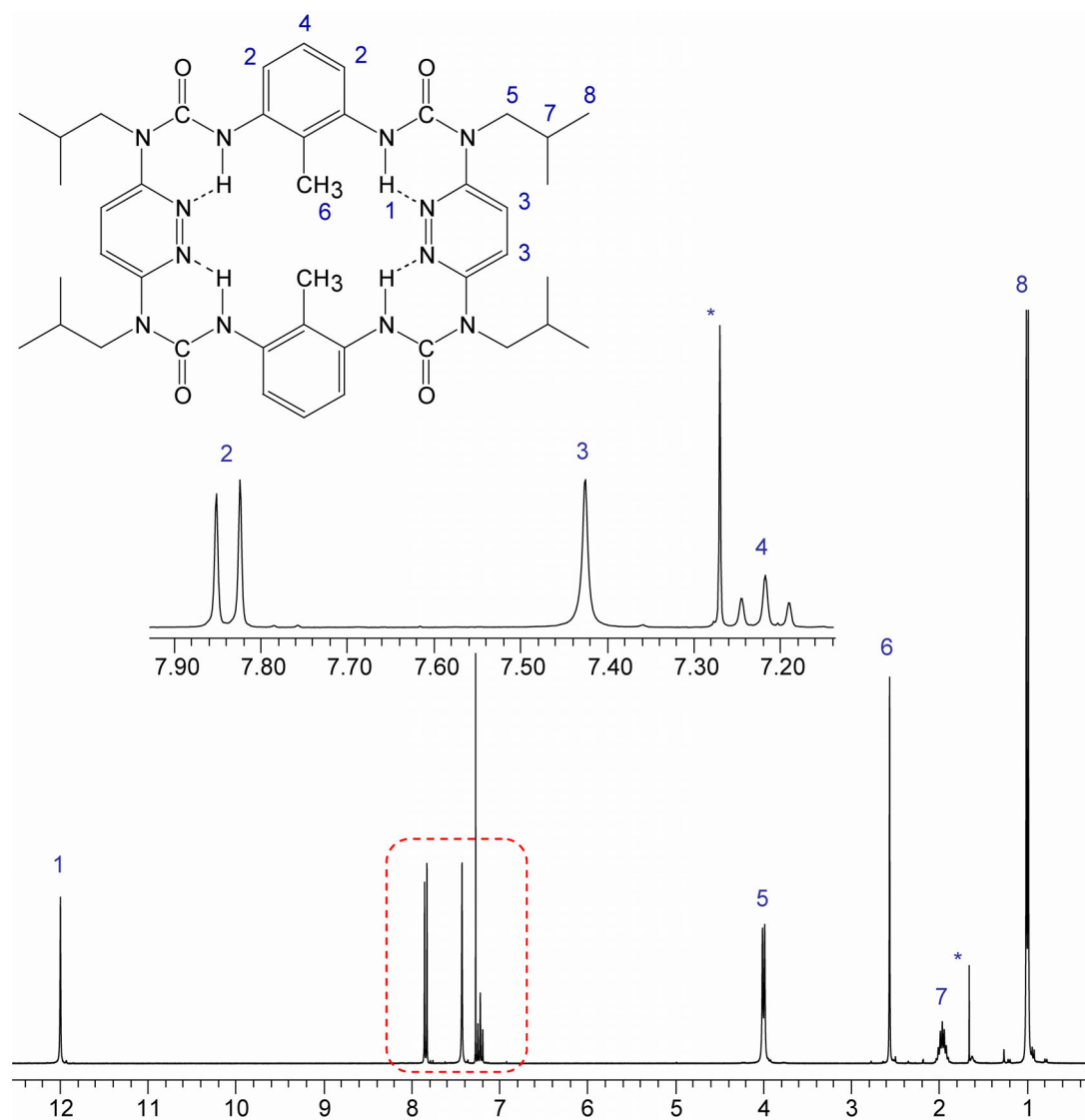


Figure S7. ¹H NMR spectrum of macrocycle **3c** in CDCl₃ with the aromatic region expanded. The signals labeled with asterisks are due to residual chloroform and water.

