Supplementary Information:

Imidazolium Ylides from a Conjugate Addition – Proton Transfer Route and their Cycloaddition Reactions

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Contents:

Typical Experimental procedure:

Reaction of 1-benzyl-4,5-dihydroimidazole (1) with dimethyl fumarate:

1-Benzyl-5,6,7-tris(methoxycarbonyl)-5-methoxycarbonylmethylhexahydro-1*H*-pyrrolo[1,2-*a*]imidazole stereoisomers 4a and 5a

Dimethyl fumarate (1.11 g, 7.5 mmol) was added to a stirred solution of 1-benzyl-4,5-dihydroimidazole **1** (0.4 g , 2.5 mmol) in dry dichloromethane (5 mL) under an atmosphere of nitrogen. The mixture was heated at reflux for 48 h, then concentrated under reduced pressure. The crude oil was purified by column chromatography at medium pressure on silica gel (Merck Kieselgel 60, Art. 9385) eluting with ethyl acetate : hexane (2:8 v/v) containing triethylamine (3% v/v), to afford major diastereoisomer **4a** as a colourless oil (0.49 g, 45%) and minor diastereoisomer **5a** as a white solid (0.11g, 10%).

Pyrroloimidazole **4a**:

□□□□□□□□□□□□□□□□□ ν_{max} (film)/cm^{-1} 3027, 2953, 1733 (C=O), 1437, 1353, 1207, 754, 702; δ_H (300 MHz; $CDCl_3$) 2.28 (1H, m, NCH_2CHHN), 2.95 (2H, m, $NCHHCH_2N$), 3.08 (1H, d, J = 14.6 $CHHCO_2CH_3$), 3.09 (1H, d, J = 13.2, PhC*H*H), 3.12 (1H, m, NCH_2CHHN), 3.13 (1H, d, J = 14.6, $CHHCO_2CH_3$), 3.66 (1H, dd, J = 4.6, 10.8, 7-CH), 3.68, 3.69, 3.71, 3.74 (each 3H, s, CH_3), 3.85 (1H, d, J = 4.6, 7a-CH), 4.12 (1H, d, J = 13.2, PhCH*H*), 4.45 (1H, d, J = 10.8, 6-CH), 7.28 (5H, m, 5 x Ar-*H*); δ_C (75 MHz; $CDCl_3$) 39.8 ($CH_2CO_2CH_3$), 47.9, 53.79 (2 x NCH_2), 49.9 (7-CH), 52.1, 52.2, 52.4, 53.1 (4 x OCH_3), 53.8 (6-CH), 56.2 (PhCH_2), 71.1 (5-C), 86.9 (7a-CH), 127.0, 128.3, 128.5 (3 x Ar-*C*H), 138.85 (Ar-*C*), 170.7, 170.9, 171.4, 172.8 (4 x CO); *m/z* (CI) 449 (MH^+, 100%). HRMS: (CI) MH^+ 449.1924; $C_{22}H_{28}N_2O_8$ requires MH^+ 449.1918.

Pyrroloimidazole **5a**:

Colourless solid; m.p. 106-107 °C; ν_{max} (film)/cm^{-1} 3028, 2952, 1736 (C=O), 1437, 1352, 1205, 741, 698; δ_H (300 MHz; $CDCl_3$) 2.56 (1H, m, NCH_2CHHN), 2.88 (3H, m, NCH_2CHHN), 2.91 (1H, d, J = 17.1, $CHHCO_2CH_3$), 3.43 (1H, d, J = 13.6, PhC*H*H), 3.55

(1H, d, *J* = 11.3, 6-CH), 3.61 (1H, dd, *J* = 11.3, 7.0, 7-CH), 3.62 (3H, s, OCH_3), 3.64 (1H, d, *J* = 17.1, CH*H*CO_2CH_3), 3.70, 3.73, 3.74 (each 3H, s, OCH_3), 4.21 (1H, d, *J* = 13.6, PhCH*H*), 4.90 (1H, d, *J* = 7.0, 7a-CH), 7.28 (5H, m, Ar-*H*); δ_C (75 MHz; $CDCl_3$) 38.3 (*C*$H_2CO_2CH_3$), 47.9, 51.9 (2 x NCH_2), 51.1 (7-CH), 51.7 (6-CH), 52.1, 52.2, 52.45, 52.8 (4 x OCH_3), 59.4 (Ph*C*H_2), 71.95 (5-C), 84.55 (7a-CH), 127.0, 128.05, 128.3 (3 x Ar-*C*H), 138.8 (Ar-*C*), 170.2, 170.3, 171.3, 171.5 (4 x CO); *m/z* (CI) 449 (MH^+, 100%). HRMS: (CI) MH^+ 449.1925; $C_{22}H_{28}N_2O_8$ requires MH^+ 449.1918.

Key Spectroscopic Data

2:1 Cycloadduct Stereoisomers 2a and 3a

Cycloadduct **2a** (major stereoisomer):

Colourless solid; m.p. 161-163 °C; ν_{max} (film)/cm^{-1} 3027, 2930, 2835, 2811, 1772 and 1714 (C=O, imide symmetric & antisymmetric stretch), 1692, 1436, 1378, 1282, 1129, 964, 742, 698; δ_H (300 MHz; $CDCl_3$) 2.42 (1H, m, NCH_2CHHN), 2.60, 2.79 (each 1H, m, NCH_2CH_2N), 2.93 (1H, d, *J* = 18.8, CC*H*HC=O), 3.00, 3.01 (each 3H, s, NCH_3), 3.03 (1H, m, NCH_2CHHN), 3.22 (1H, d, *J* = 7.9, 6-CH), 3.30 (1H, d, *J* = 12.1, PhCH*H*), 3.93 (1H, dd, *J* = 7.9, 7.6, C-7*H*), 4.10 (1H, d, *J* = 18.8, CCH*H*C=O), 4.35 (1H, d, *J* = 12.1, PhC*H*H), 4.70 (1H, d, *J* = 7.6, 7a-CH), 7.28 (5H, m, Ar-H); δ_C (75 MHz; $CDCl_3$) 24.9, 25.0 (2 x NCH_3), 32.7 (C*C*H_2CO), 49.5, 51.9 (2 x NCH_2), 49.55 (6-CH), 51.2 (7-CH), 58.5 (Ph*C*H_2), 69.4 (5-C), 85.8 (7a-CH), 127.25, 128.3, 129.1 (3 x Ar-*C*H), 137.9 (Ar-*C*), 174.4, 175.4, 176.3, 177.7 (4 x CO); *m/z* (CI) 383 (MH^+, 100%). HRMS: (CI) MH^+ 383.1716; $C_{20}H_{22}N_4O_4$ requires MH^+ 383.1714.

