Supplementary Material (ESI) for Chemical Communications

Supplementary Information

Highly Stable Cyclic Dimers Based on Non-covalent Interactions

Valérie G. H. Lafitte,[a] Abil E. Aliev,*[a] Peter N. Horton[b], Michael B. Hursthouse[b] and Helen C. Hailes*[a]

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General Experimental Methods. Unless otherwise noted, solvents and chemicals were of analytical reagent grade from commercial suppliers and used without any further purification. THF was dried by distillation from a sodium/benzophenone suspension under a dry N_2 atmosphere. CH_2Cl_2 was dried by distillation from CaH_2 under a dry N_2 atmosphere. All moisture-sensitive reactions were performed under a nitrogen atmosphere using oven-dried glassware. Reactions were monitored by TLC on Kieselgel 60 F_{254} plates with detection by UV, or permanganate, ninhydrin (for ureas) and phosphomolybdic acid stains. Flash column chromatography was carried out using silica gel (particle size 40-63 μm). Melting points are uncorrected. 2-(6-Isocyanatohexylaminocarbonylamino)-6-methyl-4[1H]pyrimidinone was prepared as previously reported.[1] Details of the NMR measurements are given on pages S6-S7.

(2*R*,3*R*)-Bis-{6[3-(1,4-dihydro-4-oxo-6-methyl-2-pyrimidinyl)ureido]-hexylaminocarbamoyloxy}-succinic acid diethyl ester (2a). Diethyl L-tartrate (0.30 g, 1.4 mmol) was dissolved in dry amylene stabilized chloroform (15 mL) in the presence of a drop of dibutyltin dilaurate as a catalyst. 2-(6-Isocyanatohexylaminocarbonylamino)-6-methyl-4[1H]pyrimidone[1] (1.0 g, 3.5 mmol) was then added and the solution was heated at 60 ºC under a flux of nitrogen. The reaction vessel was open to the air allowing a slow evaporation of the solvent over 2-3 h. The residue was redissolved in chloroform (30 mL) and the solid removed by filtration. The filtrate was concentrated *in vacuo* until approximately 10 mL remained and 0.20 g of silica was added to the solution together with a drop of the tin catalyst. The mixture was heated at reflux for a further 2 h. The solution was then filtered to remove the silica and the filtrate was concentrated *in vacuo*. The crude product was then purified using flash silica gel chromatography ($CHCl_3$/MeOH, 7:1) to afford compound ***2a*** as a glassy solid (220 mg, 30%), Mp 142–143 ºC ($CHCl_3$); $[\alpha]^{23}_D$ +346 (*c* 0.075, $CHCl_3$); ν_{max}(KBr) 3333, 3220, 2925, 2850, 1738, 1700,

1669 cm^{-1}; *m/z* (ES+) 793 (MH^+, 100%), 815 (MNa^+, 40%); *m/z* (+FAB) 1586 ($[2M + H]^+$, 1%), 793 (MH^+, 100%); *m/z* HRMS (+FAB) calcd for $C_{34}H_{53}N_{10}O_{12}$ (MH^+) 793.38442, found 793.383603.

