Supplementary Material (ESI) for Chemical Communications

Supporting Information

"The Influence of Sterics on the Formation of Polar 1-D Hydrogen-Bonded Networks"

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4-Chloro-*N*,*N*′-bis[(*S*)-2-chloro-1-methylethyl]pyridine-2,6-dicarboxamide. A mixture of (S)-(+)-2-amino-1-propanol (1.80 g, 1.90 ml, 24.40 mmol) and methyl 4-chloropyridine-2,6-dicarboxylate (2.30 g, 10.00 mmol) was heated at 90 °C for 2 h. The mixture was diluted with $CHCl_3$ (50 ml) followed by the addition of $SOCl_2$ (7.14 g, 4.38 ml, 60.00 mmol). The reaction was heated at reflux for 1 h and concentrated under vacuum. The residue was chromatographed over silica using a 95:5 CH_2Cl_2:EtOAc mobile phase. Product was isolated as a white crystalline solid (3.06 g, 87 %); mp 165-167 °C (CH_2Cl_2); ^{1}H NMR ($CDCl_3$) δ 1.43 (d, *J* = 6.7 Hz, 6H), 3.73 (dd, *J* = 3.2, 11.1 Hz, 2H), 3.86 (dd, *J* = 4.0, 11.1 Hz, 2H), 4.59 (m, 2H), 7.91 (d, *J* = 8.1 Hz, 2H), 8.33 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 18.03, 45.44, 49.60, 125.41, 148.03, 149.87, 161.48; HRMS *m/z* $[Na]^+$ 374.0206 (calcd for $C_{13}H_{16}Cl_3N_3O_2Na^+$ 374.0200).

4-Chloro-2,6-bis-[(*S*)-4-methyloxazolin-2-yl)]pyridine. To a suspension of NaH (1.38 g, 34.47 mmol) in THF (76 ml) was slowly added a solution of 4-chloro-*N*,*N*′-bis[(*S*)-2-chloro-1-methylethyl]pyridine-2,6-dicarboxamide (3.04 g, 8.62 mmol) in THF (10 ml). The reaction was stirred for 1 h, filtered, and concentrated. Residue was chromatographed over silica using 50:50 CH_2Cl_2:EtOAc. Product was isolated as a white solid (1.71 g, 71 %); mp 112-114 °C (CH_2Cl_2); ^{1}H NMR ($CDCl_3$) δ 1.39 (d, *J* = 6.6 Hz, 6H), 4.09 (t, *J* = 8.1 Hz, 2H), 4.46 (m, 2H), 4.63 (t, *J* = 8.3 Hz, 2H), 8.21 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 21.27, 62.32, 74.97, 125.83, 145.48, 148.08, 161.48; HRMS *m/z* $[Na]^+$ 302.0682 (calcd for $C_{13}H_{14}Cl_1N_3O_2Na^+$ 302.0667).

4-Amino-2,6-bis-[(*S*)-4-methyloxazolin-2-yl)]pyridine. To a solution of 4-chloro-2,6-bis-[(*S*)-4-methyloxazolin-2-yl)]pyridine (1.71 g, 6.12 mmol) in DMF (30 ml) was added NaN_3 (3.98 g, 61.20 mmol). The reaction was heated to 65 °C for 3 h. The DMF was removed under vacuum and the residue dissolved in CH_2Cl_2, filtered and re-concentrated. The resulting residue was dissolved in isopropanol (31 ml) to which $NaBH_4$ (0.23 g, 6.16 mmol) was added. The reaction was heated to reflux for 2 h, cooled to r.t., and diluted with 0.15 M NaOH (50 ml). The resulting mixture was washed with CH_2Cl_2 (3 x 50 ml) and the organic layers combined, dried over $MgSO_4$, filtered, and concentrated to a yellow crystalline solid (1.58 g, 99 %); mp (dec) 250 °C (CH_2Cl_2); ^{1}H NMR (DMSO) δ 1.23 (d, *J* = 6.6 Hz, 6H), 3.91 (t, *J* = 8.0 Hz, 2H), 4.28 (m, 2H),

4.47 (t, *J* = 8.3 Hz, 2H), 6.57 (s, 2H), 7.22 (s, 2H); ^{13}C NMR (DMSO) δ 21.25, 61.40, 73.55, 109.97, 146.68, 155.14, 162.05; HRMS (EI) *m/z* 260.1273 (calcd for $C_{13}H_{16}N_4O_2^+$ 260.1268).

4-Chloro-*N*,*N*′-bis[(*S*)-2-chloro-1-isopropylethyl]pyridine-2,6-dicarboxamide. A mixture of (*S*)-(+)-2-amino-3-methyl-1-butanol (0.49 g, 4.76 mmol) and dimethyl 4-chloropyridine-2,6-dicarboxylate (0.46 g, 2.00 mmol) was heated at 100 °C for 2 h. The mixture was diluted with $CHCl_3$ (10 ml) followed by the addition of $SOCl_2$ (1.43 g, 875 µl, 12.00 mmol). The reaction was heated to reflux for 2 h and concentrated under vacuum. The residue was chromatographed over silica using a 98:2 CH_2Cl_2:EtOAc mobile phase. Product was isolated as a white crystalline solid (0.79 g, 96 %); mp 130-132 °C (CH_2Cl_2); ^{1}H NMR ($CDCl_3$) δ 1.03 (d, 6.8 Hz, 6H), 1.07 (d, *J* = 6.7 Hz, 6H), 2.11 (oct, *J* = 6.8 Hz, 2H), 3.77 (dd, *J* = 3.9, 11.4 Hz, 2H), 3.89 (dd, *J* = 3.4, 11.4 Hz, 2H), 4.17 (m, 2H), 7.92 (d, *J* = 9.4, 2H), 8.35 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 18.81, 19.35, 29.65, 46.82, 54.97, 125.56, 148.14, 149.91, 161.91; HRMS *m/z* $[Na]^+$ 430.0815 (calcd for $C_{17}H_{24}Cl_3N_3O_2Na^+$ 430.0826).

4-Chloro-2,6-bis-[(*S*)-4-isopropyloxazolin-2-yl)]pyridine. To a suspension of NaH (0.16 g, 4.00 mmol) in THF (2 ml) was slowly added a solution of 4-chloro-*N*,*N*′-bis[(*S*)-2-chloro-1-isopropylethyl]pyridine-2,6-dicarboxamide (0.41 g, 1.00 mmol) in THF (3 ml). The reaction was stirred for 4 h, filtered, and concentrated. Residue was chromatographed over silica using 70:30 hexanes:EtOAc. Product was isolated as a white solid (0.29 g, 86 %); mp 55-57 °C (CH_2Cl_2); ^{1}H NMR ($CDCl_3$) δ 0.91 (d, 6.8 Hz, 6H), 1.01 (d, *J* = 6.4 Hz, 6H), 1.83 (oct, *J* = 6.8 Hz, 2H), 4.11 (m, 2H), 4.20 (t, *J* = 8.8 Hz, 2H), 4.50 (t, *J* = 8.4 Hz, 2H), 8.19 (s, 2H); ^{13}C NMR ($CDCl_3$) δ 18.21, 18.88, 32.71, 71.12, 72.85, 125.73, 145.17, 148.04, 161.30; HRMS *m/z* $[Na]^+$ 358.1317 (calcd for $C_{17}H_{22}Cl_1N_3O_2Na^+$ 358.1293).