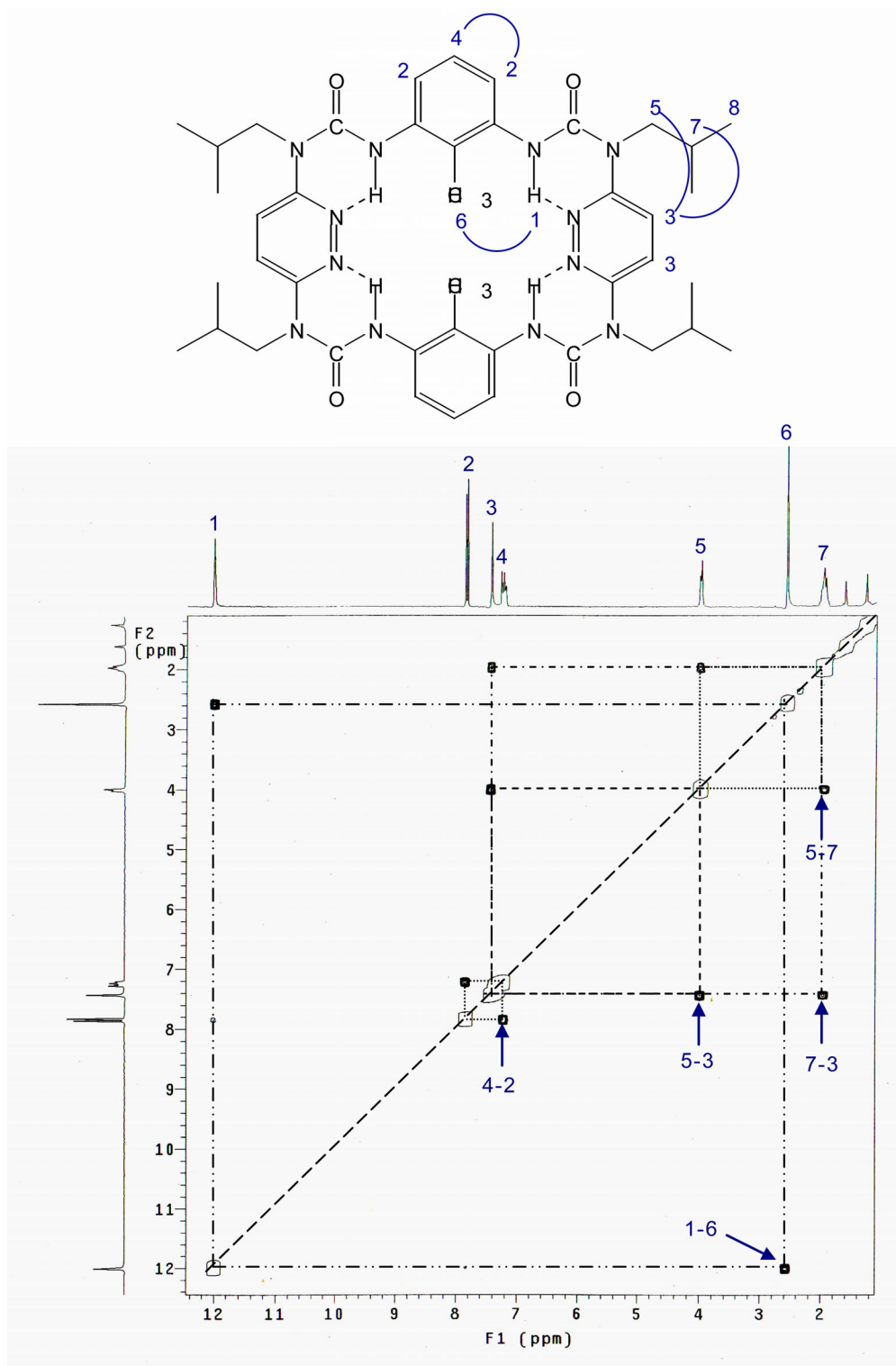


Figure S8. NOESY spectra of macrocycle **3c** in CDCl₃.