Cycloadduct **3a** (minor stereoisomer):

Colourless solid; m.p. 171-174 °C; ν_{max} (film)/cm^{-1} 3027, 2958, 2931, 2874, 2858, 1778 and 1712 (C=O, imide symmetric & antisymmetric stretch), 1673, 1435, 1383,1287, 1128, 979,

749, 699; δ_H (300 MHz; $CDCl_3$) 2.51 (1H, m, NC*H*HCH$_2$N), 2.77 (1H, m, NCH$_2$C*H*HN), 2.86 (1H, m, NCH*H*CH$_2$N), 2.94 (3H, s, NCH$_3$), 2.96 (2H, s, CCH$_2$CO), 2.99 (3H, s, NCH$_3$), 3.10 (1H, m, NCH$_2$CH*H*N), 3.39 (1H, dd, *J* = 1.8, 9.3, 7-CH), 3.48 (1H, d, *J* = 9.3, 6-CH), 3.60, 4.11 (each 1H, d, *J* = 13.25, PhC*H*$_2$), 4.42 (1H, d, *J* = 1.8, 7a-CH), 7.28 (5H, m, Ar-*H*); δ_C (75 MHz; $CDCl_3$) 25.1, 25.3 (2 x NCH$_3$), 39.2 (C*C*H$_2$CO), 46.9, 51.9 (2 x NCH$_2$), 52.0 (7-CH), 53.55 (6-CH), 57.1 (Ph*C*H$_2$), 71.85 (5-C), 87.5 (7a-CH), 127.4, 128.5, 128.6 (3 x Ar-*C*H), 137.9 (Ar-*C*), 173.5, 175.1, 175.9, 176.3 (4 x CO); *m/z* 383 (MH^+, 100%). HRMS: MH^+ 383.1723; $C_{20}H_{22}N_4O_4$ requires MH^+ 383.1714.

1:1:1 Cycloadduct 8a (major stereoisomer)

Colourless solid; m.p. 113-115 ºC; ν_{max} (film)/cm^{-1} 3028, 2952, 2846, 1784 and 1707 (C=O, imide symmetric & antisymmetric stretch), 1735 (C=O), 1438, 1386, 1349, 1288, 1166, 1030, 968, 739, 700; δ_H (300 MHz; $CDCl_3$) 2.42 (1H, m, NC*H*HCH$_2$N), 2.81 (1H, m, NCH$_2$C*H*HN), 2.95 (1H, m, NCH*H*CH$_2$N), 2.99 (3H, s, NCH$_3$), 3.03 (1H, d, *J* = 17.8, CC*H*HCO), 3.05 (1H, m, NCH$_2$CH*H*N), 3.17 (1H, d, *J* = 17.8, CCH*H*CO), 3.35 (1H, d, *J* = 13.4, PhC*H*H), 3.65, 3.72 (each 3H, s, CO$_2$CH$_3$), 3.92 (1H, d, *J* = 10.4, 6-CH), 3.99 (1H, dd, *J* = 10.4, 7.4, 7-CH), 4.13 (1H, d, *J* = 13.4, PhCH*H*), 4.83 (1H, d, *J* = 7.4, 7a-CH), 7.28 (5H, m, Ar-*H*); δ_C (75 MHz; $CDCl_3$) 24.8 (NCH$_3$), 37.1 (C*C*H$_2$CO), 49.2 (NCH$_2$), 49.7 (7-CH), 52.1 (NCH$_2$), 52.3 (6-CH), 52.4, 52.6 (2 x OCH$_3$), 59.8 (Ph*C*H$_2$), 69.8 (5-C), 85.2 (7a-CH), 127.1, 128.3, 128.3 (3 x Ar-*C*H), 138.4 (Ar-*C*), 171.4, 172.1, 174.4, 177.5 (4 x CO); *m/z* (CI) 416 (MH^+, 75%). HRMS: (CI) MH^+ 416.1826; $C_{21}H_{25}N_3O_6$ requires MH^+ 416.1816.

(3*R*,5*R*,6*S*,7*S*,7a*R*)-1-Benzyl-3-phenyl-5,6,7-tris(methoxycarbonyl)-5-methoxycarbonylmethylhexahydro-1*H*-pyrrolo[1,2-*a*]imidazole 12a

Colourless solid; m.p. 140-141 ºC; $[\alpha]_D^{23}$ 60.1 (*c* 0.61 in CH_2Cl_2); ν_{max} (film)/cm^{-1} 3028, 2952, 2815, 1737 (C=O), 1494, 1436, 1359, 1297, 1206, 1172, 1064, 753, 702; δ_H (300 MHz; $CDCl_3$) 2.27 (1H, dd, *J* = 9.7, 9.1, NCHPhC*H*HN), 2.80 (1H, d, *J* = 18.1, CC*H*HCO_2CH_3), 3.03 (3H, s, CO_2CH_3), 3.10 (1H, dd, *J* = 9.1, 5.5, NCHPhCH*H*N), 3.37 (1H, d, *J* = 13.0, PhC*H*H), 3.43 (1H, d, *J* = 18.1, CCH*H*CO_2CH_3), 3.67 (3H, s, CO_2CH_3), 3.67 (1H, d, *J* = 4.5, 6-CH), 3.68 (3H, s, CO_2CH_3), 3.71 (1H, d, *J* = 2.9, 7a-CH), 3.71 (3H, s, CO_2CH_3), 4.21 (1H, d, *J* = 13.0, PhCH*H*), 4.22 (1H, dd, *J* = 9.7, 5.5, NC*H*PhCH_2), 5.08 (1H, dd, *J* = 4.5, 2.9, 7-CH), 7.27 (10H, m, 2 x Ar-*H*); δ_C (75 MHz; $CDCl_3$) 37.5 (C*C*H$_2$$CO_2CH_3$), 51.2 (7-CH), 51.3 (6-CH), 52.1, 52.3, 52.4 (4 x OCH_3), 59.55 (Ph*C*H_2), 63.4 (NCHPh*C*H_2N), 64.1 (N*C*HPhCH_2N), 71.9 (5-C), 85.6 (7a-CH), 127.0, 127.5, 128.1, 128.2, 128.3 (6 x Ar-*C*H), 138.5, 140.2 (2 x Ar-*C*), 169.6, 170.2, 171.45, 171.45 (4 x CO); *m/z* (CI) 525 (MH^+, 65%). HRMS: MH^+ 525.2237; $C_{28}H_{32}N_2O_8$ requires MH^+ 525.2231.

Crystallographic data tables for compound **2a** (compound identification code rcfjdl2)

Table 1. Crystal data and structure refinement for rcfjdl2.