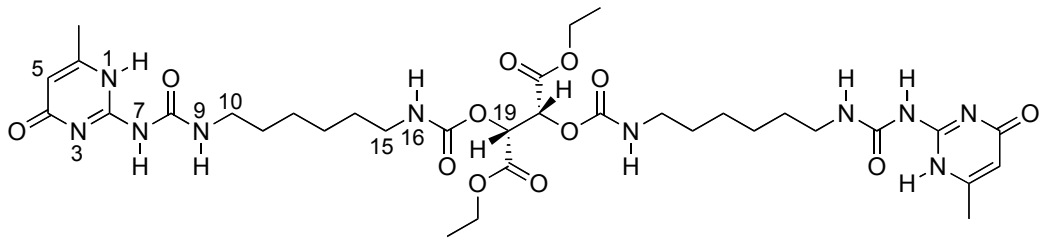


Figure 1: 4[1H]-pyrimidinone (4-keto) tautomer of **2a**

^{1}H NMR δ (500 MHz; $CDCl_3$) **4-keto tautomer** (Figure 1): 13.41 (1H, s, 1-H), 11.68 (1H, s, 7-H), 10.21 (1H, dd, *J* 7.3, 4.5, Hz, 9-H), 7.68 (1H, dd, *J* 7.1, 4.7, Hz, 16-H), 6.41 (1H, s, 5-H), 5.92 (1H, s, 19-H), 4.34 (1H, dq, *J* 10.7, 7.2 Hz, COO*CH*H), 4.19 (1H, dq, *J* 10.7, 7.2 Hz, COO*C*H*H*), 3.68 (1H, ddt, *J* 13.6, 7.3, ~4 Hz, 10-C*H*H), 3.40 (1H, ddt, *J* 14.2, 7.1, ~4 Hz, 15-C*H*H), 2.86 (1H, dddd, *J* 14.2, 11, 4.7, ~3 Hz, 15-CH*H*), 2.78 (1H, dddd, *J* 13.6, 11.5, 4.5, ~4 Hz, 10-CH*H*), 2.23 (3H, s, 6-CH_3), 1.72 (1H, m, 11-C*H*H), 1.66 (1H, m, 14-C*H*H), 1.51 (2H, m, 12-C*H*H, 13-C*H*H), 1.35 (1H, m, 11-CH*H*), 1.30 (1H, m, 14-CH*H*), 1.26 (3H, t, *J* 7.2 Hz, $COOCH_2CH_3$) 1.12 (2H, m, 12-CH*H*, 13-CH*H*); ^{13}C NMR δ (125 MHz; $CDCl_3$) **4-keto tautomer**: 174.0 (C-4), 167.4 (COOEt), 156.5 (C-8), 155.0 (C-17), 154.5 (C-2), 148.6 (C-6), 107.0 (C-5), 71.1 (C-19), 61.7 ($COOCH_2$), 38.4 (C-10), 37.1 (C-15), 28.9 (C-11), 28.7 (C-14), 22.8 (C-12, C-13), 18.2 (6-CH_3), 14.0 ($COOCH_2CH_3$); ^{13}C CPMAS NMR δ (75 MHz) **4-keto tautomer**: 174.4 (C-4), 168.0 (COOEt), 156.6 (C-8), 155.0 (C-17, C-2), 149.3 (C-6), 107.5 (C-5), 71.6 (C-19), 62.2 ($COOCH_2$), 37.8 (C-10, C-15), 29.8 (C-11, C-14), 23.7 (C-12, C-13), 18.4 (6-CH_3), 14.2 ($COOCH_2CH_3$); ^{15}N NMR δ (51 MHz; $CDCl_3$) **4-keto tautomer**: -299.2 (N-16), -284.1 (N-9), -266.5 (N-7), -246.3 (N-1), -172.2 (N-3); ^{15}N CPMAS NMR δ (30 MHz) **4-keto tautomer**: -299.0 (N-16), -283.8 (N-9), -266.4 (N-7), -245.9 (N-1), -172.1 (N-3).

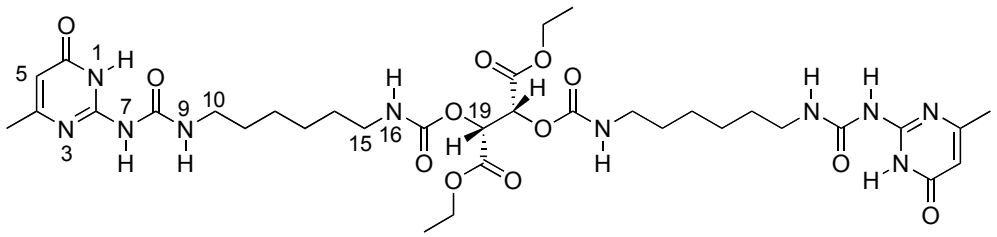
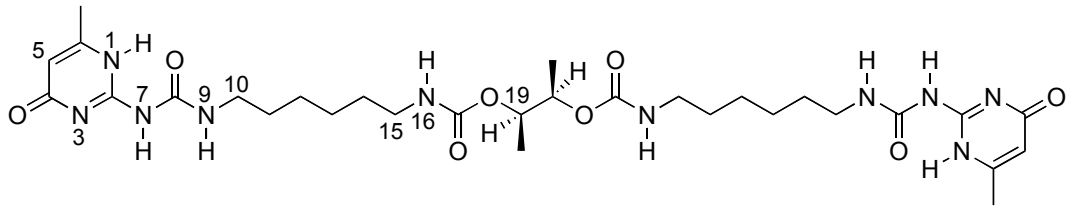