4-Amino-2,6-bis-[(*S*)-4-isopropyloxazolin-2-yl)]pyridine. To a solution of 4-chloro-2,6-bis-[(*S*)-4-isopropyloxazolin-2-yl)pyridine (0.10 g, 0.30 mmol) in DMF (1.5 ml) was added NaN_3 (0.19 g, 2.98 mmol). The reaction was heated to 65 °C for 15 h. The DMF was removed under vacuum and the residue dissolved in CH_2Cl_2, filtered and re-concentrated. The resulting residue was dissolved in isopropanol (1.0 ml) to which $NaBH_4$ (0.011 g, 0.30 mmol) was added. The

reaction was heated to reflux for 2 h, cooled to r.t., and diluted with 0.1 M NaOH (10 ml). The resulting mixture was washed with CH_2Cl_2 (3 x 10 ml) and the organic layers combined, dried over $MgSO_4$, filtered, and concentrated to a yellow crystalline solid (0.055 g, 87 %); mp dec 230 °C (CH_2Cl_2); ^{1}H NMR (DMSO) δ 0.87 (d, 6.8 Hz, 6H), 0.94 (d, *J* = 6.8 Hz, 6H), 1.74 (oct, *J* = 6.4 Hz, 2H), 4.03 (m, 2H), 4.09 (t, *J* = 8.4 Hz, 2H), 4.40 (t, *J* = 8.6 Hz, 2H), 6.45 (s, 2H), 7.27 (s, 2H); ^{13}C NMR (DMSO) δ 18.22, 18.52, 32.22, 69.82, 71.87, 110.04, 146.67, 155.12, 162.23; HRMS *m/z* $[Na]^+$ 339.1792 (calcd for $C_{17}H_{24}N_4O_2Na^+$ 339.1791).

Supplementary Material (ESI) for Chemical Communications

Crystal Data for Ampybox 1.

The data collection crystal was a clear, colorless rectangular rod. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated an orthorhombic crystal system. All work was done at 200 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure an octant of reciprocal space with a redundancy factor of 4.9, which means that 90% of the reflections were measured at least 4.9 times. A combination of phi and omega scans with a frame width of 1.0˚ was used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.042.

The structure was solved by the direct methods procedure in SHELXD(2). There are 2.5 molecules in the asymmetric unit. Two molecules are in general positions (labeled as A and B), while one molecule contains a crystallographic two-fold axis (labeled as C). The correct enantiomer was chosen based on the known chiral centers. Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4).

The hydrogen atoms bonded to the nitrogen atoms were refined isotropically. For the methyl groups, the hydrogen atoms were added at calculated positions using a riding model with U(H) = 1.5 *Ueq(bonded carbon atom). The torsion angle, which defines the orientation of the methyl group about the C-C bond, was refined. The remaining hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on all 3345 intensities and 455 variables and resulted in agreement factors of R1(F) = 0.055 and wR2(F^2) = 0.096. For the subset of data with I > 2*sigma(I),the R1(F) value is 0.038 for 2628 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.23 and -0.16 e/ Å ^{3}. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

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Crystallographic details.

Empirical formula	C13 H16 N4 O2
Formula weight	260.30
Temperature	200(2) K
Wavelength	0.71073
Crystal system	Orthorhombic
Space group	P 2_12_12
Unit cell dimensions	a = 32.0792(5) Å b = 14.4080(2) Å c = 7.20350(10) Å
Volume	3329.44(8) $Å^3$
Z	10
Calculated density	1.298 Mg/m^3
Absorption coefficient	0.091 mm^{-1}
F(000)	1380
Crystal size	0.42 x 0.17 x 0.15 mm
Theta range for data collection	2.37 to 25.01 deg.
Limiting indices	-38<=h<=37, -17<=k<=17, -8<=l<=8
Reflections collected / unique	34401 / 3345 [R(int) = 0.042]
Completeness to theta = 25.01	99.6%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3345 / 0 / 455

Goodness-of-fit on F^2	1.045
Final R indices [I>2sigma(I)]	R1 = 0.0380, wR2 = 0.0877
R indices (all data)	R1 = 0.0554, wR2 = 0.0960
Largest diff. peak and hole	0.228 and -0.161 e/Å^3

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Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for Parquette 1274. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1A)	7981(1)	5007(2)	8045(3)	35(1)
C(1B)	6013(1)	4952(2)	9344(3)	33(1)
C(1C)	5000	10000	1200(4)	31(1)
C(2A)	7665(1)	4571(2)	7016(3)	34(1)
C(2B)	5687(1)	4558(2)	8313(3)	34(1)
C(2C)	5328(1)	9614(2)	2224(3)	31(1)
C(3A)	7683(1)	4580(1)	5102(3)	29(1)
C(3B)	5703(1)	4558(1)	6406(3)	29(1)
C(3C)	5309(1)	9616(1)	4131(3)	27(1)
C(4A)	8283(1)	5432(1)	5115(3)	30(1)
C(4B)	6318(1)	5345(2)	6394(3)	29(1)
C(4C)	5645(1)	9164(1)	5231(3)	28(1)
C(5A)	8294(1)	5441(2)	7026(3)	34(1)
C(5B)	6337(1)	5347(2)	8311(3)	33(1)
C(5C)	6212(1)	8279(2)	5506(3)	41(1)
C(6A)	7358(1)	4096(2)	4002(3)	29(1)
C(6B)	5363(1)	4116(2)	5320(3)	32(1)
C(6C)	6065(1)	8623(2)	7408(3)	39(1)
C(7A)	6776(1)	3260(2)	3687(4)	43(1)
C(7B)	4777(1)	3294(2)	5062(4)	49(1)
C(7C)	5972(1)	7846(2)	8766(4)	57(1)
C(8A)	6972(1)	3464(2)	1797(4)	41(1)
C(8B)	4933(1)	3600(2)	3163(4)	48(1)
C(9A)	7114(1)	2617(2)	726(4)	53(1)
C(9B)	4989(1)	2802(2)	1806(4)	74(1)
C(10A)	8616(1)	5896(2)	4033(3)	31(1)
C(10B)	6645(1)	5818(1)	5296(3)	29(1)
C(11A)	9207(1)	6702(2)	3767(4)	52(1)
C(11B)	7194(1)	6762(2)	4982(4)	41(1)
C(12A)	9035(1)	6449(2)	1845(4)	45(1)
C(12B)	7042(1)	6422(2)	3082(4)	38(1)
C(13A)	8971(1)	7278(2)	599(4)	72(1)
C(13B)	6895(1)	7190(2)	1796(4)	49(1)
N(1A)	7979(1)	5008(2)	9918(3)	49(1)
N(1B)	6014(1)	4941(2)	11210(3)	49(1)

N(1C)	5000	10000	-677(4)	45(1)
N(2A)	7984(1)	5006(1)	4095(2)	31(1)
N(2B)	6012(1)	4945(1)	5401(2)	30(1)
N(2C)	5000	10000	5139(3)	28(1)
N(3A)	7335(1)	4057(1)	2253(3)	36(1)
N(3B)	5334(1)	4071(1)	3578(3)	40(1)
N(3C)	5684(1)	9161(1)	6971(3)	36(1)
N(4A)	8638(1)	5967(1)	2281(3)	37(1)
N(4B)	6688(1)	5800(1)	3558(3)	36(1)
O(1A)	7066(1)	3642(1)	5028(2)	40(1)
O(1B)	5056(1)	3730(1)	6362(2)	47(1)
O(1C)	5933(1)	8701(1)	4188(2)	43(1)
O(2A)	8927(1)	6268(1)	5077(3)	45(1)
O(2B)	6913(1)	6346(1)	6313(2)	41(1)