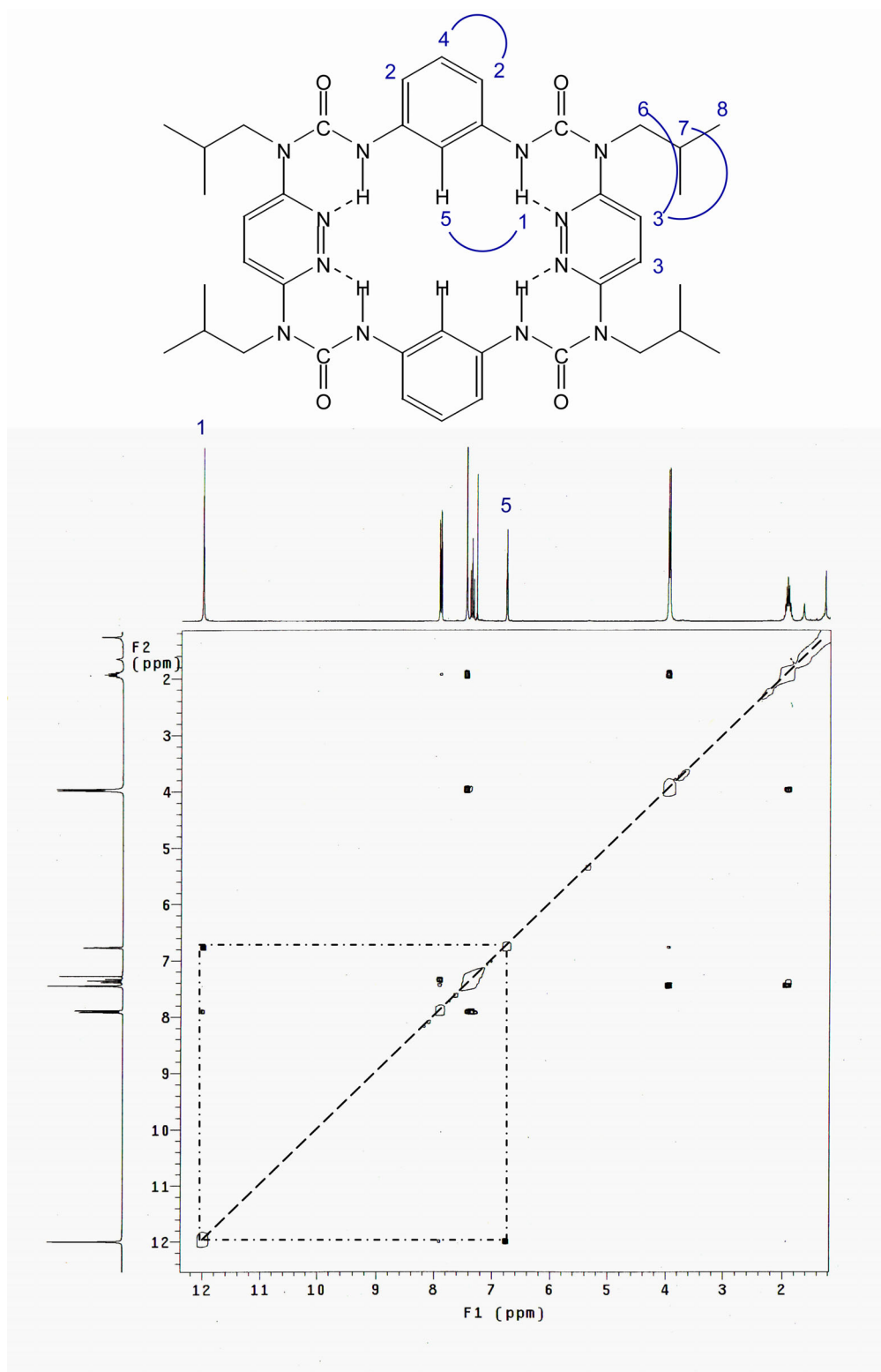


Figure S9. NOESY spectra of macrocycle **4** in CDCl₃.

Table S1. Crystallographic data and summary of data collection and refinement for **2**, **3c** and **4**.