Identification code	rcfjdl2	
Empirical formula	C20 H22 N4 O4	
Formula weight	382.42	
Temperature	150(2) K	
Wavelength	0.84640 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 6.561(3) Å	α= 90°.
	b = 9.171(4) Å	β= 94.608(6)°.
	c = 32.072(13) Å	γ = 90°.
Volume	1923.4(14) $Å^3$	
Z	4	
Density (calculated)	1.321 Mg/m^3	
Absorption coefficient	0.094 mm^{-1}	
F(000)	808	
Crystal size	0.02 x 0.05 x 0.09 mm^3	
Crystal description	colourless plate	
Theta range for data collection	3.71 to 22.50°.	
Index ranges	-5<=h<=5, -8<=k<=8, -28<=l<=28	
Reflections collected	6398	
Independent reflections	1480 [R(int) = 0.1271]	
Completeness to theta = 22.50°	99.6 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1480 / 0 / 256	
Goodness-of-fit on F^2	1.139	
Final R indices [I>2sigma(I)]	R1 = 0.0834, wR2 = 0.2070	
R indices (all data)	R1 = 0.1062, wR2 = 0.2177	
Extinction coefficient	0.009(2)	
Largest diff. peak and hole	0.273 and -0.254 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for rcfjdl2. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	941(13)	6779(9)	6005(3)	33(3)
N(1)	1702(11)	6732(8)	6440(3)	40(2)
C(2)	2966(15)	8026(11)	6508(3)	51(3)
C(3)	4044(15)	8113(10)	6102(3)	51(3)
N(2)	2310(10)	7749(7)	5790(2)	32(2)
C(4)	2769(13)	7093(9)	5396(3)	31(2)
C(5)	1077(17)	7561(9)	5079(3)	30(2)
O(1)	-734(10)	7292(6)	5087(2)	41(2)
N(3)	1878(12)	8383(7)	4774(2)	28(2)
C(6)	676(14)	9058(10)	4433(3)	43(3)
C(7)	4001(16)	8394(9)	4824(3)	32(3)
O(2)	5018(9)	9011(7)	4577(2)	44(2)
C(8)	4703(13)	7640(9)	5213(3)	35(3)
C(9)	2554(12)	5408(9)	5468(3)	32(3)
C(10)	4401(15)	4673(9)	5667(3)	29(2)
O(3)	6126(10)	4622(6)	5554(2)	41(2)
N(4)	3876(12)	3945(8)	6020(3)	35(2)
C(11)	5234(15)	3020(10)	6276(3)	49(3)
C(12)	1818(15)	4126(10)	6081(3)	33(3)
O(4)	964(10)	3449(7)	6338(2)	49(2)
C(13)	975(12)	5259(10)	5781(3)	32(2)
C(14)	192(15)	6572(11)	6735(3)	54(3)
C(15)	1093(17)	6221(11)	7170(3)	48(3)
C(16)	2839(19)	5360(12)	7229(4)	64(3)
C(17)	3680(20)	5026(12)	7618(5)	76(4)
C(18)	2780(20)	5470(14)	7967(4)	78(4)
C(19)	1070(30)	6311(14)	7917(4)	81(4)
C(20)	240(20)	6713(13)	7520(4)	79(4)

Table 3. Bond lengths [Å] and angles [°] for rcfjdl2.

C(1)-N(1)	1.443(11)	O(2)-C(7)#1	3.094(10)
C(1)-N(2)	1.473(10)	C(9)-C(10)	1.484(12)
C(1)-C(13)	1.569(12)	C(9)-C(13)	1.507(11)
N(1)-C(14)	1.433(11)	C(10)-O(3)	1.217(9)
N(1)-C(2)	1.454(12)	C(10)-N(4)	1.383(11)
C(2)-C(3)	1.533(13)	N(4)-C(12)	1.389(11)
C(3)-N(2)	1.491(11)	N(4)-C(11)	1.439(11)
N(2)-C(4)	1.453(10)	C(12)-O(4)	1.206(10)
C(4)-C(5)	1.506(12)	C(12)-C(13)	1.491(12)
C(4)-C(8)	1.525(11)	C(14)-C(15)	1.506(13)
C(4)-C(9)	1.571(12)	C(15)-C(20)	1.368(14)
C(5)-O(1)	1.216(10)	C(15)-C(16)	1.392(14)
C(5)-N(3)	1.372(11)	C(16)-C(17)	1.357(14)
N(3)-C(7)	1.389(11)	C(17)-C(18)	1.365(15)
N(3)-C(6)	1.436(10)	C(18)-C(19)	1.364(16)
C(7)-O(2)	1.217(10)	C(19)-C(20)	1.395(16)
C(7)-C(8)	1.468(12)		

N(1)-C(1)-N(2)	107.0(7)	O(1)-C(5)-C(4)	126.7(9)
N(1)-C(1)-C(13)	113.5(7)	N(3)-C(5)-C(4)	109.5(8)
N(2)-C(1)-C(13)	107.0(6)	C(5)-N(3)-C(7)	111.1(8)
C(14)-N(1)-C(1)	116.0(8)	C(5)-N(3)-C(6)	124.2(8)
C(14)-N(1)-C(2)	113.6(8)	C(7)-N(3)-C(6)	124.6(8)
C(1)-N(1)-C(2)	105.7(7)	O(2)-C(7)-N(3)	121.7(9)
N(1)-C(2)-C(3)	102.2(7)	O(2)-C(7)-C(8)	128.5(9)
N(2)-C(3)-C(2)	100.2(7)	N(3)-C(7)-C(8)	109.7(9)
C(4)-N(2)-C(1)	109.5(7)	C(7)-O(2)-C(7)#1	92.9(6)
C(4)-N(2)-C(3)	118.4(7)	C(7)-C(8)-C(4)	105.6(7)
C(1)-N(2)-C(3)	106.4(7)	C(10)-C(9)-C(13)	104.6(7)
N(2)-C(4)-C(5)	106.0(7)	C(10)-C(9)-C(4)	115.4(7)
N(2)-C(4)-C(8)	115.8(7)	C(13)-C(9)-C(4)	105.2(7)
C(5)-C(4)-C(8)	103.7(7)	O(3)-C(10)-N(4)	121.6(8)
N(2)-C(4)-C(9)	104.7(7)	O(3)-C(10)-C(9)	129.5(9)
C(5)-C(4)-C(9)	108.0(7)	N(4)-C(10)-C(9)	108.9(8)
C(8)-C(4)-C(9)	117.9(7)	C(10)-N(4)-C(12)	111.4(8)
O(1)-C(5)-N(3)	123.8(9)	C(10)-N(4)-C(11)	124.7(8)

C(12)-N(4)-C(11)	123.7(8)	C(20)-C(15)-C(16)	117.5(10)
O(4)-C(12)-N(4)	123.0(9)	C(20)-C(15)-C(14)	122.1(11)
O(4)-C(12)-C(13)	129.1(9)	C(16)-C(15)-C(14)	120.3(9)
N(4)-C(12)-C(13)	107.9(8)	C(17)-C(16)-C(15)	121.5(11)
C(12)-C(13)-C(9)	104.7(7)	C(16)-C(17)-C(18)	121.0(12)
C(12)-C(13)-C(1)	110.1(7)	C(19)-C(18)-C(17)	118.6(12)
C(9)-C(13)-C(1)	105.1(7)	C(18)-C(19)-C(20)	120.8(11)
N(1)-C(14)-C(15)	113.2(8)	C(15)-C(20)-C(19)	120.4(12)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+2,-z+1