Bond lengths [A] and angles [deg] for Parquette 1274

C(1A)-N(1A)	1.349(3)
C(1A)-C(5A)	1.393(3)
C(1A)-C(2A)	1.404(3)
C(1B)-N(1B)	1.344(3)
C(1B)-C(5B)	1.399(3)
C(1B)-C(2B)	1.403(3)
C(1C)-N(1C)	1.352(4)
C(1C)-C(2C)#1	1.400(3)
C(1C)-C(2C)	1.400(3)
C(2A)-C(3A)	1.380(3)
C(2A)-H(2A)	0.9500
C(2B)-C(3B)	1.375(3)
C(2B)-H(2B)	0.9500
C(2C)-C(3C)	1.376(3)
C(2C)-H(2C)	0.9500
C(3A)-N(2A)	1.354(3)
C(3A)-C(6A)	1.483(3)
C(3B)-N(2B)	1.349(3)
C(3B)-C(6B)	1.484(3)
C(3C)-N(2C)	1.348(2)
C(3C)-C(4C)	1.486(3)
C(4A)-N(2A)	1.355(3)
C(4A)-C(5A)	1.377(3)
C(4A)-C(10A)	1.482(3)
C(4B)-N(2B)	1.345(3)
C(4B)-C(5B)	1.382(3)
C(4B)-C(10B)	1.478(3)
C(4C)-N(3C)	1.260(3)
C(4C)-O(1C)	1.365(2)
C(5A)-H(5A)	0.9500
C(5B)-H(5B)	0.9500
C(5C)-O(1C)	1.441(3)
C(5C)-C(6C)	1.532(4)
C(5C)-H(5C1)	0.9900
C(5C)-H(5C2)	0.9900
C(6A)-N(3A)	1.263(3)
C(6A)-O(1A)	1.360(3)
C(6B)-N(3B)	1.260(3)
C(6B)-O(1B)	1.358(2)
C(6C)-N(3C)	1.483(3)
C(6C)-C(7C)	1.517(3)
C(6C)-H(6C)	1.0000

C(7A)-O(1A)	1.450(3)
C(7A)-C(8A)	1.529(4)
C(7A)-H(7A1)	0.9900
C(7A)-H(7A2)	0.9900
C(7B)-O(1B)	1.439(3)
C(7B)-C(8B)	1.522(4)
C(7B)-H(7B1)	0.9900
C(7B)-H(7B2)	0.9900
C(7C)-H(7C1)	0.9800
C(7C)-H(7C2)	0.9800
C(7C)-H(7C3)	0.9800
C(8A)-N(3A)	1.481(3)
C(8A)-C(9A)	1.513(3)
C(8A)-H(8A)	1.0000
C(8B)-N(3B)	1.485(3)
C(8B)-C(9B)	1.520(3)
C(8B)-H(8B)	1.0000
C(9A)-H(9A1)	0.9800
C(9A)-H(9A2)	0.9800
C(9A)-H(9A3)	0.9800
C(9B)-H(9B1)	0.9800
C(9B)-H(9B2)	0.9800
C(9B)-H(9B3)	0.9800
C(10A)-N(4A)	1.268(3)
C(10A)-O(2A)	1.361(3)
C(10B)-N(4B)	1.261(3)
C(10B)-O(2B)	1.363(2)
C(11A)-O(2A)	1.445(3)
C(11A)-C(12A)	1.535(4)
C(11A)-H(11A)	0.9900
C(11A)-H(11B)	0.9900
C(11B)-O(2B)	1.446(3)
C(11B)-C(12B)	1.534(4)
C(11B)-H(11C)	0.9900
C(11B)-H(11D)	0.9900
C(12A)-N(4A)	1.484(3)
C(12A)-C(13A)	1.508(4)
C(12A)-H(12A)	1.0000
C(12B)-N(4B)	1.486(3)
C(12B)-C(13B)	1.518(3)
C(12B)-H(12B)	1.0000
C(13A)-H(13A)	0.9800
C(13A)-H(13B)	0.9800
C(13A)-H(13C)	0.9800
C(13B)-H(13D)	0.9800
C(13B)-H(13E)	0.9800

C(13B)-H(13F)	0.9800
N(1A)-H(1A1)	0.93(3)
N(1A)-H(1A2)	1.01(2)
N(1B)-H(1B1)	0.97(3)
N(1B)-H(1B2)	1.04(2)
N(1C)-H(1C1)	0.95(2)
N(2C)-C(3C)#1	1.348(2)

N(1A)-C(1A)-C(5A)	122.0(2)
N(1A)-C(1A)-C(2A)	121.7(2)
C(5A)-C(1A)-C(2A)	116.3(2)
N(1B)-C(1B)-C(5B)	122.4(2)
N(1B)-C(1B)-C(2B)	121.7(2)
C(5B)-C(1B)-C(2B)	115.9(2)
N(1C)-C(1C)-C(2C)#1	121.78(13)
N(1C)-C(1C)-C(2C)	121.78(13)
C(2C)#1-C(1C)-C(2C)	116.4(3)
C(3A)-C(2A)-C(1A)	119.5(2)
C(3A)-C(2A)-H(2A)	120.2
C(1A)-C(2A)-H(2A)	120.2
C(3B)-C(2B)-C(1B)	120.0(2)
C(3B)-C(2B)-H(2B)	120.0
C(1B)-C(2B)-H(2B)	120.0
C(3C)-C(2C)-C(1C)	119.5(2)
C(3C)-C(2C)-H(2C)	120.3
C(1C)-C(2C)-H(2C)	120.3
N(2A)-C(3A)-C(2A)	124.7(2)
N(2A)-C(3A)-C(6A)	115.3(2)
C(2A)-C(3A)-C(6A)	120.0(2)
N(2B)-C(3B)-C(2B)	124.3(2)
N(2B)-C(3B)-C(6B)	115.71(19)
C(2B)-C(3B)-C(6B)	120.0(2)
N(2C)-C(3C)-C(2C)	124.8(2)
N(2C)-C(3C)-C(4C)	115.2(2)
C(2C)-C(3C)-C(4C)	120.0(2)
N(2A)-C(4A)-C(5A)	124.5(2)
N(2A)-C(4A)-C(10A)	115.4(2)
C(5A)-C(4A)-C(10A)	120.1(2)
N(2B)-C(4B)-C(5B)	124.3(2)
N(2B)-C(4B)-C(10B)	115.50(19)
C(5B)-C(4B)-C(10B)	120.2(2)
N(3C)-C(4C)-O(1C)	118.6(2)
N(3C)-C(4C)-C(3C)	127.1(2)
O(1C)-C(4C)-C(3C)	114.3(2)
C(4A)-C(5A)-C(1A)	120.2(2)
C(4A)-C(5A)-H(5A)	119.9

C(1A)-C(5A)-H(5A)	119.9
C(4B)-C(5B)-C(1B)	119.9(2)
C(4B)-C(5B)-H(5B)	120.0
C(1B)-C(5B)-H(5B)	120.0
O(1C)-C(5C)-C(6C)	105.16(16)
O(1C)-C(5C)-H(5C1)	110.7
C(6C)-C(5C)-H(5C1)	110.7
O(1C)-C(5C)-H(5C2)	110.7
C(6C)-C(5C)-H(5C2)	110.7
H(5C1)-C(5C)-H(5C2)	108.8
N(3A)-C(6A)-O(1A)	118.7(2)
N(3A)-C(6A)-C(3A)	126.5(2)
O(1A)-C(6A)-C(3A)	114.8(2)
N(3B)-C(6B)-O(1B)	118.3(2)
N(3B)-C(6B)-C(3B)	127.0(2)
O(1B)-C(6B)-C(3B)	114.61(19)
N(3C)-C(6C)-C(7C)	111.2(2)
N(3C)-C(6C)-C(5C)	103.48(18)
C(7C)-C(6C)-C(5C)	113.5(2)
N(3C)-C(6C)-H(6C)	109.5
C(7C)-C(6C)-H(6C)	109.5
C(5C)-C(6C)-H(6C)	109.5
O(1A)-C(7A)-C(8A)	104.84(17)
O(1A)-C(7A)-H(7A1)	110.8
C(8A)-C(7A)-H(7A1)	110.8
O(1A)-C(7A)-H(7A2)	110.8
C(8A)-C(7A)-H(7A2)	110.8
H(7A1)-C(7A)-H(7A2)	108.9
O(1B)-C(7B)-C(8B)	104.74(17)
O(1B)-C(7B)-H(7B1)	110.8
C(8B)-C(7B)-H(7B1)	110.8
O(1B)-C(7B)-H(7B2)	110.8
C(8B)-C(7B)-H(7B2)	110.8
H(7B1)-C(7B)-H(7B2)	108.9
C(6C)-C(7C)-H(7C1)	109.5
C(6C)-C(7C)-H(7C2)	109.5
H(7C1)-C(7C)-H(7C2)	109.5
C(6C)-C(7C)-H(7C3)	109.5
H(7C1)-C(7C)-H(7C3)	109.5
H(7C2)-C(7C)-H(7C3)	109.5
N(3A)-C(8A)-C(9A)	110.07(19)
N(3A)-C(8A)-C(7A)	103.7(2)
C(9A)-C(8A)-C(7A)	115.0(2)
N(3A)-C(8A)-H(8A)	109.3
C(9A)-C(8A)-H(8A)	109.3
C(7A)-C(8A)-H(8A)	109.3