Figure 2: The 6[1H]-pyrimidinone (6-keto) tautomer of **2a** which predominates in DMSO-d_6 solution.

^{1}H NMR δ (500 MHz; DMSO-d_6) **6-keto tautomer** (Figure 2): 11.4 (1H, bs, 1-H), 9.75 (1H, bs, 7-H), 7.43 (1H, t, *J* 5.6 Hz, 9-H), 7.48 (1H, bs, 16-H), 5.77 (1H, s, 5-H), 5.44 (1H, s, 19-H), 4.17 (1H, dq, *J* 10.7, 7.1 Hz, COO*CH*H), 4.07 (1H, dq, *J* 10.7, 7.1 Hz, COO*C*H*H*), 3.10 (2H, dt, *J* 5.6, 7 Hz, 10-H), 2.96 (1H, m, 15-C*H*H), 2.93 (1H, m, 15-CH*H*), 2.10 (3H, s, 4-CH_3), 1.41 (2H, m, 11-CH_2), 1.37 (2H, m, 14-CH_2), 1.23 (4H, m, 12-CH_2, 13-CH_2), 1.13 (3H, t, *J* 7.1 Hz, $COOCH_2CH_3$); ^{13}C NMR δ (125 MHz; DMSO-d_6) **6-keto tautomer**: 166.8 (COOEt), 164.8 (C-4), 161.9 (C-6), 155.0 (C-8), 154.8 (C-17), 151.6 (C-2), 104.6 (C-5), 71.0 (C-19), 61.6 ($COOCH_2$), 39.2 (C-10), 38.7 (C-15), 29.2 (C-11, C-14), 26.0 (C-12), 25.8 (C-13), 23.2 (4-CH_3), 14.0 ($COOCH_2CH_3$); ^{15}N NMR δ (51 MHz; DMSO-d_6) **6-keto tautomer**: -296.2 (N-16), -285.8 (N-9), -174.8 (N-3).

(2*S*,3*S*)-Bis-{6[3-(1,4-dihydro-4-oxo-6-methyl-2-pyrimidinyl)ureido]-hexylaminocarbamoyloxy}-succinic acid diethyl ester (2b). The reaction was carried as for compound **2a** but using diethyl D-tartrate to give ***2b*** in an identical yield, $[\alpha]^{23}_D$ -350 (*c* 0.075, $CHCl_3$); *m/z* HRMS (+FAB) calcd for $C_{34}H_{53}N_{10}O_{12}$ (MH^+) 793.38442, found 793.385137.

(1*R*,2*R*)-{6-[3-(6-Methyl-4-oxo-1,4-dihydro-pyrimidin-2-yl)-ureido]-hexyl}-carbamic acid 1-methyl-2-{6-[3-(6-methyl-4-oxo-1,4-dihydro-pyrimidin-2-yl)-ureido]-hexylcarbamoyloxy}-propyl ester (3a).