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for rcfjdl2. 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}
C(1)	17(5)	35(6)	51(8)	-6(6)	15(5)	4(5)
N(1)	42(5)	36(5)	45(6)	-7(4)	12(5)	4(4)
C(2)	56(7)	43(7)	54(8)	-8(6)	4(6)	8(6)
C(3)	62(7)	34(6)	56(7)	-7(5)	-2(6)	-2(5)
N(2)	20(5)	25(5)	52(5)	-3(4)	10(4)	2(4)
C(4)	22(6)	24(6)	48(7)	-8(5)	7(6)	3(5)
C(5)	26(8)	24(6)	42(7)	-2(5)	14(6)	-3(5)
O(1)	13(4)	44(4)	66(5)	3(3)	7(3)	-1(3)
N(3)	16(6)	26(5)	42(5)	3(4)	4(4)	3(4)
C(6)	49(7)	30(6)	49(7)	6(5)	-1(6)	0(5)
C(7)	29(8)	14(6)	54(8)	-9(6)	14(6)	4(5)
O(2)	34(4)	42(4)	57(5)	0(4)	14(4)	-5(3)
C(8)	28(6)	27(6)	50(7)	4(5)	8(5)	-9(5)
C(9)	16(6)	30(6)	50(6)	-8(5)	11(5)	-3(5)
C(10)	6(7)	24(6)	58(8)	-13(6)	5(6)	1(5)
O(3)	21(4)	30(4)	71(5)	0(3)	3(4)	5(3)
N(4)	20(6)	33(5)	54(6)	14(5)	7(4)	8(4)
C(11)	57(7)	32(6)	59(7)	4(6)	8(6)	7(6)
C(12)	34(8)	17(6)	49(7)	4(6)	8(6)	1(5)
O(4)	59(5)	32(4)	56(5)	6(4)	14(4)	-7(4)
C(13)	16(6)	39(6)	44(6)	2(6)	13(5)	-4(5)
C(14)	58(7)	53(7)	54(8)	-2(6)	27(7)	19(6)
C(15)	57(8)	51(7)	37(8)	-4(6)	18(6)	2(6)
C(16)	91(10)	59(8)	42(9)	-1(6)	10(7)	28(7)
C(17)	113(11)	56(8)	57(9)	-2(8)	7(9)	-2(7)
C(18)	114(12)	55(9)	64(11)	-7(8)	5(9)	-1(8)
C(19)	145(13)	61(9)	43(10)	-23(7)	37(8)	9(9)
C(20)	105(10)	82(10)	54(9)	-7(8)	32(8)	12(8)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for rcfjdl2.

	x	y	z	U(eq)
H(1)	-480	7181	5979	40
H(2A)	3962	7909	6754	61
H(2B)	2121	8903	6546	61
H(3A)	4588	9102	6056	61
H(3B)	5166	7392	6098	61
H(6A)	816	8501	4176	64
H(6B)	1156	10058	4396	64
H(6C)	-764	9075	4494	64
H(8A)	5461	8318	5410	42
H(8B)	5610	6814	5155	42
H(9)	2080	4910	5200	38
H(11A)	4728	2923	6554	74
H(11B)	6605	3452	6303	74
H(11C)	5294	2056	6146	74
H(13)	-397	4983	5646	39
H(14A)	-765	5784	6639	64
H(14B)	-603	7487	6744	64
H(16)	3458	4999	6992	76
H(17)	4908	4475	7649	91
H(18)	3345	5198	8238	94
H(19)	428	6628	8157	98
H(20)	-924	7333	7491	95

Table 6. Torsion angles [°] for rcfjdl2.

N(2)-C(1)-N(1)-C(14)	147.2(7)	N(3)-C(7)-C(8)-C(4)	2.2(9)
C(13)-C(1)-N(1)-C(14)	-95.0(9)	N(2)-C(4)-C(8)-C(7)	-114.1(8)
N(2)-C(1)-N(1)-C(2)	20.4(8)	C(5)-C(4)-C(8)-C(7)	1.6(8)
C(13)-C(1)-N(1)-C(2)	138.1(7)	C(9)-C(4)-C(8)-C(7)	120.8(8)
C(14)-N(1)-C(2)-C(3)	-167.3(8)	N(2)-C(4)-C(9)-C(10)	-85.0(8)
C(1)-N(1)-C(2)-C(3)	-39.0(8)	C(5)-C(4)-C(9)-C(10)	162.3(8)
N(1)-C(2)-C(3)-N(2)	41.8(8)	C(8)-C(4)-C(9)-C(10)	45.3(11)
N(1)-C(1)-N(2)-C(4)	136.2(7)	N(2)-C(4)-C(9)-C(13)	29.7(9)
C(13)-C(1)-N(2)-C(4)	14.2(9)	C(5)-C(4)-C(9)-C(13)	-82.9(9)
N(1)-C(1)-N(2)-C(3)	7.2(8)	C(8)-C(4)-C(9)-C(13)	160.1(7)
C(13)-C(1)-N(2)-C(3)	-114.8(7)	C(13)-C(9)-C(10)-O(3)	-172.5(8)
C(2)-C(3)-N(2)-C(4)	-153.5(7)	C(4)-C(9)-C(10)-O(3)	-57.4(12)
C(2)-C(3)-N(2)-C(1)	-29.8(8)	C(13)-C(9)-C(10)-N(4)	9.7(9)
C(1)-N(2)-C(4)-C(5)	86.9(8)	C(4)-C(9)-C(10)-N(4)	124.8(8)
C(3)-N(2)-C(4)-C(5)	-150.9(7)	O(3)-C(10)-N(4)-C(12)	-178.3(7)
C(1)-N(2)-C(4)-C(8)	-158.6(7)	C(9)-C(10)-N(4)-C(12)	-0.2(9)
C(3)-N(2)-C(4)-C(8)	-36.5(10)	O(3)-C(10)-N(4)-C(11)	-2.9(12)
C(1)-N(2)-C(4)-C(9)	-27.1(8)	C(9)-C(10)-N(4)-C(11)	175.1(8)
C(3)-N(2)-C(4)-C(9)	95.0(8)	C(10)-N(4)-C(12)-O(4)	170.7(8)
N(2)-C(4)-C(5)-O(1)	-61.4(10)	C(11)-N(4)-C(12)-O(4)	-4.7(13)
C(8)-C(4)-C(5)-O(1)	176.1(8)	C(10)-N(4)-C(12)-C(13)	-9.6(9)
C(9)-C(4)-C(5)-O(1)	50.3(11)	C(11)-N(4)-C(12)-C(13)	174.9(7)
N(2)-C(4)-C(5)-N(3)	117.5(7)	O(4)-C(12)-C(13)-C(9)	-165.2(9)
C(8)-C(4)-C(5)-N(3)	-5.0(8)	N(4)-C(12)-C(13)-C(9)	15.2(9)
C(9)-C(4)-C(5)-N(3)	-130.8(7)	O(4)-C(12)-C(13)-C(1)	82.3(11)
O(1)-C(5)-N(3)-C(7)	-174.4(7)	N(4)-C(12)-C(13)-C(1)	-97.3(8)
C(4)-C(5)-N(3)-C(7)	6.7(9)	C(10)-C(9)-C(13)-C(12)	-14.8(8)
O(1)-C(5)-N(3)-C(6)	1.7(12)	C(4)-C(9)-C(13)-C(12)	-136.8(7)
C(4)-C(5)-N(3)-C(6)	-177.2(7)	C(10)-C(9)-C(13)-C(1)	101.2(7)
C(5)-N(3)-C(7)-O(2)	176.9(8)	C(4)-C(9)-C(13)-C(1)	-20.8(9)
C(6)-N(3)-C(7)-O(2)	0.9(12)	N(1)-C(1)-C(13)-C(12)	-0.4(10)
C(5)-N(3)-C(7)-C(8)	-5.6(9)	N(2)-C(1)-C(13)-C(12)	117.4(8)
C(6)-N(3)-C(7)-C(8)	178.3(7)	N(1)-C(1)-C(13)-C(9)	-112.6(8)
N(3)-C(7)-O(2)-C(7)#1	104.1(7)	N(2)-C(1)-C(13)-C(9)	5.2(9)
C(8)-C(7)-O(2)-C(7)#1	-72.7(8)	C(1)-N(1)-C(14)-C(15)	168.1(8)
O(2)-C(7)-C(8)-C(4)	179.4(8)	C(2)-N(1)-C(14)-C(15)	-69.1(10)

N(1)-C(14)-C(15)-C(20)	146.4(10)	C(16)-C(17)-C(18)-C(19)	-2.7(18)
N(1)-C(14)-C(15)-C(16)	-34.2(13)	C(17)-C(18)-C(19)-C(20)	0.0(19)
C(20)-C(15)-C(16)-C(17)	-0.4(17)	C(16)-C(15)-C(20)-C(19)	-2.3(17)
C(14)-C(15)-C(16)-C(17)	-179.8(11)	C(14)-C(15)-C(20)-C(19)	177.1(11)
C(15)-C(16)-C(17)-C(18)	3.0(18)	C(18)-C(19)-C(20)-C(15)	2.5(19)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+2,-z+1

Crystallographic data tables for compound **5a** (compound identification code 00src257)

Table 1. Crystal data and structure refinement.