N(3B)-C(8B)-C(9B)	111.9(2)
N(3B)-C(8B)-C(7B)	103.6(2)
C(9B)-C(8B)-C(7B)	113.4(2)
N(3B)-C(8B)-H(8B)	109.3
C(9B)-C(8B)-H(8B)	109.3
C(7B)-C(8B)-H(8B)	109.3
C(8A)-C(9A)-H(9A1)	109.5
C(8A)-C(9A)-H(9A2)	109.5
H(9A1)-C(9A)-H(9A2)	109.5
C(8A)-C(9A)-H(9A3)	109.5
H(9A1)-C(9A)-H(9A3)	109.5
H(9A2)-C(9A)-H(9A3)	109.5
C(8B)-C(9B)-H(9B1)	109.5
C(8B)-C(9B)-H(9B2)	109.5
H(9B1)-C(9B)-H(9B2)	109.5
C(8B)-C(9B)-H(9B3)	109.5
H(9B1)-C(9B)-H(9B3)	109.5
H(9B2)-C(9B)-H(9B3)	109.5
N(4A)-C(10A)-O(2A)	118.5(2)
N(4A)-C(10A)-C(4A)	126.9(2)
O(2A)-C(10A)-C(4A)	114.6(2)
N(4B)-C(10B)-O(2B)	118.4(2)
N(4B)-C(10B)-C(4B)	126.9(2)
O(2B)-C(10B)-C(4B)	114.68(19)
O(2A)-C(11A)-C(12A)	105.18(18)
O(2A)-C(11A)-H(11A)	110.7
C(12A)-C(11A)-H(11A)	110.7
O(2A)-C(11A)-H(11B)	110.7
C(12A)-C(11A)-H(11B)	110.7
H(11A)-C(11A)-H(11B)	108.8
O(2B)-C(11B)-C(12B)	105.13(16)
O(2B)-C(11B)-H(11C)	110.7
C(12B)-C(11B)-H(11C)	110.7
O(2B)-C(11B)-H(11D)	110.7
C(12B)-C(11B)-H(11D)	110.7
H(11C)-C(11B)-H(11D)	108.8
N(4A)-C(12A)-C(13A)	112.4(2)
N(4A)-C(12A)-C(11A)	103.3(2)
C(13A)-C(12A)-C(11A)	113.4(2)
N(4A)-C(12A)-H(12A)	109.2
C(13A)-C(12A)-H(12A)	109.2
C(11A)-C(12A)-H(12A)	109.2
N(4B)-C(12B)-C(13B)	110.04(19)
N(4B)-C(12B)-C(11B)	103.29(18)
C(13B)-C(12B)-C(11B)	114.3(2)
N(4B)-C(12B)-H(12B)	109.7

C(13B)-C(12B)-H(12B)	109.7
C(11B)-C(12B)-H(12B)	109.7
C(12A)-C(13A)-H(13A)	109.5
C(12A)-C(13A)-H(13B)	109.5
H(13A)-C(13A)-H(13B)	109.5
C(12A)-C(13A)-H(13C)	109.5
H(13A)-C(13A)-H(13C)	109.5
H(13B)-C(13A)-H(13C)	109.5
C(12B)-C(13B)-H(13D)	109.5
C(12B)-C(13B)-H(13E)	109.5
H(13D)-C(13B)-H(13E)	109.5
C(12B)-C(13B)-H(13F)	109.5
H(13D)-C(13B)-H(13F)	109.5
H(13E)-C(13B)-H(13F)	109.5
C(1A)-N(1A)-H(1A1)	121.3(17)
C(1A)-N(1A)-H(1A2)	120.9(14)
H(1A1)-N(1A)-H(1A2)	118(2)
C(1B)-N(1B)-H(1B1)	120.5(17)
C(1B)-N(1B)-H(1B2)	124.0(14)
H(1B1)-N(1B)-H(1B2)	115(2)
C(1C)-N(1C)-H(1C1)	122.6(16)
C(3A)-N(2A)-C(4A)	114.75(18)
C(4B)-N(2B)-C(3B)	115.42(18)
C(3C)#1-N(2C)-C(3C)	114.8(3)
C(6A)-N(3A)-C(8A)	107.1(2)
C(6B)-N(3B)-C(8B)	106.8(2)
C(4C)-N(3C)-C(6C)	107.19(19)
C(10A)-N(4A)-C(12A)	107.2(2)
C(10B)-N(4B)-C(12B)	107.53(19)
C(6A)-O(1A)-C(7A)	105.22(18)
C(6B)-O(1B)-C(7B)	105.69(17)
C(4C)-O(1C)-C(5C)	105.37(18)
C(10A)-O(2A)-C(11A)	105.4(2)
C(10B)-O(2B)-C(11B)	105.61(17)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+2,z

Anisotropic displacement parameters (A^2 x 10^3) for Parquette 1274.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1A)	42(1)	32(1)	30(1)	3(1)	-3(1)	-1(1)
C(1B)	35(1)	35(1)	30(1)	1(1)	1(1)	1(1)
C(1C)	36(2)	37(2)	20(2)	0	0	-1(2)
C(2A)	40(1)	32(1)	29(1)	5(1)	4(1)	-1(1)
C(2B)	37(1)	38(1)	27(1)	5(1)	3(1)	-1(1)
C(2C)	31(1)	36(1)	25(1)	-2(1)	3(1)	2(1)
C(3A)	30(1)	26(1)	32(1)	0(1)	0(1)	1(1)
C(3B)	29(1)	29(1)	29(1)	2(1)	-1(1)	0(1)
C(3C)	28(1)	29(1)	24(1)	-1(1)	1(1)	1(1)
C(4A)	31(1)	26(1)	31(1)	1(1)	-3(1)	0(1)
C(4B)	29(1)	29(1)	29(1)	-1(1)	-1(1)	0(1)
C(4C)	26(1)	29(1)	30(1)	-5(1)	2(1)	4(1)
C(5A)	41(1)	34(1)	27(1)	-1(1)	-4(1)	-3(1)
C(5B)	36(1)	36(1)	27(1)	-3(1)	-4(1)	-1(1)
C(5C)	33(1)	38(1)	51(2)	2(1)	-4(1)	11(1)
C(6A)	28(1)	24(1)	33(1)	4(1)	2(1)	-1(1)
C(6B)	32(1)	32(1)	33(1)	5(1)	1(1)	-5(1)
C(6C)	37(1)	38(1)	43(2)	-3(1)	-13(1)	10(1)
C(7A)	37(1)	45(1)	48(2)	-5(1)	2(1)	-11(1)
C(7B)	37(1)	47(2)	62(2)	-7(2)	-1(1)	-14(1)
C(7C)	77(2)	49(2)	45(2)	6(1)	-6(2)	21(2)
C(8A)	37(1)	43(2)	42(2)	1(1)	-6(1)	-6(1)
C(8B)	45(2)	41(2)	59(2)	6(2)	-20(1)	-13(1)
C(9A)	55(2)	53(2)	50(2)	-9(1)	0(1)	-10(1)
C(9B)	96(2)	66(2)	59(2)	-9(2)	-4(2)	-41(2)
C(10A)	29(1)	28(1)	36(2)	-2(1)	-1(1)	0(1)
C(10B)	31(1)	28(1)	29(1)	-5(1)	-1(1)	-2(1)
C(11A)	38(1)	53(2)	64(2)	13(2)	-6(2)	-15(1)
C(11B)	31(1)	38(1)	53(2)	3(1)	-2(1)	-6(1)
C(12A)	36(1)	45(2)	55(2)	1(2)	12(1)	-8(1)
C(12B)	32(1)	36(1)	44(2)	-1(1)	10(1)	-5(1)
C(13A)	78(2)	73(2)	64(2)	24(2)	-5(2)	-37(2)
C(13B)	57(2)	45(2)	45(2)	2(2)	7(1)	-5(1)
N(1A)	60(2)	63(2)	26(1)	3(1)	-3(1)	-21(1)
N(1B)	53(2)	71(2)	23(1)	-1(1)	1(1)	-13(1)
N(1C)	46(2)	70(2)	21(2)	0	0	13(2)