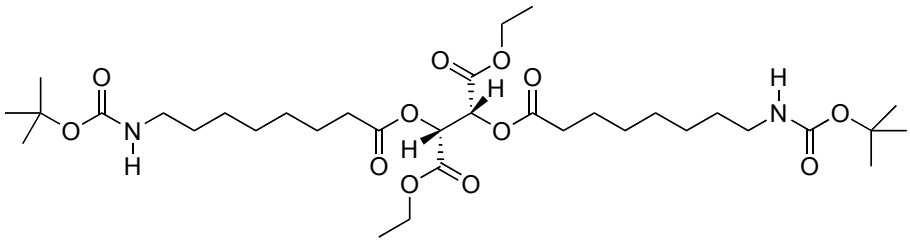


The same procedure was used as for compound **2a** above but using (2*R*,3*R*)-butanediol rather than diethyl tartrate. Compound ***3a*** was isolated in 25% yield, Mp 122–124 ºC ($CHCl_3$); $[\alpha]^{21}_D$ +217 (*c* 0.34, $CHCl_3$); ν_{max} (KBr) 3338, 3220, 3035, 2935, 2856, 1701, 16662, 1582 cm^{-1}; ^1H NMR δ (400 MHz; $CDCl_3$) **4-keto tautomer**: 13.51 (1H, s, 1-H), 11.67 (1H, s, N*H*CONHCH$_2$), 10.13 (1H, dd, *J* 6.7, 4.8 Hz, NHCON*H*CH$_2$), 7.13 (1H, t, *J* 5.0 Hz, NHCOO), 6.08 (1H, s, 5-H), 5.20 (1H, q, *J* 6.6 Hz, C*H*CH$_3$), 3.63 (1H, m, 10-C*H*H), 3.33 (1H, m, 15-C*H*H), 2.96 (2H, m, 10-CH*H*, 15-CH*H*), 2.24 (3H, s, 6-CH$_3$), 1.1-1.8 (8H, m, 11-CH$_2$, 12-CH$_2$, 13-CH$_2$, 14-CH$_2$), 1.33 (3H, d, *J* 6.6 Hz, CHC*H*$_3$); ^{13}C NMR δ (125 MHz, $CDCl_3$) **4-keto tautomer**: 173.7 (C-4), 156.5 (C-8), 156.4 (C-18), 154.7 (C-2), 148.4 (C-6), 106.7 (C-5), 71.5 (*C*HCH$_3$), 39.0 (C-10), 37.5 (C-15), 29.0 (C-11), 28.5 (C-14), 23.4 (C-12), 22.7 (C-13), 18.8 (CH*C*H$_3$), 18.1 (6-CH$_3$); *m/z* HRMS calcd for $C_{30}H_{48}N_{10}O_8Na$ (MNa^+) 699.35488, found 699.35678.

8-*tert*-Butoxycarbonylamino-octanoic acid[2] To a solution of aminocaprilic acid (1.50 g, 9.40 mmol) in water (5 mL) and CH_2Cl_2 (5 mL) was added sodium hydroxide (0.752 g, 18.8 mmol). The solution was cooled to 0 ºC and a solution of Boc_2O (2.05 g, 9.4 mmol) in CH_2Cl_2 (10 mL) was added slowly, then the reaction was stirred at r.t. for 16 h. The mixture was acidified using conc. HCl, the organic layer separated and the aqueous phase was extracted with CH_2Cl_2 (2 × 20 mL). The organic phases were combined and dried ($MgSO_4$) and the solvent evaporated *in vacuo*. The crude material was purified

using flash silica gel chromatography ($CHCl_3/CH_3CN/CH_3OH$, 18:1:1) to afford 8-*tert*-butoxycarbonylamino-octanoic acid as a solid (0.733 g, 30%), Mp 54-56 ºC (chloroform);[2] ν_{max}(KBr) 3365, 2934, 2843, 1714, 1683, 1520 cm^{-1}; ^{1}H NMR δ (400 MHz; $CDCl_3$) 11.50 (1H, bs, COOH), 4.56 (1H, bs, NHCOO), 3.07 (2H, m, C*H*$_2$NHCOO), 2.32 (2H, t, *J* 7.4 Hz, C*H*$_2$COOH), 1.60 (2H, q, *J* 7.2 Hz, C*H*$_2$CH$_2$NHCOO), 1.41 (11H, m, CH_3, CH_2), 1.30 (6H, m, CH_2); ^{13}C NMR δ (100 MHz; $CDCl_3$) 179.3 (COOH), 155.9 (NHCOO), 79.0 (*C*-CH_3), 40.4 (*C*H$_2$NHCOO), 33.9 (*C*H$_2$COOH), 29.8 (*C*H$_2$CH$_2$NHCOO), 28.9 (*C*H$_2$CH$_2$COOH, CH_2), 28.3 (CH_3), 26.5 (CH_2), 24.5 (CH_2); *m/z* HRMS calcd for $C_{13}H_{25}O_4NNa$ (MNa^+) 282.16758, found 282.16736.