Identification code	**00src257**	
Empirical formula	$C_{22}H_{28}N_2O_8$	
Formula weight	448.46	
Temperature	150(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	$P\bar{1}$	
Unit cell dimensions	a = 7.4904(10) Å	α = 68.762(6)°
	b = 12.842(2) Å	β = 75.646(6)°
	c = 13.370(2) Å	γ = 80.916(8)°
Volume	1158.0(3) Å^3	
Z	2	
Density (calculated)	1.286 Mg / m^3	
Absorption coefficient	0.098 mm^{-1}	
F(000)	476	
Crystal	Block; colourless	
Crystal size	0.10 × 0.10 × 0.05 mm^3	
θ range for data collection	3.00 – 24.50°	
Index ranges	$-8 \le h \le 8$, $-13 \le k \le 14$, $-14 \le l \le 15$	
Reflections collected	4414	
Independent reflections	2823 [R_{int} = 0.0537]	
Completeness to θ = 24.50°	73.3 %	
Max. and min. transmission	0.9951 and 0.9902	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2823 / 0 / 290	
Goodness-of-fit on F^2	1.153	
Final R indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.0605, *wR2* = 0.1049	
R indices (all data)	*R1* = 0.1317, *wR2* = 0.1183	
Extinction coefficient	0.022(3)	
Largest diff. peak and hole	0.170 and –0.153 e Å^{-3}	

Diffractometer: *Enraf Nonius KappaCCD* area detector (ϕ scans and ω scans to fill *Ewald* sphere). **Data collection and cell refinement**: *Denzo* (Z. Otwinowski & W. Minor, *Methods in Enzymology* (1997) Vol. **276**: *Macromolecular Crystallography*, part A, pp. 307–326; C. W. Carter, Jr. & R. M. Sweet, Eds., Academic Press). **Absorption correction**: *SORTAV* (R. H. Blessing, Acta Cryst. A**51** (1995) 33–37; R. H. Blessing, J. Appl. Cryst. **30** (1997) 421–426). **Program used to solve structure**: *SHELXS97* (G. M. Sheldrick, Acta Cryst. (1990) A**46** 467–473). **Program used to refine structure**: *SHELXL97* (G. M. Sheldrick (1997), University of Göttingen, Germany).

Further information: http://www.soton.ac.uk/~xservice/strat.htm

Special details:

Table 2. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
O1	–2504(4)	9215(2)	3599(2)	65(1)	1
O2	–1453(4)	8334(2)	2392(2)	87(1)	1
O3	3781(3)	7451(2)	1887(2)	72(1)	1
O4	2641(4)	8924(3)	2413(2)	88(1)	1
O5	–1776(3)	5566(2)	3895(2)	64(1)	1
O6	–4353(4)	6580(3)	4367(2)	90(1)	1
O7	–4581(4)	6704(3)	7498(2)	108(1)	1
O8	–2256(5)	5446(3)	7202(2)	100(1)	1
N1	–558(4)	7854(2)	6650(2)	56(1)	1
N2	115(4)	8221(2)	4754(2)	50(1)	1
C1	–1428(5)	8137(3)	5702(2)	55(1)	1
C2	–2650(5)	7219(3)	5751(2)	55(1)	1
C3	–1526(5)	6690(3)	4916(2)	48(1)	1
C4	–278(5)	7642(3)	4078(2)	46(1)	1
C5	1839(5)	7861(3)	5184(2)	66(1)	1
C6	1341(5)	8203(3)	6198(3)	71(1)	1
C7	–1442(5)	8434(3)	3241(3)	53(1)	1
C8	–3746(6)	9945(4)	2913(3)	81(1)	1
C9	1441(4)	7125(3)	3462(2)	53(1)	1
C10	2620(5)	7959(4)	2545(3)	58(1)	1
C11	5044(6)	8154(4)	972(3)	95(2)	1
C12	–2746(6)	6286(3)	4381(3)	56(1)	1
C13	–2720(6)	5279(3)	3205(3)	76(1)	1
C14	–3082(6)	6331(4)	6887(3)	71(1)	1
C15	–5113(8)	5925(5)	8616(3)	170(3)	1
C16	–1561(6)	8408(3)	7446(2)	70(1)	1
C17	–820(5)	7948(4)	8506(3)	64(1)	1
C18	–595(7)	6826(4)	9058(3)	92(2)	1
C19	41(7)	6412(4)	10041(3)	100(2)	1
C20	456(6)	7152(4)	10466(3)	81(1)	1
C21	233(6)	8259(4)	9937(3)	85(2)	1
C22	–418(6)	8677(3)	8952(3)	70(1)	1

Table 3. Bond lengths [Å] and angles [°].

O1–C7	1.333(4)
O1–C8	1.452(4)
O2–C7	1.189(3)
O3–C10	1.346(4)
O3–C11	1.450(3)
O4–C10	1.189(4)
O5–C12	1.340(4)
O5–C13	1.462(4)
O6–C12	1.206(5)
O7–C14	1.348(5)
O7–C15	1.464(4)
O8–C14	1.189(5)
N1–C6	1.472(4)
N1–C1	1.472(4)
N1–C16	1.478(4)
N2–C4	1.465(4)
N2–C1	1.472(3)
N2–C5	1.483(4)
C1–C2	1.577(4)
C1–H1	1.0000
C2–C3	1.523(4)
C2–C14	1.527(5)
C2–H2	1.0000
C3–C12	1.536(5)
C3–C4	1.572(4)
C3–H3	1.0000
C4–C9	1.542(4)
C4–C7	1.558(5)
C5–C6	1.516(4)
C5–H5A	0.9900
C5–H5B	0.9900
C6–H6A	0.9900
C6–H6B	0.9900
C8–H8A	0.9800
C8–H8B	0.9800
C8–H8C	0.9800
C9–C10	1.497(4)
C9–H9A	0.9900
C9–H9B	0.9900
C11–H11A	0.9800
C11–H11B	0.9800
C11–H11C	0.9800
C13–H13A	0.9800
C13–H13B	0.9800
C13–H13C	0.9800
C15–H15A	0.9800
C15–H15B	0.9800
C15–H15C	0.9800
C16–C17	1.532(5)
C16–H16A	0.9900
C16–H16B	0.9900
C17–C18	1.365(5)
C17–C22	1.381(5)
C18–C19	1.400(5)
C18–H18	0.9500
C19–C20	1.376(5)
C19–H19	0.9500
C20–C21	1.343(6)
C20–H20	0.9500
C21–C22	1.408(5)
C21–H21	0.9500
C22–H22	0.9500