N(2A)	33(1)	31(1)	29(1)	2(1)	0(1)	-3(1)
N(2B)	32(1)	33(1)	26(1)	-1(1)	1(1)	-4(1)
N(2C)	29(1)	34(1)	21(1)	0	0	6(1)
N(3A)	38(1)	38(1)	32(1)	1(1)	-3(1)	-9(1)
N(3B)	41(1)	41(1)	37(1)	5(1)	-9(1)	-13(1)
N(3C)	38(1)	40(1)	28(1)	0(1)	-5(1)	13(1)
N(4A)	37(1)	36(1)	37(1)	1(1)	4(1)	-7(1)
N(4B)	37(1)	38(1)	32(1)	-2(1)	3(1)	-12(1)
O(1A)	37(1)	45(1)	38(1)	2(1)	4(1)	-11(1)
O(1B)	38(1)	59(1)	45(1)	-2(1)	7(1)	-18(1)
O(1C)	37(1)	57(1)	36(1)	-3(1)	4(1)	19(1)
O(2A)	38(1)	55(1)	43(1)	-2(1)	-6(1)	-13(1)
O(2B)	36(1)	48(1)	38(1)	-5(1)	-3(1)	-15(1)

Supplementary Material (ESI) for Chemical Communications

Hydrogen coordinates (x 10^4) and isotropic
displacement parameters (A^2 x 10^3) for Parquette 1274

	x	y	z	U(eq)	
H(2A)	7441	4271	7633	40	
H(2B)	5454	4291	8934	41	
H(2C)	5562	9352	1605	37	
H(5A)	8517	5744	7651	41	
H(5B)	6569	5616	8926	39	
H(5C1)	6503	8473	5268	49	
H(5C2)	6196	7594	5433	49	
H(6C)	6281	9046	7950	47	
H(7A1)	6500	3562	3797	52	
H(7A2)	6742	2583	3873	52	
H(7B1)	4487	3502	5267	59	
H(7B2)	4789	2610	5181	59	
H(7C1)	5895	8110	9971	86	
H(7C2)	6221	7456	8913	86	
H(7C3)	5742	7468	8292	86	
H(8A)	6771	3826	1024	49	
H(8B)	4733	4061	2626	58	
H(9A1)	7223	2809	-484	79	
H(9A2)	6877	2197	543	79	
H(9A3)	7332	2295	1426	79	
H(9B1)	5071	3046	591	111	
H(9B2)	4726	2461	1686	111	
H(9B3)	5206	2382	2267	111	
H(11A)	9494	6463	3922	62	
H(11B)	9210	7384	3941	62	
H(11C)	7484	6559	5211	49	
H(11D)	7182	7448	5055	49	
H(12A)	9229	6002	1228	54	
H(12B)	7267	6057	2462	45	
H(13A)	8857	7073	-594	108	
H(13B)	9239	7588	392	108	
H(13C)	8777	7712	1188	108	
H(13D)	6831	6928	573	73	
H(13E)	7114	7659	1674	73	
H(13F)	6643	7478	2313	73	
H(1A1)	8190(8)	5293(18)	10590(40)	69(9)*	
H(1A2)	7742(7)	4714(17)	10630(30)	58(8)*	

H(1B1)	6238(9)	5231(19)	11900(40)	73(9)*
H(1B2)	5775(7)	4657(16)	12020(40)	53(8)*
H(1C1)	4779(7)	10255(18)	-1390(40)	65(9)*

*Refined isotropically

Supplementary Material (ESI) for Chemical Communications

Crystallographic Data for Ampybox 2.

The data collection crystal was a colorless rectangular block, cut from a very thick plate. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 150 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 3.2, which means that 90% of the reflections were measured at least 3.2 times. A combination of phi and omega scans with a frame width of 1.0˚ was used. Data integration was done with Denzo(1), and scaling and merging of the data was done with Scalepack(1). Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.035.

The structure was solved by the direct methods procedure in SHELXS-97(2). There are 2 molecules in the asymmetric unit, and they are labeled as molecules A and B. The correct enantiomer was chosen based on the known chiral centers. Full-matrix least-squares refinements based on F^2 were performed in SHELXL-97(3), as incorporated in the WinGX package(4).

The hydrogen atoms bonded to the nitrogen atoms were refined isotropically. For the methyl groups, the hydrogen atoms were added at calculated positions using a riding model with U(H) = 1.5 *Ueq(bonded carbon atom). The torsion angle, which defines the orientation of the methyl group about the C-C bond, was refined. The remaining hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The final refinement cycle was based on all 4105 intensities and 439 variables and resulted in agreement factors of R1(F) = 0.042 and wR2(F^2) = 0.083. For the subset of data with I > 2*sigma(I), the R1(F) value is 0.034 for 3588 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.17 and -0.18 e/ Å ^{3}. Neutral atom scattering factors were used and include terms for anomalous dispersion(5).

Supplementary Material (ESI) for Chemical Communications

Table 1. Crystallographic details for Parquette 1286.

Empirical formula	C17 H24 N4 O2
Formula weight	316.40
Temperature	150(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_1$
Unit cell dimensions	a = 6.131(1) Å
	b = 13.950(2) Å
	c = 20.220(3) Å
	β= 91.021(4)°
Volume	1729.1(5) $Å^3$
Z	4
Density (calculated)	1.215 Mg/m^3
Absorption coefficient	0.082 mm^{-1}
F(000)	680
Crystal size	0.23 x 0.23 x 0.31 mm^3
Theta range for data collection	2.49 to 27.48°.
Index ranges	-7<=h<=7, -17<=k<=18, -26<=l<=26
Reflections collected	27009
Independent reflections	4105 [R(int) = 0.035]
Completeness to theta = 27.48°	99.9 %
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4105 / 1 / 439
Goodness-of-fit on F^2	1.075
Final R indices [I>2sigma(I)]	R1 = 0.0339, wR2 = 0.0791
R indices (all data)	R1 = 0.0424, wR2 = 0.0827
Largest diff. peak and hole	0.174 and -0.182 e/$Å^3$