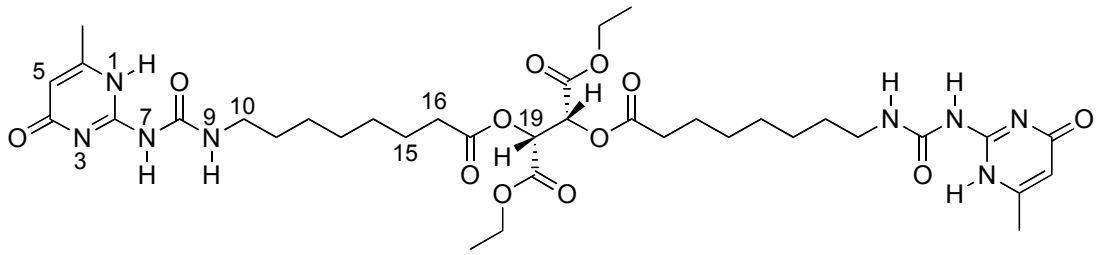
(2*R*,3*R*)-2,3-Bis-(8-tert-butoxycarbonylamino-octanoyloxy)-succinic acid diethyl ester



To a solution of 8-*tert*-butoxycarbonylamino-octanoic acid (0.30 g, 1.16 mmol) in CH_2Cl_2 (12 mL) was added at 0 ºC, DCC (0.262 g, 1.27 mmol) and DMAP (0.052 g). The solution was stirred at 0 ºC for 1 h and diethyl L-tartrate (0.103 g, 0.50 mmol) in CH_2Cl_2 (3 mL) was added. The resulting solution was stirred at r.t. for 16 h. The solution was filtered and the filtrate evaporated *in vacuo*. The residue was washed with water (10 mL) and saturated sodium chloride solution (10 mL) and the organic phase was dried ($MgSO_4$). The solvent was evaporated *in vacuo* and the crude material purified using flash silica gel chromatography ($CHCl_3$/MeOH, 10:1) to give compound the *title compound* as an oil (0.140 g, 50%). ν_{max}(KBr) 3365, 2930, 1858, 1755, 1697, 1529 cm^{-1}; ^{1}H NMR δ (300 MHz; $CDCl_3$) 4.55 (1H, bs, NHCOO), 4.18 (2H, q, *J* 7.1 Hz, COOC*H*$_2$CH$_3$), 3.01 (2H, q, *J* 6.4 Hz, C*H*$_2$NHCOO), 2.42 (2H, q, *J* 8.1 Hz, CH_2COO), 1.60 (2H, m, C*H*$_2$CH$_2$COO), 1.41-1.42 (12H, m, C*H*$_2$CH$_2$NHCOO, C-CH_3), 1.28 (6H,

m, C*H*$_2$CH$_2$CH$_2$COO, CH$_2$), 1.22 (3H, t, *J* 7.1 Hz, CH$_2$C*H*$_3$); ^{13}C NMR δ (75 MHz; CDCl$_3$) 172.4 (*C*OOCH), 165.9 (*C*OOCH$_2$CH$_3$), 156.0 (NHCOO), 79.0 (*C*-CH$_3$), 70.6 (CH), 62.1 (*C*H$_2$CH$_3$), 40.5 (CH$_2$NH), 33.6 (*C*H$_2$COOCH), 30.0 (*C*H$_2$CH$_2$NHCOO), 28.8 (*C*H$_2$CH$_2$CH$_2$COO), 28.4 (C-*C*H$_3$), 26.5 (CH$_2$), 24.6 (CH$_2$), 14.1 (CH$_2$*C*H$_3$); *m/z* (ES+) 689.7 (MH$^+$, 100%), 711.7 (MNa$^+$, 20%); *m/z* HRMS calcd for $C_{34}H_{60}O_{12}N_2Na$ (MNa$^+$) 711.40385, found 711.4048.