C7–O1–C8	116.9(3)
C10–O3–C11	116.4(3)
C12–O5–C13	115.1(3)
C14–O7–C15	114.0(4)
C6–N1–C1	106.0(2)
C6–N1–C16	111.1(3)
C1–N1–C16	112.7(3)
C4–N2–C1	109.6(3)
C4–N2–C5	118.0(3)
C1–N2–C5	107.2(2)
N2–C1–N1	105.3(3)
N2–C1–C2	106.2(2)
N1–C1–C2	115.0(3)
N2–C1–H1	110.0
N1–C1–H1	110.0
C2–C1–H1	110.0
C3–C2–C14	110.5(3)
C3–C2–C1	105.1(2)
C14–C2–C1	113.0(3)
C3–C2–H2	109.4
C14–C2–H2	109.4
C1–C2–H2	109.4
C2–C3–C12	112.6(3)
C2–C3–C4	103.5(2)
C12–C3–C4	113.4(2)
C2–C3–H3	109.1
C12–C3–H3	109.1
C4–C3–H3	109.1
N2–C4–C9	114.9(3)
N2–C4–C7	110.9(3)
C9–C4–C7	109.3(2)
N2–C4–C3	104.2(2)
C9–C4–C3	109.8(2)
C7–C4–C3	107.5(3)
N2–C5–C6	101.7(3)
N2–C5–H5A	111.4
C6–C5–H5A	111.4
N2–C5–H5B	111.4
C6–C5–H5B	111.4
H5A–C5–H5B	109.3
N1–C6–C5	101.0(3)
N1–C6–H6A	111.6
C5–C6–H6A	111.6
N1–C6–H6B	111.6
C5–C6–H6B	111.6
H6A–C6–H6B	109.4
O2–C7–O1	122.9(4)
O2–C7–C4	124.3(3)
O1–C7–C4	112.7(3)
O1–C8–H8A	109.5
O1–C8–H8B	109.5
H8A–C8–H8B	109.5
O1–C8–H8C	109.5
H8A–C8–H8C	109.5
H8B–C8–H8C	109.5
C10–C9–C4	114.7(3)
C10–C9–H9A	108.6
C4–C9–H9A	108.6
C10–C9–H9B	108.6
C4–C9–H9B	108.6
H9A–C9–H9B	107.6
O4–C10–O3	122.7(3)

O4–C10–C9	127.0(3)
O3–C10–C9	110.2(3)
O3–C11–H11A	109.5
O3–C11–H11B	109.5
H11A–C11–H11B	109.5
O3–C11–H11C	109.5
H11A–C11–H11C	109.5
H11B–C11–H11C	109.5
O6–C12–O5	123.2(4)
O6–C12–C3	125.8(4)
O5–C12–C3	110.9(4)
O5–C13–H13A	109.5
O5–C13–H13B	109.5
H13A–C13–H13B	109.5
O5–C13–H13C	109.5
H13A–C13–H13C	109.5
H13B–C13–H13C	109.5
O8–C14–O7	124.3(4)
O8–C14–C2	126.7(4)
O7–C14–C2	109.1(4)
O7–C15–H15A	109.5
O7–C15–H15B	109.5
H15A–C15–H15B	109.5
O7–C15–H15C	109.5
H15A–C15–H15C	109.5
H15B–C15–H15C	109.5
N1–C16–C17	111.3(3)
N1–C16–H16A	109.4
C17–C16–H16A	109.4
N1–C16–H16B	109.4
C17–C16–H16B	109.4
H16A–C16–H16B	108.0
C18–C17–C22	118.0(4)
C18–C17–C16	122.1(3)
C22–C17–C16	119.9(4)
C17–C18–C19	121.8(4)
C17–C18–H18	119.1
C19–C18–H18	119.1
C20–C19–C18	119.4(4)
C20–C19–H19	120.3
C18–C19–H19	120.3
C21–C20–C19	119.6(4)
C21–C20–H20	120.2
C19–C20–H20	120.2
C20–C21–C22	121.1(4)
C20–C21–H21	119.5
C22–C21–H21	119.5
C17–C22–C21	120.1(4)
C17–C22–H22	119.9
C21–C22–H22	119.9

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

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O1	67(2)	73(2)	62(1)	–30(1)	–26(1)	17(2)
O2	105(2)	109(2)	57(2)	–45(2)	–27(2)	21(2)
O3	65(2)	91(2)	60(1)	–39(1)	12(1)	–9(2)
O4	97(2)	73(2)	85(2)	–32(2)	16(2)	–20(2)
O5	64(2)	71(2)	67(1)	–39(1)	–10(1)	0(2)
O6	51(2)	133(3)	115(2)	–81(2)	–16(2)	2(2)
O7	83(2)	167(3)	55(2)	–37(2)	12(2)	0(2)
O8	129(3)	85(2)	63(2)	–13(2)	5(2)	–7(2)
N1	54(2)	75(2)	46(2)	–32(2)	–15(2)	12(2)
N2	42(2)	63(2)	49(2)	–26(1)	–11(2)	2(2)
C1	55(3)	68(3)	45(2)	–29(2)	–12(2)	8(2)
C2	48(2)	77(3)	41(2)	–26(2)	–7(2)	9(2)
C3	41(2)	59(2)	39(2)	–18(2)	–3(2)	6(2)
C4	50(2)	51(2)	39(2)	–21(2)	–2(2)	–4(2)
C5	58(3)	88(3)	58(2)	–31(2)	–7(2)	–10(2)
C6	72(3)	90(3)	62(2)	–33(2)	–24(2)	–4(3)
C7	49(3)	72(3)	44(2)	–29(2)	–5(2)	–6(2)
C8	75(3)	83(3)	86(3)	–28(2)	–32(2)	15(3)
C9	50(2)	60(2)	47(2)	–23(2)	–2(2)	1(2)
C10	52(3)	76(3)	54(2)	–31(2)	–10(2)	–6(3)
C11	81(3)	131(4)	59(2)	–35(2)	28(2)	–32(3)
C12	54(3)	65(3)	47(2)	–17(2)	–4(2)	–11(2)
C13	87(3)	85(3)	72(2)	–40(2)	–21(2)	–11(3)
C14	63(3)	97(4)	49(2)	–31(3)	4(2)	–10(3)
C15	151(5)	262(8)	47(2)	–17(3)	30(3)	–43(5)
C16	83(3)	79(3)	57(2)	–39(2)	–25(2)	23(2)
C17	74(3)	77(3)	47(2)	–33(2)	–11(2)	1(3)
C18	139(5)	77(4)	81(3)	–44(3)	–55(3)	26(3)
C19	148(5)	91(4)	69(3)	–27(3)	–42(3)	7(4)
C20	92(4)	104(4)	48(2)	–28(3)	–16(2)	–3(3)
C21	105(4)	107(4)	55(2)	–39(2)	–11(3)	–28(4)
C22	89(3)	73(3)	50(2)	–25(2)	–6(2)	–13(3)

Table 5. Hydrogen coordinates [$\times 10^4$] and isotropic displacement parameters [$Å^2 \times 10^3$].