Supplementary Material (ESI) for Chemical Communications

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3)
for Parquette 1286. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1A)	10926(3)	4509(1)	5386(1)	29(1)
N(2A)	9647(2)	1640(1)	4899(1)	21(1)
N(3A)	5918(2)	755(1)	5433(1)	25(1)
N(4A)	12572(2)	704(1)	4054(1)	24(1)
O(1A)	5265(2)	2106(1)	6015(1)	28(1)
O(2A)	14611(2)	2048(1)	4016(1)	27(1)
C(1A)	10503(3)	3586(1)	5234(1)	22(1)
C(2A)	11861(3)	3065(1)	4812(1)	22(1)
C(3A)	11366(3)	2125(1)	4663(1)	20(1)
C(4A)	8378(3)	2138(1)	5313(1)	20(1)
C(5A)	8718(3)	3088(1)	5491(1)	22(1)
C(6A)	6492(3)	1602(1)	5582(1)	21(1)
C(7A)	3527(3)	1465(1)	6207(1)	26(1)
C(8A)	3844(3)	559(1)	5782(1)	23(1)
C(9A)	3860(3)	-389(1)	6152(1)	27(1)
C(10A)	1620(3)	-576(2)	6443(1)	37(1)
C(11A)	5644(3)	-468(2)	6679(1)	43(1)
C(12A)	12826(3)	1570(1)	4229(1)	20(1)
C(13A)	15887(3)	1344(1)	3659(1)	28(1)
C(14A)	14432(3)	444(1)	3638(1)	26(1)
C(15A)	13651(3)	132(2)	2948(1)	32(1)
C(16A)	15577(4)	-220(2)	2546(1)	47(1)
C(17A)	12394(4)	918(2)	2587(1)	48(1)
N(1B)	4028(3)	12388(1)	-1(1)	36(1)
N(2B)	5692(2)	9502(1)	274(1)	21(1)
N(3B)	2863(2)	8308(1)	960(1)	23(1)
N(4B)	9431(2)	8817(1)	-384(1)	26(1)
O(1B)	699(2)	9578(1)	1176(1)	27(1)
O(2B)	9983(2)	10304(1)	-788(1)	28(1)

C(6B)	2508(3)	9200(1)	899(1)	21(1)
C(3B)	6942(3)	10116(1)	-69(1)	21(1)
C(12B)	8845(3)	9685(1)	-405(1)	21(1)
C(5B)	3276(3)	10837(1)	479(1)	23(1)
C(2B)	6483(3)	11078(1)	-151(1)	24(1)
C(4B)	3900(3)	9888(1)	540(1)	20(1)
C(13B)	11762(3)	9745(1)	-1054(1)	27(1)
C(1B)	4564(3)	11468(1)	112(1)	24(1)
C(8B)	1091(3)	7911(1)	1368(1)	25(1)
C(14B)	11272(3)	8711(2)	-843(1)	27(1)
C(9B)	2020(3)	7505(2)	2019(1)	32(1)
C(15B)	10621(4)	8032(2)	-1410(1)	37(1)
C(10B)	219(4)	7060(2)	2431(1)	50(1)
C(7B)	-469(3)	8765(2)	1453(1)	32(1)
C(11B)	3806(4)	6763(2)	1893(1)	46(1)
C(16B)	12608(4)	7781(2)	-1823(1)	52(1)
C(17B)	8756(4)	8437(2)	-1839(1)	55(1)

Table 3. Bond lengths [Å] and angles [°] for Parquette 1286.

N(1A)-C(1A)	1.348(2)
N(1A)-H(1NA)	0.84(2)
N(1A)-H(2NA)	0.87(2)
N(2A)-C(4A)	1.346(2)
N(2A)-C(3A)	1.347(2)
N(3A)-C(6A)	1.267(3)
N(3A)-C(8A)	1.491(2)
N(4A)-C(12A)	1.269(2)
N(4A)-C(14A)	1.475(2)
O(1A)-C(6A)	1.360(2)
O(1A)-C(7A)	1.449(2)
O(2A)-C(12A)	1.358(2)
O(2A)-C(13A)	1.455(2)
C(1A)-C(5A)	1.404(3)
C(1A)-C(2A)	1.406(2)
C(2A)-C(3A)	1.379(3)
C(2A)-H(2A)	0.9500
C(3A)-C(12A)	1.483(2)
C(4A)-C(5A)	1.389(3)
C(4A)-C(6A)	1.488(2)
C(5A)-H(5A)	0.9500
C(7A)-C(8A)	1.542(3)
C(7A)-H(7A1)	0.9900
C(7A)-H(7A2)	0.9900
C(8A)-C(9A)	1.520(3)
C(8A)-H(8A)	1.0000
C(9A)-C(11A)	1.517(3)
C(9A)-C(10A)	1.526(3)
C(9A)-H(9A)	1.0000
C(10A)-H(10A)	0.9800
C(10A)-H(10B)	0.9800
C(10A)-H(10C)	0.9800
C(11A)-H(11A)	0.9800

C(11A)-H(11B)	0.9800
C(11A)-H(11C)	0.9800
C(13A)-C(14A)	1.540(3)
C(13A)-H(13A)	0.9900
C(13A)-H(13B)	0.9900
C(14A)-C(15A)	1.529(3)
C(14A)-H(14A)	1.0000
C(15A)-C(17A)	1.520(3)
C(15A)-C(16A)	1.527(3)
C(15A)-H(15A)	1.0000
C(16A)-H(16A)	0.9800
C(16A)-H(16B)	0.9800
C(16A)-H(16C)	0.9800
C(17A)-H(17A)	0.9800
C(17A)-H(17B)	0.9800
C(17A)-H(17C)	0.9800
N(1B)-C(1B)	1.344(3)
N(1B)-H(1NB)	0.84(2)
N(1B)-H(2NB)	0.92(3)
N(2B)-C(4B)	1.345(2)
N(2B)-C(3B)	1.350(2)
N(3B)-C(6B)	1.269(2)
N(3B)-C(8B)	1.483(2)
N(4B)-C(12B)	1.262(3)
N(4B)-C(14B)	1.482(2)
O(1B)-C(6B)	1.358(2)
O(1B)-C(7B)	1.458(2)
O(2B)-C(12B)	1.362(2)
O(2B)-C(13B)	1.452(2)
C(6B)-C(4B)	1.484(2)
C(3B)-C(2B)	1.381(3)
C(3B)-C(12B)	1.487(2)
C(5B)-C(4B)	1.383(3)
C(5B)-C(1B)	1.403(3)
C(5B)-H(5B)	0.9500

C(2B)-C(1B)	1.408(3)
C(2B)-H(2B)	0.9500
C(13B)-C(14B)	1.535(3)
C(13B)-H(13C)	0.9900
C(13B)-H(13D)	0.9900
C(8B)-C(9B)	1.532(3)
C(8B)-C(7B)	1.539(3)
C(8B)-H(8B)	1.0000
C(14B)-C(15B)	1.534(3)
C(14B)-H(14B)	1.0000
C(9B)-C(10B)	1.527(3)
C(9B)-C(11B)	1.532(3)
C(9B)-H(9B)	1.0000
C(15B)-C(16B)	1.531(3)
C(15B)-C(17B)	1.531(4)
C(15B)-H(15B)	1.0000
C(10B)-H(10D)	0.9800
C(10B)-H(10E)	0.9800
C(10B)-H(10F)	0.9800
C(7B)-H(7B1)	0.9900
C(7B)-H(7B2)	0.9900
C(11B)-H(11D)	0.9800
C(11B)-H(11E)	0.9800
C(11B)-H(11F)	0.9800
C(16B)-H(16D)	0.9800
C(16B)-H(16E)	0.9800
C(16B)-H(16F)	0.9800
C(17B)-H(17D)	0.9800
C(17B)-H(17E)	0.9800
C(17B)-H(17F)	0.9800

C(1A)-N(1A)-H(1NA)	111.2(16)
C(1A)-N(1A)-H(2NA)	117.4(15)
H(1NA)-N(1A)-H(2NA)	124(2)
C(4A)-N(2A)-C(3A)	115.19(15)