(2*R*,3*R*)-2,3-Bis-{8-[3-(6-methyl-4-oxo-1,4-dihydro-pyrimidin-2-yl)-ureido]-octanoyloxy}-succinic acid diethyl ester (4a).



(2*R*,3*R*)-2,3-Bis-(8-tert-butoxycarbonylamino-octanoyloxy)-succinic acid diethyl ester (0.131 g, 0.190 mmol) was dissolved in CH_2Cl_2 (10 mL) and TFA (10 mL). The solution was stirred at r.t. for 2 h and the solvents were evaporated *in vacuo*. The remaining salt was dried under reduced pressure for 16 h (0.121 g, 0.17 mmol) and then dissolved in THF (15 mL) and triethylamine (0.2 mL). To this solution was added diethyl L-tartrate (0.192 g, 1.0 mmol) and the solution was heated at reflux for 16 h. The solid was filtered and the filtrate evaporated under reduced pressure. The residue was purified using flash silica gel chromatography ($CHCl_3$/MeOH, 10:1) to give ***4a*** as a solid (0.107 g, 80%), Mp 100–102 ºC ($CHCl_3$); ν_{max}(KBr) 3478, 3415, 3227, 2935, 2856, 1751, 1701, 1666, 1589, 1521 cm^{-1}; ^{1}H NMR δ (500 MHz; $CDCl_3$) **4-keto tautomer**: 13.12 (1H, s, 1-H), 11.81 (1H, bs, 7-H), 10.15 (1H, s, 9-H), 5.80 (1H, s, 5-H), 5.69 (1H, s, CH), 4.22 (1H, dq, *J* 10.7, 7.1 Hz, COO*CH*H), 4.20 (1H, dq, *J* 10.7, 7.1 Hz, COO*CH*H), 3.21 (2H, bs, NHCONHC*H*$_2$), 2.42 (1H, m, C*H*HCOO), 2.38 (1H, m, CH*H*COO), 2.23 (3H, s, CH$_3$), 1.63 (2H, bm, C*H*$_2$CH$_2$COO), 1.58 (2H, bm, C*H*$_2$CH$_2$NHCONH), 1.33 (6H, bm, CH$_2$), 1.25

(3H, t, *J* 7.2 Hz, $COOCH_2CH_3$); ^{13}C NMR δ (125 MHz; $CDCl_3$) **4-keto tautomer**: 172.9 (C-4), 172.4 (C-17), 165.9 ($COOCH_2CH_3$), 156.5 (C-8), 154.7 (C-2), 148.3 (C-6), 106.6 (C-5), 70.6 (C-19), 62.2 ($COOCH_2CH_3$), 39.9 (C-10), 33.8 (C-16), 29.4 (C-11), 28.9 (C-14), 28.8 (C-13), 26.7 (C-12), 24.7 (C-15), 18.9 (6-CH_3), 14.1 (CH_2CH_3); ^1H NMR δ (500 MHz; DMSO-d_6) **6-keto tautomer** (numbering of protons and carbons is similar to that used in Figure 2): 11.4 (1H, bs, 1-H), 9.7 (1H, bs, 7-H), 7.41 (1H, bs, 9-H), 5.76 (1H, s, 5-H), 5.63 (1H, s, 19-H), 4.15 (1H, dq, *J* 10.7, 7.1 Hz, COO*CH*H), 4.10 (1H, dq, *J* 10.7, 7.1 Hz, COO*C*H*H*), 3.10 (2H, dt, *J* 5.7, 7 Hz, 10-H), 2.37 (1H, dt, *J* 15.9, 7.3 Hz, C*H*HCOO), 2.34 (1H, dt, *J* 15.9, 7.1 Hz, CH*H*COO), 2.08 (3H, s, 4-CH_3), 1.50 (2H, m, 15-CH_2), 1.41 (2H, m, 11-CH_2), 1.37 (2H, m, 14-CH_2), 1.25 (6H, m, 12-CH_2, 13-CH_2, 14-CH_2), 1.13 (3H, t, *J* 7.1 Hz, $COOCH_2CH_3$); ^{13}C NMR δ (125 MHz; DMSO-d_6) **6-keto tautomer**: 172.0 (C-17), 165.7 (COOEt), 164.9 (C-4), 161.8 (C-6), 154.9 (C-8), 151.5 (C-2), 104.7 (C-5), 70.4 (C-19), 62.1 ($COOCH_2$), 39.1 (C-10), 29.2 (C-11), 28.4 (C-14), 28.3 (C-13), 26.2 (C-12), 24.4 (C-15), 23.2 (4-CH_3), 14.0 ($COOCH_2CH_3$); *m/z* (ES+) 791.6 (MH^+, 100%), 813.5 (MNa^+, 40%); *m/z* HRMS calcd for $C_{36}H_{54}O_{12}N_8Na$ (MNa^+) 813.37534, found 813.37343.