| | 2 | 3c | 4 |
|---|---|---------------------------|------------------------|
| crystal dimensions, mm ³ | 0.5mm X 0.11mm X 0.1mm | 0.7 mm X 0.11 mm X 0.1 mm | 0.5mm X 0.11mm X 0.1mm |
| crystal system | Orthorhombic | Trigonal | Monoclinic |
| space group | Pbcn | P-3 | P21/c |
| <i>a</i> | 31.1325(6) | 20.888(4) | 7.25 |
| <i>b</i> | 8.5333(2) | 20.884(4) | 15.874 |
| <i>c</i> | 22.9610(4) | 8.774(4) | 19.137 |
| α | 90 | 90 | 90 |
| β | 90 | 90 | 100.56 |
| γ | 90 | 120 | 90 |
| <i>V</i> Å ³ | 6099.9(2) | 3315.2(17) | 2165.6(6) |
| <i>Z</i> | 8 | 6 | 1 |
| ρ_{calc} Mg m ⁻³ | 1.437 Mg/cm ³ | | |
| Max 2 θ deg | 55 | 27 | 27 |
| Radiation | CuK α | MoK α | MoK α |
| λ Å | 1.54178 | 0.71073 | 0.71073 |
| scan mode | CCD | CCD | CCD |
| <i>T</i> K | 100(2) | 100(2) | 373 |
| no. of measured/independent reflections | 57006/3816 | 16251/4348 | 16157/3810 |
| no. of reflections included in the refinement | 3816 | 4348 | 3810 |
| Sigma limits | | $I > 2\sigma(I)$ | |
| method of structure solution and program | | SHELXS-97 | |
| method of refinement and program | | SHELXL-97 | |
| no. of parameters | 448 | 283 | 267 |
| treatment of H atoms | The H atoms were generated geometrically (C-H 0.95 to 0.98 N-H 0.88 and O-H 0.84 Å) and were included in the refinement in the riding model approximation | | |
| <i>R</i> (<i>F</i>) for $I > 2\sigma(I)$ | 0.0394 | 0.0493 | 0.07 |
| <i>wR</i> | 0.0629 | 0.1164 | 0.2213 |
| whether refined against <i>F</i> or <i>F</i> ² | | <i>F</i> ² | |
| database at which the detailed results are deposited. | CCDC 267802 | CCDC 267803 | CCDC 267804 |