C(6A)-N(3A)-C(8A)	107.08(15)
C(12A)-N(4A)-C(14A)	107.50(15)
C(6A)-O(1A)-C(7A)	105.75(13)
C(12A)-O(2A)-C(13A)	105.62(14)
N(1A)-C(1A)-C(5A)	122.42(17)
N(1A)-C(1A)-C(2A)	121.28(17)
C(5A)-C(1A)-C(2A)	116.29(16)
C(3A)-C(2A)-C(1A)	119.60(16)
C(3A)-C(2A)-H(2A)	120.2
C(1A)-C(2A)-H(2A)	120.2
N(2A)-C(3A)-C(2A)	124.86(16)
N(2A)-C(3A)-C(12A)	115.62(15)
C(2A)-C(3A)-C(12A)	119.49(16)
N(2A)-C(4A)-C(5A)	124.64(16)
N(2A)-C(4A)-C(6A)	115.42(15)
C(5A)-C(4A)-C(6A)	119.93(16)
C(4A)-C(5A)-C(1A)	119.40(16)
C(4A)-C(5A)-H(5A)	120.3
C(1A)-C(5A)-H(5A)	120.3
N(3A)-C(6A)-O(1A)	118.70(15)
N(3A)-C(6A)-C(4A)	126.57(16)
O(1A)-C(6A)-C(4A)	114.71(15)
O(1A)-C(7A)-C(8A)	104.83(13)
O(1A)-C(7A)-H(7A1)	110.8
C(8A)-C(7A)-H(7A1)	110.8
O(1A)-C(7A)-H(7A2)	110.8
C(8A)-C(7A)-H(7A2)	110.8
H(7A1)-C(7A)-H(7A2)	108.9
N(3A)-C(8A)-C(9A)	113.26(15)
N(3A)-C(8A)-C(7A)	103.33(14)
C(9A)-C(8A)-C(7A)	116.04(14)
N(3A)-C(8A)-H(8A)	107.9
C(9A)-C(8A)-H(8A)	107.9
C(7A)-C(8A)-H(8A)	107.9
C(11A)-C(9A)-C(8A)	114.00(17)

C(11A)-C(9A)-C(10A)	111.09(16)
C(8A)-C(9A)-C(10A)	109.89(16)
C(11A)-C(9A)-H(9A)	107.2
C(8A)-C(9A)-H(9A)	107.2
C(10A)-C(9A)-H(9A)	107.2
C(9A)-C(10A)-H(10A)	109.5
C(9A)-C(10A)-H(10B)	109.5
H(10A)-C(10A)-H(10B)	109.5
C(9A)-C(10A)-H(10C)	109.5
H(10A)-C(10A)-H(10C)	109.5
H(10B)-C(10A)-H(10C)	109.5
C(9A)-C(11A)-H(11A)	109.5
C(9A)-C(11A)-H(11B)	109.5
H(11A)-C(11A)-H(11B)	109.5
C(9A)-C(11A)-H(11C)	109.5
H(11A)-C(11A)-H(11C)	109.5
H(11B)-C(11A)-H(11C)	109.5
N(4A)-C(12A)-O(2A)	118.36(15)
N(4A)-C(12A)-C(3A)	126.14(16)
O(2A)-C(12A)-C(3A)	115.49(15)
O(2A)-C(13A)-C(14A)	104.35(13)
O(2A)-C(13A)-H(13A)	110.9
C(14A)-C(13A)-H(13A)	110.9
O(2A)-C(13A)-H(13B)	110.9
C(14A)-C(13A)-H(13B)	110.9
H(13A)-C(13A)-H(13B)	108.9
N(4A)-C(14A)-C(15A)	110.93(15)
N(4A)-C(14A)-C(13A)	103.74(14)
C(15A)-C(14A)-C(13A)	115.41(16)
N(4A)-C(14A)-H(14A)	108.8
C(15A)-C(14A)-H(14A)	108.8
C(13A)-C(14A)-H(14A)	108.8
C(17A)-C(15A)-C(16A)	111.44(18)
C(17A)-C(15A)-C(14A)	112.44(17)
C(16A)-C(15A)-C(14A)	110.17(17)

C(17A)-C(15A)-H(15A)	107.5
C(16A)-C(15A)-H(15A)	107.5
C(14A)-C(15A)-H(15A)	107.5
C(15A)-C(16A)-H(16A)	109.5
C(15A)-C(16A)-H(16B)	109.5
H(16A)-C(16A)-H(16B)	109.5
C(15A)-C(16A)-H(16C)	109.5
H(16A)-C(16A)-H(16C)	109.5
H(16B)-C(16A)-H(16C)	109.5
C(15A)-C(17A)-H(17A)	109.5
C(15A)-C(17A)-H(17B)	109.5
H(17A)-C(17A)-H(17B)	109.5
C(15A)-C(17A)-H(17C)	109.5
H(17A)-C(17A)-H(17C)	109.5
H(17B)-C(17A)-H(17C)	109.5
C(1B)-N(1B)-H(1NB)	122.8(17)
C(1B)-N(1B)-H(2NB)	121.1(15)
H(1NB)-N(1B)-H(2NB)	116(2)
C(4B)-N(2B)-C(3B)	115.26(15)
C(6B)-N(3B)-C(8B)	107.09(16)
C(12B)-N(4B)-C(14B)	107.11(15)
C(6B)-O(1B)-C(7B)	105.46(14)
C(12B)-O(2B)-C(13B)	105.42(13)
N(3B)-C(6B)-O(1B)	118.73(17)
N(3B)-C(6B)-C(4B)	125.68(16)
O(1B)-C(6B)-C(4B)	115.58(15)
N(2B)-C(3B)-C(2B)	124.17(16)
N(2B)-C(3B)-C(12B)	115.82(15)
C(2B)-C(3B)-C(12B)	119.92(16)
N(4B)-C(12B)-O(2B)	118.62(16)
N(4B)-C(12B)-C(3B)	126.66(16)
O(2B)-C(12B)-C(3B)	114.67(15)
C(4B)-C(5B)-C(1B)	119.34(16)
C(4B)-C(5B)-H(5B)	120.3
C(1B)-C(5B)-H(5B)	120.3

C(3B)-C(2B)-C(1B)	120.01(17)
C(3B)-C(2B)-H(2B)	120.0
C(1B)-C(2B)-H(2B)	120.0
N(2B)-C(4B)-C(5B)	125.09(16)
N(2B)-C(4B)-C(6B)	114.67(15)
C(5B)-C(4B)-C(6B)	120.20(15)
O(2B)-C(13B)-C(14B)	104.46(13)
O(2B)-C(13B)-H(13C)	110.9
C(14B)-C(13B)-H(13C)	110.9
O(2B)-C(13B)-H(13D)	110.9
C(14B)-C(13B)-H(13D)	110.9
H(13C)-C(13B)-H(13D)	108.9
N(1B)-C(1B)-C(5B)	123.41(18)
N(1B)-C(1B)-C(2B)	120.53(18)
C(5B)-C(1B)-C(2B)	116.05(17)
N(3B)-C(8B)-C(9B)	110.54(15)
N(3B)-C(8B)-C(7B)	103.66(14)
C(9B)-C(8B)-C(7B)	114.38(16)
N(3B)-C(8B)-H(8B)	109.4
C(9B)-C(8B)-H(8B)	109.4
C(7B)-C(8B)-H(8B)	109.4
N(4B)-C(14B)-C(15B)	109.80(15)
N(4B)-C(14B)-C(13B)	103.60(15)
C(15B)-C(14B)-C(13B)	114.94(16)
N(4B)-C(14B)-H(14B)	109.4
C(15B)-C(14B)-H(14B)	109.4
C(13B)-C(14B)-H(14B)	109.4
C(10B)-C(9B)-C(11B)	109.95(18)
C(10B)-C(9B)-C(8B)	110.94(17)
C(11B)-C(9B)-C(8B)	111.32(16)
C(10B)-C(9B)-H(9B)	108.2
C(11B)-C(9B)-H(9B)	108.2
C(8B)-C(9B)-H(9B)	108.2
C(16B)-C(15B)-C(17B)	111.6(2)
C(16B)-C(15B)-C(14B)	110.49(18)