NMR Measurements. Solution ^1H and ^{13}C NMR spectra were recorded on Bruker NMR spectrometers AMX300, AMX400 and AVANCE500. ^1H and ^{13}C chemical shifts are given relative to TMS. ^{15}N chemical shifts are given relative to $MeNO_2$. All spectra were recorded at 25ºC. Diffusion, selective NOE/ROE and 2D experiments were measured using AVANCE500, equipped with z-gradient facilities.

^1H NMR spectra of very dilute solutions were measured using solvent suppression. Full details of diffusion NMR experiments (all at 25ºC) were described previously.[3] A convection compensated pulse sequence was used in this work.[4] The mean deviation was in the range ±0.05 - ±0.08 × 10^{-10} m^2 s^{-1} for the diffusion coefficient (*D*) measurements. In order to account for viscosity changes, the solvent

corrected D values are reported in the text: $D=D_{\text{meas}} \times (D^{sol}_{pure} / D^{sol}_{meas})$, where D_{meas} and D^{sol}_{meas} are the measured values for the solute and the residual solvent peak ($CHCl_3$), respectively, and D^{sol}_{pure} is the diffusion coefficient measured for the residual $CHCl_3$ in pure $CDCl_3$ ($D^{sol}_{pure} = 2.366 \times 10^{-9}$ m^2 s^{-1}).

Solid-state ^{13}C and ^{15}N spectra were recorded at 75.5 MHz and 30.1 MHz, respectively, using a standard 7 mm double-resonance magic-angle spinning (MAS) probe on MSL300 (Bruker). Samples in zirconia rotors of 7 mm external diameter were spun at 5 kHz with stability better than ±3 Hz. Spectra were recorded using cross-polarization (CP), MAS and high-power ^1H decoupling. Dipolar-dephased CPMAS NMR spectra were also acquired.[5] ^{13}C chemical shifts are reported relative to TMS and solid adamantane (29.47 ppm, CH) was used as external chemical shift reference. ^{15}N chemical shifts are given relative to $MeNO_2$ and solid NH_4NO_3 (-358.4 ppm, NH_4) was used as external chemical shift reference.

X-ray Crystallography methods: Although it was only possible to grow small crystals of stacked plates, they were of sufficient quality for obtaining the single crystal XRD data to help confirm the core hydrogen bonding arrangement. Suitable crystals (from dichloroethane/heptane) were selected and data collected on a Bruker Nonius KappaCCD Area Detector at the window of a Bruker Nonius FR591 rotating anode ($\lambda_{\text{Mo-K}\alpha}$ = 0.71073 Å) driven by COLLECT[6] and DENZOO[7] software at 120 K. The structures were determined in SHELXS-97[8] and refined using SHELXL-97[9]. All non-hydrogen atoms were refined anisotropically with hydrogen atoms included in idealized positions with thermal parameters riding on those of the parent atom. The crystal contained unrefineable solvent which was removed by using the SQUEEZE[10] program, and the ethyl ester groups were thermally disordered.

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