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
H1	–2173	8873	5595	66	1
H2	–3832	7590	5522	66	1
H3	–719	6040	5288	58	1
H5A	2900	8255	4650	80	1
H5B	2132	7042	5374	80	1
H6A	2168	7802	6714	86	1
H6B	1377	9021	6006	86	1
H8A	–4452	10480	3259	121	1
H8B	–4599	9493	2825	121	1
H8C	–3026	10353	2192	121	1
H9A	2206	6657	3995	64	1
H9B	1031	6625	3161	64	1
H11A	5812	7702	548	142	1
H11B	5836	8478	1249	142	1
H11C	4337	8758	500	142	1
H13A	–1912	4751	2885	114	1
H13B	–3018	5960	2617	114	1
H13C	–3862	4933	3652	114	1
H15A	–6210	6250	9009	255	1
H15B	–4094	5788	8999	255	1
H15C	–5392	5216	8591	255	1
H16A	–2892	8287	7618	84	1
H16B	–1429	9224	7114	84	1
H18	–879	6312	8768	110	1
H19	184	5627	10411	120	1
H20	897	6881	11129	97	1
H21	519	8768	10232	101	1
H22	–582	9463	8594	85	1

Crystallographic data tables for compound **8a** (compound identification code 01src383)

Table 1. Crystal data and structure refinement.

Identification code	**01src383**	
Empirical formula	$C_{21}H_{25}N_3O_6$	
Formula weight	415.44	
Temperature	120(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*–1	
Unit cell dimensions	a = 11.331(2) Å	α = 100.18(3)°
	b = 13.023(3) Å	β = 108.98(3)°
	c = 15.005(3) Å	γ = 90.44(3)°
Volume	2055.7(7) Å^3	
Z	4	
Density (calculated)	1.342 Mg / m^3	
Absorption coefficient	0.099 mm^{-1}	
F(000)	880	
Crystal	Block; colourless	
Crystal size	0.10 × 0.04 × 0.02 mm^3	
θ range for data collection	1.91 – 20.76°	
Index ranges	$-11 \le h \le 10$, $-12 \le k \le 12$, $-14 \le l \le 14$	
Reflections collected	7560	
Independent reflections	4048 [R_{int} = 0.0718]	
Completeness to θ = 20.76°	94.6 %	
Absorption correction	Semi–empirical from equivalents	
Max. and min. transmission	0.9980 and 0.9901	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4048 / 6 / 549	
Goodness-of-fit on F^2	1.054	
Final *R* indices [$F^2 > 2\sigma(F^2)$]	*R1* = 0.1033, *wR2* = 0.2628	
R indices (all data)	*R1* = 0.1437, *wR2* = 0.3028	
Extinction coefficient	0.020(4)	
Largest diff. peak and hole	0.712 and –0.395 e Å^{-3}	

Diffractometer: *Enraf Nonius KappaCCD* area detector (ϕ scans and ω scans to fill *Ewald* sphere). **Data collection and cell refinement**: *Denzo* (Z. Otwinowski & W. Minor, *Methods in Enzymology* (1997) Vol. **276**: *Macromolecular Crystallography*, part A, pp. 307–326; C. W. Carter, Jr. & R. M. Sweet, Eds., Academic Press). **Absorption correction**: *SORTAV* (R. H. Blessing, Acta Cryst. A**51** (1995) 33–37; R. H. Blessing, J. Appl. Cryst. **30** (1997) 421–426). **Program used to solve structure**: *SHELXS97* (G. M. Sheldrick, Acta Cryst. (1990) A**46** 467–473). **Program used to refine structure**: *SHELXL97* (G. M. Sheldrick (1997), University of Göttingen, Germany).

Further information: http://www.soton.ac.uk/~xservice/strat.htm

Special details:

R factors poor due to crystal size and quality

Table 2. Atomic coordinates [$\times 10^4$], equivalent isotropic displacement parameters [$Å^2 \times 10^3$] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
C1	–4815(9)	12466(7)	–7205(6)	37(2)	1
C2	–6062(9)	12031(8)	–7615(7)	52(3)	1
C3	–6784(10)	12187(8)	–8509(7)	55(3)	1
C4	–6320(12)	12769(9)	–9017(8)	67(3)	1
C5	–5085(11)	13198(8)	–8625(8)	64(3)	1
C6	–4349(10)	13057(7)	–7711(6)	44(3)	1
C7	–4017(8)	12314(7)	–6225(6)	37(2)	1
C8	–5275(8)	13258(6)	–5356(6)	31(2)	1
C9	–4997(7)	13793(6)	–4322(6)	32(2)	1
C10	–3187(7)	13066(6)	–4516(6)	27(2)	1
C11	–2031(8)	13894(6)	–4089(6)	28(2)	1
C12	–2017(8)	14603(7)	–4783(6)	29(2)	1
C13	–1428(9)	14786(7)	–6112(7)	40(2)	1
C14	–2196(8)	14494(7)	–3167(6)	30(2)	1
C15	–959(9)	14891(6)	–2386(6)	30(2)	1
C16	–24(12)	15852(11)	–843(9)	92(4)	1
C17	–3061(7)	13759(6)	–2909(6)	25(2)	1
C18	–3728(8)	14229(7)	–2220(6)	34(2)	1
C19	–3412(8)	13617(7)	–1426(6)	32(2)	1
C20	–2336(8)	12921(7)	–2426(6)	29(2)	1
C21	–2048(9)	12149(7)	–974(7)	44(3)	1
C27	147(8)	11529(7)	2691(6)	35(2)	1
C28	–1060(9)	11846(8)	2287(7)	51(3)	1
C29	–1834(10)	11296(9)	1411(7)	59(3)	1
C30	–1441(10)	10497(9)	914(7)	52(3)	1
C31	–246(11)	10204(8)	1292(7)	54(3)	1
C32	514(9)	10726(8)	2186(7)	45(3)	1
C33	994(8)	12117(7)	3656(6)	36(2)	1
C34	–293(8)	11583(7)	4550(6)	30(2)	1
C35	–30(7)	11495(6)	5569(6)	27(2)	1
C36	1797(7)	12124(6)	5384(6)	27(2)	1
C37	2961(7)	11486(6)	5849(6)	24(2)	1
C38	3006(8)	10497(7)	5181(6)	25(2)	1
C39	3554(9)	9738(7)	3846(6)	39(2)	1
C40	2778(7)	11260(6)	6758(5)	26(2)	1
C41	3980(9)	11229(6)	7574(6)	28(2)	1
C42	4854(9)	11072(10)	9202(7)	65(3)	1
C43	1884(8)	12095(6)	6990(6)	25(2)	1
C44	1190(8)	11892(7)	7647(6)	36(2)	1
C45	1511(8)	12799(7)	8466(6)	34(2)	1
C46	2640(8)	13142(7)	7520(6)	32(2)	1
C47	2926(9)	14464(7)	8995(6)	46(3)	1
N1	–4039(6)	13174(5)	–5454(5)	28(2)	1
N2	–3901(6)	13208(5)	–3842(5)	30(2)	1
N3	–2581(6)	12902(5)	–1587(5)	31(2)	1
N4	948(6)	11601(5)	4450(5)	29(2)	1
N5	1072(6)	12248(5)	6048(5)	28(2)	1
N6	2371(7)	13478(5)	8348(5)	31(2)	1
O1	–2323(5)	15478(5)	–4728(4)	36(2)	1
O2	–1525(5)	14138(4)	–5438(4)	33(2)	1
O3	51(6)	14792(5)	–2446(4)	45(2)	1
O4	–1147(6)	15382(6)	–1602(5)	74(3)	1
O5	–3785(6)	13711(5)	–747(5)	49(2)	1
O6	–1636(5)	12348(5)	–2715(4)	37(2)	1
O7	2644(5)	9628(4)	5224(4)	32(2)	1
O8	3475(5)	10657(4)	4513(4)	32(2)	1
O9	5006(6)	11300(5)	7526(4)	41(2)	1
O10	3770(6)	11148(6)	8379(4)	51(2)	1
O11	1114(6)	12964(5)	9131(5)	50(2)	1
O12	3364(6)	13608(4)	7251(4)	37(2)	1