C(17B)-C(15B)-C(14B)	112.19(18)
C(16B)-C(15B)-H(15B)	107.4
C(17B)-C(15B)-H(15B)	107.4
C(14B)-C(15B)-H(15B)	107.4
C(9B)-C(10B)-H(10D)	109.5
C(9B)-C(10B)-H(10E)	109.5
H(10D)-C(10B)-H(10E)	109.5
C(9B)-C(10B)-H(10F)	109.5
H(10D)-C(10B)-H(10F)	109.5
H(10E)-C(10B)-H(10F)	109.5
O(1B)-C(7B)-C(8B)	104.40(14)
O(1B)-C(7B)-H(7B1)	110.9
C(8B)-C(7B)-H(7B1)	110.9
O(1B)-C(7B)-H(7B2)	110.9
C(8B)-C(7B)-H(7B2)	110.9
H(7B1)-C(7B)-H(7B2)	108.9
C(9B)-C(11B)-H(11D)	109.5
C(9B)-C(11B)-H(11E)	109.5
H(11D)-C(11B)-H(11E)	109.5
C(9B)-C(11B)-H(11F)	109.5
H(11D)-C(11B)-H(11F)	109.5
H(11E)-C(11B)-H(11F)	109.5
C(15B)-C(16B)-H(16D)	109.5
C(15B)-C(16B)-H(16E)	109.5
H(16D)-C(16B)-H(16E)	109.5
C(15B)-C(16B)-H(16F)	109.5
H(16D)-C(16B)-H(16F)	109.5
H(16E)-C(16B)-H(16F)	109.5
C(15B)-C(17B)-H(17D)	109.5
C(15B)-C(17B)-H(17E)	109.5
H(17D)-C(17B)-H(17E)	109.5
C(15B)-C(17B)-H(17F)	109.5
H(17D)-C(17B)-H(17F)	109.5
H(17E)-C(17B)-H(17F)	109.5

Supplementary Material (ESI) for Chemical Communications

Symmetry transformations used to generate equivalent atoms:

Supplementary Material (ESI) for Chemical Communications

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for Parquette 1286. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2 a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1A)	30(1)	17(1)	41(1)	-2(1)	12(1)	-1(1)
N(2A)	20(1)	19(1)	22(1)	1(1)	3(1)	0(1)
N(3A)	24(1)	23(1)	27(1)	0(1)	9(1)	-4(1)
N(4A)	26(1)	21(1)	25(1)	-2(1)	7(1)	0(1)
O(1A)	23(1)	24(1)	38(1)	-7(1)	12(1)	-3(1)
O(2A)	24(1)	23(1)	35(1)	-5(1)	11(1)	-2(1)
C(1A)	25(1)	16(1)	26(1)	1(1)	0(1)	3(1)
C(2A)	22(1)	19(1)	26(1)	2(1)	5(1)	-1(1)
C(3A)	21(1)	18(1)	20(1)	1(1)	1(1)	1(1)
C(4A)	19(1)	19(1)	22(1)	1(1)	-1(1)	0(1)
C(5A)	22(1)	20(1)	25(1)	-2(1)	4(1)	3(1)
C(6A)	20(1)	23(1)	19(1)	1(1)	3(1)	2(1)
C(7A)	22(1)	28(1)	27(1)	0(1)	6(1)	-1(1)
C(8A)	19(1)	26(1)	24(1)	0(1)	4(1)	-2(1)
C(9A)	27(1)	24(1)	32(1)	1(1)	8(1)	-2(1)
C(10A)	33(1)	42(1)	36(1)	2(1)	10(1)	-10(1)
C(11A)	33(1)	47(1)	49(1)	16(1)	-1(1)	6(1)
C(12A)	19(1)	20(1)	21(1)	3(1)	3(1)	-1(1)
C(13A)	23(1)	32(1)	29(1)	-8(1)	8(1)	-1(1)
C(14A)	25(1)	23(1)	28(1)	-3(1)	7(1)	2(1)
C(15A)	30(1)	35(1)	32(1)	-13(1)	8(1)	-5(1)
C(16A)	44(1)	54(2)	42(1)	-21(1)	14(1)	-2(1)
C(17A)	46(1)	64(2)	34(1)	-6(1)	-4(1)	5(1)
N(1B)	36(1)	18(1)	54(1)	4(1)	18(1)	3(1)
N(2B)	21(1)	19(1)	24(1)	1(1)	3(1)	1(1)
N(3B)	25(1)	21(1)	25(1)	4(1)	8(1)	-1(1)
N(4B)	26(1)	23(1)	29(1)	0(1)	7(1)	1(1)
O(1B)	27(1)	21(1)	33(1)	-1(1)	12(1)	1(1)
O(2B)	23(1)	24(1)	38(1)	4(1)	10(1)	-1(1)
C(6B)	23(1)	22(1)	19(1)	-2(1)	3(1)	2(1)

C(3B)	20(1)	20(1)	24(1)	0(1)	1(1)	-2(1)
C(12B)	20(1)	20(1)	24(1)	1(1)	1(1)	-4(1)
C(5B)	24(1)	20(1)	25(1)	-4(1)	6(1)	2(1)
C(2B)	23(1)	20(1)	29(1)	2(1)	4(1)	-3(1)
C(4B)	22(1)	20(1)	19(1)	-2(1)	2(1)	0(1)
C(13B)	20(1)	30(1)	30(1)	-2(1)	8(1)	-1(1)
C(1B)	28(1)	17(1)	29(1)	-2(1)	3(1)	-1(1)
C(8B)	26(1)	23(1)	26(1)	2(1)	6(1)	-3(1)
C(14B)	21(1)	28(1)	31(1)	2(1)	6(1)	2(1)
C(9B)	39(1)	31(1)	27(1)	4(1)	2(1)	-8(1)
C(15B)	39(1)	29(1)	44(1)	-11(1)	15(1)	-5(1)
C(10B)	58(2)	52(2)	40(1)	19(1)	14(1)	-7(1)
C(7B)	31(1)	29(1)	37(1)	5(1)	14(1)	-1(1)
C(11B)	46(1)	51(2)	40(1)	17(1)	-4(1)	5(1)
C(16B)	54(2)	52(2)	51(2)	-17(1)	21(1)	4(1)
C(17B)	38(1)	76(2)	50(1)	-24(1)	-5(1)	-11(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for Parquette 1286.

	x	y	z	U(eq)
H(1NA)	11820(40)	4748(17)	5125(11)	37(6)
H(2NA)	9910(40)	4830(17)	5581(10)	35(6)
H(2A)	13112	3359	4630	27
H(5A)	7751	3399	5784	27
H(7A1)	2082	1758	6115	31
H(7A2)	3644	1309	6684	31
H(8A)	2637	537	5443	28
H(9A)	4126	-905	5820	33
H(10A)	1340	-106	6792	55
H(10B)	1582	-1223	6631	55
H(10C)	498	-518	6094	55
H(11A)	7035	-263	6494	64
H(11B)	5764	-1135	6828	64
H(11C)	5285	-57	7055	64
H(13A)	17284	1211	3894	33
H(13B)	16198	1571	3206	33
H(14A)	15243	-99	3851	31
H(15A)	12637	-422	3005	39
H(16A)	16596	311	2477	70
H(16B)	15046	-458	2116	70
H(16C)	16325	-738	2786	70
H(17A)	13355	1468	2516	72
H(17B)	11151	1117	2852	72
H(17C)	11864	675	2158	72
H(1NB)	2890(40)	12640(18)	146(11)	35(6)
H(2NB)	4830(40)	12765(19)	-283(11)	41(7)
H(5B)	1989	11060	684	28
H(2B)	7461	11477	-384	29
H(13C)	13182	9961	-868	32

H(13D)	11792	9799	-1542	32
H(8B)	323	7390	1117	30
H(14B)	12565	8442	-598	32
H(9B)	2682	8045	2278	39
H(15B)	10087	7424	-1206	45
H(10D)	-486	6541	2179	75
H(10E)	-865	7550	2536	75
H(10F)	852	6801	2842	75
H(7B1)	-1862	8654	1210	39
H(7B2)	-779	8873	1926	39
H(11D)	3182	6220	1647	68
H(11E)	4411	6536	2317	68
H(11F)	4966	7056	1635	68
H(16D)	12177	7327	-2171	78
H(16E)	13734	7489	-1538	78
H(16F)	13186	8365	-2024	78
H(17D)	9252	9018	-2064	82
H(17E)	7518	8594	-1559	82
H(17F)	8305	7959	-2169	82