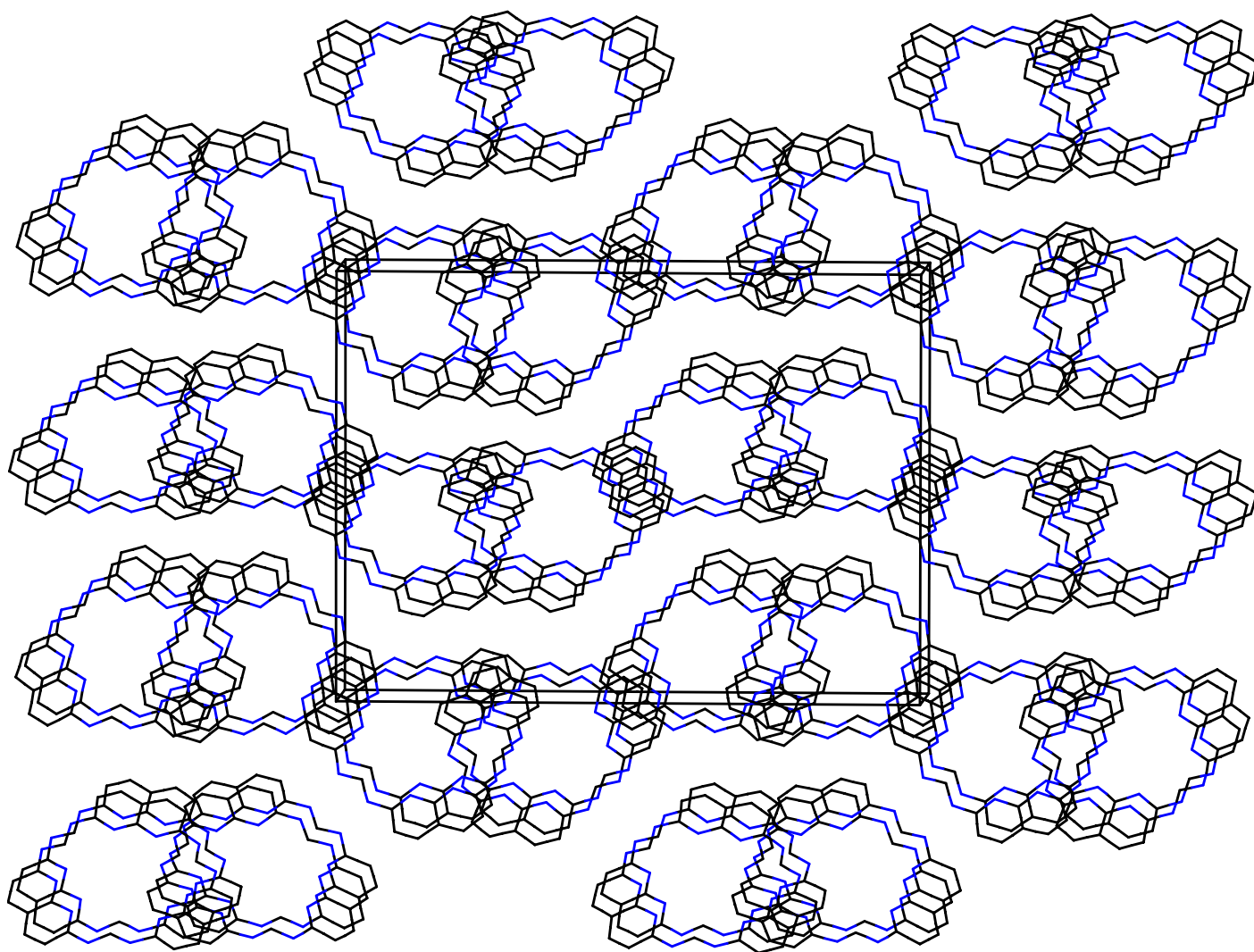


Figure S10. Crystal packing of macrocycle 2.

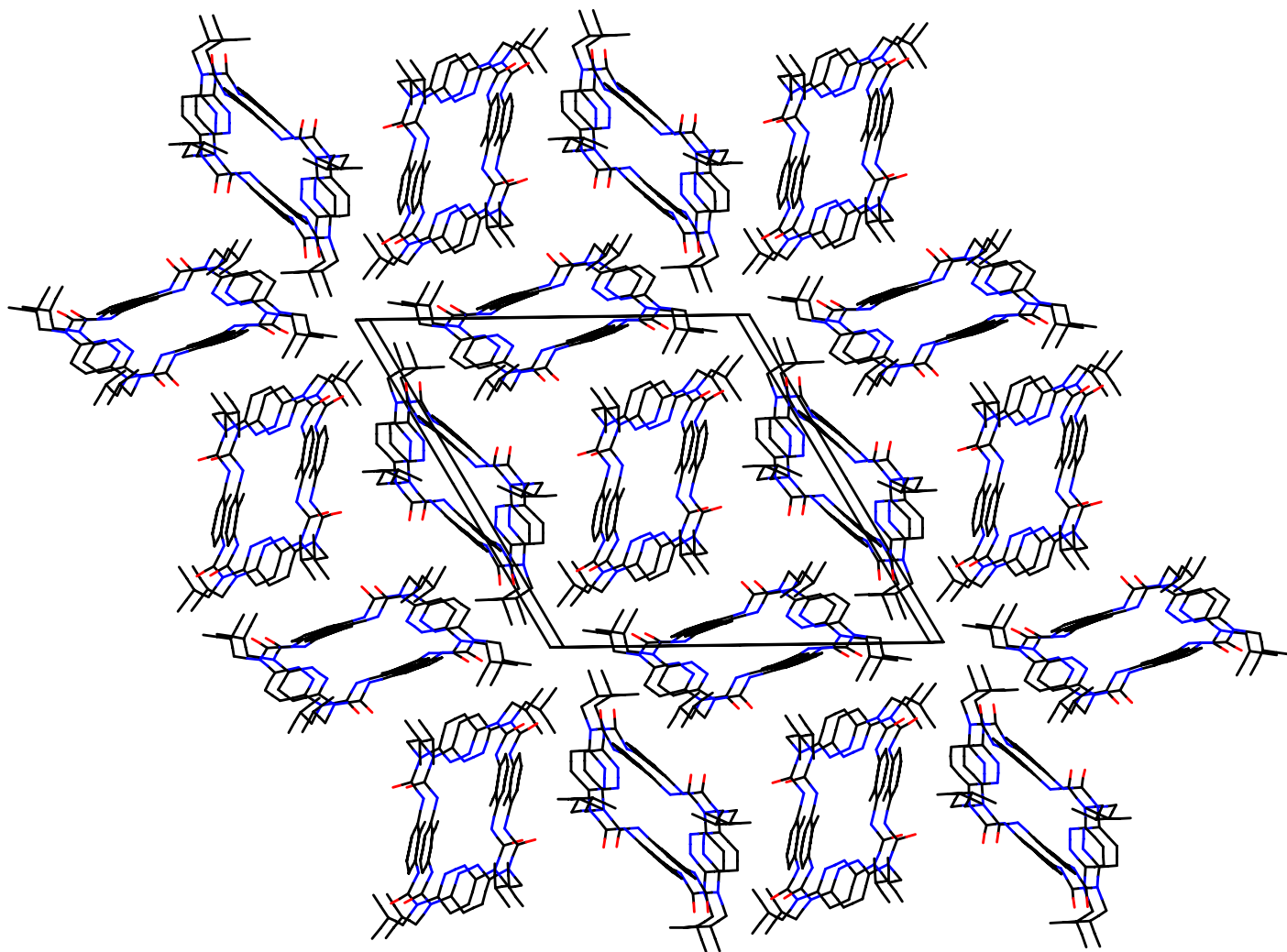


Figure S11. Crystal packing of macrocycle **3c**.

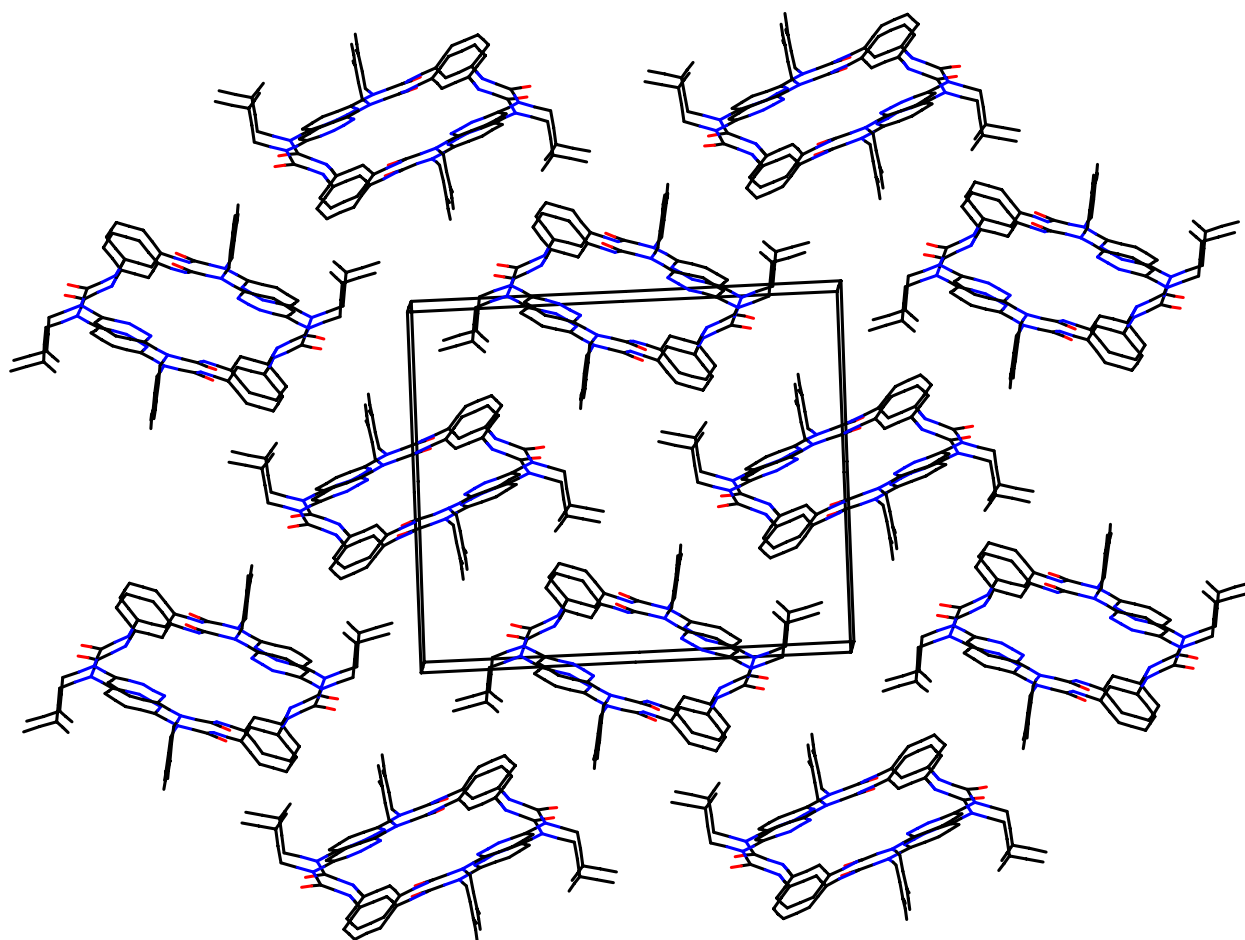


Figure S12. Crystal packing of macrocycle 4.

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