Table 3. Bond lengths [Å] and angles [°].

C1–C6	1.382(13)
C1–C2	1.412(13)
C1–C7	1.503(12)
C2–C3	1.377(13)
C3–C4	1.375(15)
C4–C5	1.398(15)
C5–C6	1.399(13)
C7–N1	1.467(10)
C8–N1	1.458(10)
C8–C9	1.513(11)
C9–N2	1.506(10)
C10–N1	1.456(10)
C10–N2	1.476(10)
C10–C11	1.573(11)
C11–C12	1.513(12)
C11–C14	1.531(11)
C12–O1	1.190(9)
C12–O2	1.339(10)
C13–O2	1.457(10)
C14–C15	1.521(12)
C14–C17	1.546(11)
C15–O3	1.183(9)
C15–O4	1.317(10)
C16–O4	1.445(14)
C17–N2	1.466(10)
C17–C20	1.517(12)
C17–C18	1.519(11)
C18–C19	1.497(12)
C19–O5	1.212(10)
C19–N3	1.376(11)
C20–O6	1.222(10)
C20–N3	1.379(11)
C21–N3	1.462(11)
C27–C32	1.332(12)
C27–C28	1.402(13)
C27–C33	1.515(12)
C28–C29	1.385(14)
C29–C30	1.337(14)
C30–C31	1.373(14)
C31–C32	1.385(13)
C33–N4	1.482(10)
C34–N4	1.462(10)
C34–C35	1.485(11)
C35–N5	1.478(10)
C36–N4	1.459(10)
C36–N5	1.474(10)
C36–C37	1.595(11)
C37–C38	1.498(11)
C37–C40	1.522(11)
C38–O7	1.221(9)
C38–O8	1.323(10)
C39–O8	1.440(10)
C40–C41	1.513(12)
C40–C43	1.562(11)
C41–O9	1.192(9)
C41–O10	1.327(10)
C42–O10	1.450(11)
C43–N5	1.464(10)
C43–C44	1.502(11)
C43–C46	1.543(12)
C44–C45	1.492(12)
C45–O11	1.209(10)

C45–N6	1.385(12)
C46–O12	1.224(10)
C46–N6	1.372(11)
C47–N6	1.462(11)

C6–C1–C2	118.8(9)
C6–C1–C7	120.4(8)
C2–C1–C7	120.8(8)
C3–C2–C1	120.0(9)
C2–C3–C4	121.3(10)
C3–C4–C5	119.4(10)
C4–C5–C6	119.7(11)
C1–C6–C5	120.7(9)
N1–C7–C1	113.2(7)
N1–C8–C9	103.5(6)
N2–C9–C8	99.9(6)
N1–C10–N2	107.5(6)
N1–C10–C11	114.7(6)
N2–C10–C11	107.1(6)
C12–C11–C14	112.2(7)
C12–C11–C10	111.5(7)
C14–C11–C10	103.8(6)
O1–C12–O2	124.0(8)
O1–C12–C11	125.3(8)
O2–C12–C11	110.6(7)
C15–C14–C11	113.0(7)
C15–C14–C17	116.3(7)
C11–C14–C17	105.2(7)
O3–C15–O4	122.8(9)
O3–C15–C14	126.4(8)
O4–C15–C14	110.8(8)
N2–C17–C20	105.5(6)
N2–C17–C18	114.1(7)
C20–C17–C18	103.2(6)
N2–C17–C14	104.1(6)
C20–C17–C14	111.1(6)
C18–C17–C14	118.3(7)
C19–C18–C17	106.8(7)
O5–C19–N3	124.0(8)
O5–C19–C18	128.3(9)
N3–C19–C18	107.7(8)
O6–C20–N3	124.3(8)
O6–C20–C17	126.6(8)
N3–C20–C17	109.1(7)
C32–C27–C28	117.7(9)
C32–C27–C33	122.4(9)
C28–C27–C33	119.9(9)
C29–C28–C27	119.5(10)
C30–C29–C28	121.6(10)
C29–C30–C31	119.0(10)
C30–C31–C32	119.5(10)
C27–C32–C31	122.6(10)
N4–C33–C27	111.7(7)
N4–C34–C35	103.5(6)
N5–C35–C34	100.9(6)
N4–C36–N5	106.8(6)
N4–C36–C37	115.0(6)
N5–C36–C37	105.9(6)
C38–C37–C40	111.4(7)
C38–C37–C36	111.6(6)
C40–C37–C36	104.9(6)
O7–C38–O8	122.2(7)
O7–C38–C37	124.9(8)

O8–C38–C37	112.8(7)
C41–C40–C37	114.5(7)
C41–C40–C43	114.8(6)
C37–C40–C43	104.5(6)
O9–C41–O10	122.8(8)
O9–C41–C40	125.2(8)
O10–C41–C40	112.0(8)
N5–C43–C44	113.9(7)
N5–C43–C46	107.7(6)
C44–C43–C46	102.8(7)
N5–C43–C40	104.3(6)
C44–C43–C40	117.9(7)
C46–C43–C40	110.0(7)
C45–C44–C43	107.4(7)
O11–C45–N6	123.1(9)
O11–C45–C44	128.6(9)
N6–C45–C44	108.2(8)
O12–C46–N6	124.8(8)
O12–C46–C43	126.0(8)
N6–C46–C43	109.2(8)
C10–N1–C8	106.2(6)
C10–N1–C7	113.1(6)
C8–N1–C7	112.4(6)
C17–N2–C10	107.8(6)
C17–N2–C9	115.5(6)
C10–N2–C9	103.8(6)
C19–N3–C20	113.1(7)
C19–N3–C21	124.8(7)
C20–N3–C21	122.1(7)
C36–N4–C34	105.4(6)
C36–N4–C33	112.9(7)
C34–N4–C33	112.6(6)
C43–N5–C36	108.7(6)
C43–N5–C35	116.2(6)
C36–N5–C35	104.7(6)
C46–N6–C45	112.3(8)
C46–N6–C47	122.0(8)
C45–N6–C47	125.6(7)
C12–O2–C13	114.1(6)
C15–O4–C16	114.6(8)
C38–O8–C39	115.9(6)
C41–O10–C42	116.8(7)

Symmetry transformations used to generate equivalent atoms: