APPENDIX 4

X-Ray Crystallography Reports Relevant to Chapter 4:

Progress Toward the Total Synthesis of Acutumine[†]

[†] The work disclosed in this appendix for the X-ray crystallographic analysis of **230**, **238**, and **255** was completed entirely by Larry Henling and Dr. Michael Day in the Caltech X-ray crystallography lab.

A4.1. Crystal Structure analysis of bromohydrin 230.

Figure A4.1. Bromohydrin **230**. Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 821739.

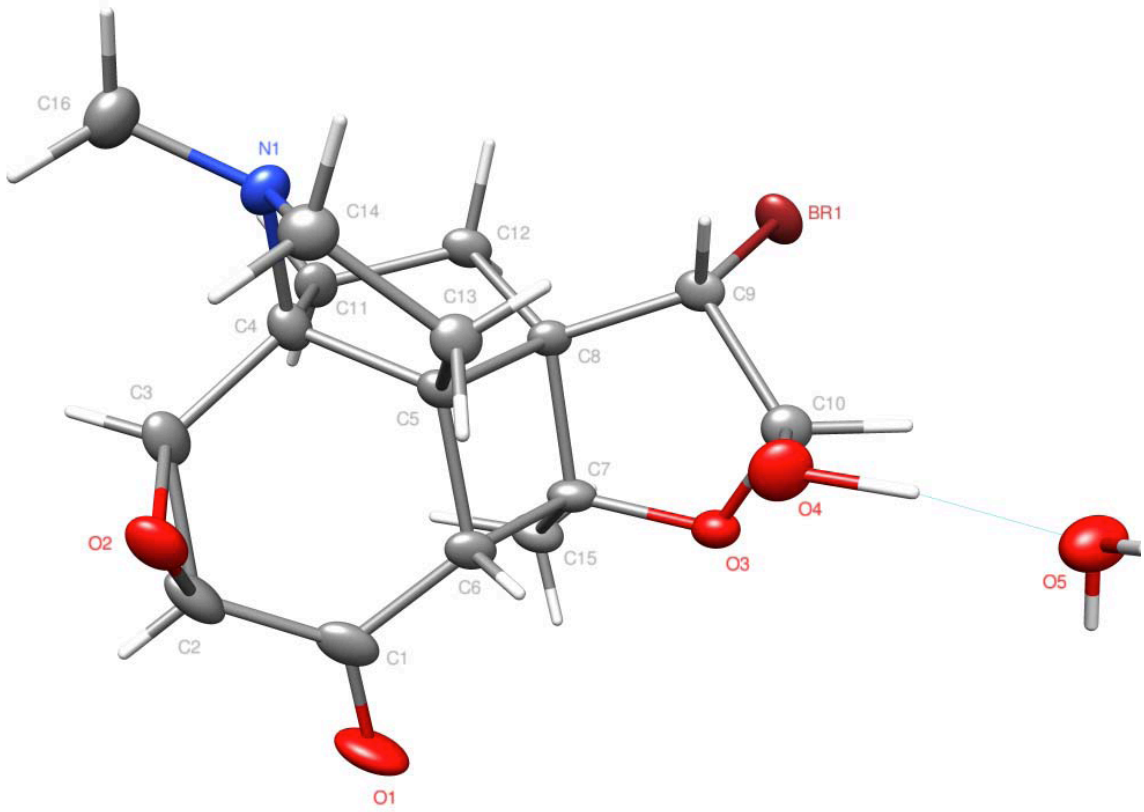


Table A4.1. Crystal data and structure refinement for bromohydrin **230**.

Empirical formula	$C_{16}H_{20}BrNO_4 \bullet H_2O$
Formula weight	388.26
Crystallization Solvent	Diethyl ether/hexanes
Crystal Habit	Plate
Crystal size	0.41 x 0.21 x 0.02 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9272 reflections used in lattice determination	2.31 to 25.62°	
Unit cell dimensions	a = 7.2279(3) Å b = 12.4336(5) Å c = 17.6522(7) Å	α= 90° β= 90° γ = 90°
Volume	1586.38(11) $Å^3$	
Z	4	
Crystal system	Orthorhombic	
Space group	$P\ 2_12_12_1$	
Density (calculated)	1.626 Mg/m^3	
F(000)	800	
Data collection program	Bruker APEX2 v2009.7-0	
θ range for data collection	2.00 to 32.34°	
Completeness to θ = 32.34°	96.5 %	
Index ranges	$-10 \le h \le 10$, $-18 \le k \le 18$, $-26 \le l \le 25$	
Data collection scan type	ω scans; 10 settings	
Data reduction program	Bruker SAINT-Plus v7.66A	
Reflections collected	40470	
Independent reflections	5378 [R_{int}= 0.0867]	
Absorption coefficient	2.617 mm^{-1}	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7464 and 0.5033	

Table A4.1. (continued)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	5378 / 0 / 296
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.446
Final R indices [$I>2\sigma(I)$, 4263 reflections]	R1 = 0.0481, *w*R2 = 0.0693
R indices (all data)	R1 = 0.0722, *w*R2 = 0.0719
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.002
Average shift/error	0.000
Absolute structure determination	Anomalous differences
Absolute structure parameter	0.014(8)
Largest diff. peak and hole	1.337 and -1.269 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table A4.2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for RUN03 (CCDC 821739). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Br(1)	2711(1)	7317(1)	6311(1)	22(1)
O(1)	10028(3)	9360(2)	8022(1)	35(1)
O(2)	10424(3)	10993(2)	6421(1)	26(1)
O(3)	6896(2)	7229(2)	7034(1)	19(1)
O(4)	8161(3)	6973(2)	5832(1)	27(1)
N(1)	6744(3)	10932(2)	5416(1)	20(1)
C(1)	9499(4)	9698(3)	7407(2)	24(1)
C(2)	9762(4)	10833(3)	7189(2)	26(1)
C(3)	8553(4)	11292(3)	6596(2)	21(1)
C(4)	7033(3)	10649(2)	6224(2)	18(1)
C(5)	7549(3)	9440(2)	6164(1)	15(1)
C(6)	8568(4)	8965(3)	6854(2)	17(1)
C(7)	6774(4)	8365(2)	7124(2)	17(1)
C(8)	5722(3)	8816(2)	6408(2)	14(1)
C(9)	5200(3)	7802(3)	5993(2)	16(1)
C(10)	6550(3)	6935(2)	6265(2)	19(1)
C(11)	5229(4)	10722(3)	6674(2)	19(1)
C(12)	4221(4)	9662(3)	6521(2)	18(1)
C(13)	8419(4)	9312(2)	5377(2)	17(1)
C(14)	8237(4)	10395(3)	4995(2)	22(1)
C(15)	6041(4)	8593(3)	7910(2)	22(1)
C(16)	6597(5)	12081(3)	5251(2)	28(1)
O(5)	8790(3)	4904(2)	5492(2)	28(1)

Table A4.3. Bond lengths [Å] and angles [°] for RUN03 (CCDC 821739).

Br(1)-C(9)	1.979(2)	C(7)-O(3)-C(10)	110.4(2)
O(1)-C(1)	1.225(4)	C(10)-O(4)-H(4)	100.3(19)
O(2)-C(3)	1.437(3)	C(16)-N(1)-C(14)	113.3(2)
O(2)-C(2)	1.452(4)	C(16)-N(1)-C(4)	115.7(2)
O(3)-C(7)	1.424(3)	C(14)-N(1)-C(4)	105.9(2)
O(3)-C(10)	1.429(3)	O(1)-C(1)-C(2)	121.3(3)
O(4)-C(10)	1.394(3)	O(1)-C(1)-C(6)	120.6(3)
O(4)-H(4)	1.14(4)	C(2)-C(1)-C(6)	118.1(3)
N(1)-C(16)	1.462(4)	O(2)-C(2)-C(1)	114.7(3)
N(1)-C(14)	1.471(4)	O(2)-C(2)-C(3)	58.70(19)
N(1)-C(4)	1.482(4)	C(1)-C(2)-C(3)	118.6(3)
C(1)-C(2)	1.475(5)	O(2)-C(2)-H(2)	110(2)
C(1)-C(6)	1.495(4)	C(1)-C(2)-H(2)	119(2)
C(2)-C(3)	1.479(5)	C(3)-C(2)-H(2)	120(2)
C(2)-H(2)	0.86(3)	O(2)-C(3)-C(2)	59.73(19)
C(3)-C(4)	1.509(4)	O(2)-C(3)-C(4)	117.0(2)
C(3)-H(3)	0.93(3)	C(2)-C(3)-C(4)	122.3(3)
C(4)-C(11)	1.530(4)	O(2)-C(3)-H(3)	110.7(16)
C(4)-C(5)	1.553(4)	C(2)-C(3)-H(3)	112.8(16)
C(5)-C(13)	1.533(3)	C(4)-C(3)-H(3)	119.5(16)
C(5)-C(6)	1.542(4)	N(1)-C(4)-C(3)	113.3(2)
C(5)-C(8)	1.591(3)	N(1)-C(4)-C(11)	111.4(2)
C(6)-C(7)	1.570(4)	C(3)-C(4)-C(11)	111.3(2)
C(6)-H(6)	0.80(3)	N(1)-C(4)-C(5)	101.4(2)
C(7)-C(15)	1.512(4)	C(3)-C(4)-C(5)	111.5(2)
C(7)-C(8)	1.578(4)	C(11)-C(4)-C(5)	107.3(2)
C(8)-C(9)	1.507(4)	C(13)-C(5)-C(6)	118.7(2)
C(8)-C(12)	1.524(4)	C(13)-C(5)-C(4)	105.1(2)
C(9)-C(10)	1.531(4)	C(6)-C(5)-C(4)	115.6(2)
C(9)-H(9)	0.85(3)	C(13)-C(5)-C(8)	122.4(2)
C(10)-H(10)	1.118(17)	C(6)-C(5)-C(8)	89.73(19)
C(11)-C(12)	1.529(4)	C(4)-C(5)-C(8)	104.72(19)
C(11)-H(11A)	0.98(3)	C(1)-C(6)-C(5)	119.8(3)
C(11)-H(11B)	0.86(3)	C(1)-C(6)-C(7)	117.6(2)
C(12)-H(12A)	0.97(3)	C(5)-C(6)-C(7)	91.6(2)
C(12)-H(12B)	1.05(3)	C(1)-C(6)-H(6)	109(2)
C(13)-C(14)	1.512(4)	C(5)-C(6)-H(6)	109(2)
C(13)-H(13A)	1.00(4)	C(7)-C(6)-H(6)	108(2)
C(13)-H(13B)	0.86(3)	O(3)-C(7)-C(15)	108.1(2)
C(14)-H(14A)	0.98(3)	O(3)-C(7)-C(6)	112.7(2)
C(14)-H(14B)	1.00(3)	C(15)-C(7)-C(6)	118.6(3)
C(15)-H(15A)	0.92(4)	O(3)-C(7)-C(8)	107.0(2)
C(15)-H(15B)	1.12(3)	C(15)-C(7)-C(8)	120.0(2)
C(15)-H(15C)	0.93(3)	C(6)-C(7)-C(8)	89.20(19)
C(16)-H(16A)	1.00(3)	C(9)-C(8)-C(12)	117.6(2)
C(16)-H(16B)	0.97(4)	C(9)-C(8)-C(7)	102.3(2)
C(16)-H(16C)	0.98(3)	C(12)-C(8)-C(7)	118.9(2)
O(5)-H(5A)	0.79(4)	C(9)-C(8)-C(5)	119.0(2)
O(5)-H(5B)	0.86(5)	C(12)-C(8)-C(5)	106.8(2)
		C(7)-C(8)-C(5)	89.46(18)
C(3)-O(2)-C(2)	61.6(2)	C(8)-C(9)-C(10)	106.1(2)

C(8)-C(9)-Br(1)	110.18(17)	C(14)-C(13)-H(13A)	111(2)
C(10)-C(9)-Br(1)	106.04(19)	C(5)-C(13)-H(13A)	114(2)
C(8)-C(9)-H(9)	112(2)	C(14)-C(13)-H(13B)	110.1(17)
C(10)-C(9)-H(9)	115.2(19)	C(5)-C(13)-H(13B)	112.6(17)
Br(1)-C(9)-H(9)	107.2(19)	H(13A)-C(13)-H(13B)	104(3)
O(4)-C(10)-O(3)	111.5(2)	N(1)-C(14)-C(13)	104.1(2)
O(4)-C(10)-C(9)	109.7(2)	N(1)-C(14)-H(14A)	108.3(19)
O(3)-C(10)-C(9)	103.3(2)	C(13)-C(14)-H(14A)	110.0(19)
O(4)-C(10)-H(10)	103.2(8)	N(1)-C(14)-H(14B)	103.5(19)
O(3)-C(10)-H(10)	111.7(9)	C(13)-C(14)-H(14B)	110.8(17)
C(9)-C(10)-H(10)	117.6(8)	H(14A)-C(14)-H(14B)	119(2)
C(12)-C(11)-C(4)	105.3(2)	C(7)-C(15)-H(15A)	109(2)
C(12)-C(11)-H(11A)	113.0(16)	C(7)-C(15)-H(15B)	114.7(15)
C(4)-C(11)-H(11A)	109.1(16)	H(15A)-C(15)-H(15B)	111(2)
C(12)-C(11)-H(11B)	113(2)	C(7)-C(15)-H(15C)	109.5(16)
C(4)-C(11)-H(11B)	105(2)	H(15A)-C(15)-H(15C)	104(3)
H(11A)-C(11)-H(11B)	111(3)	H(15B)-C(15)-H(15C)	108(2)
C(8)-C(12)-C(11)	106.2(2)	N(1)-C(16)-H(16A)	118.8(18)
C(8)-C(12)-H(12A)	107.0(19)	N(1)-C(16)-H(16B)	106(2)
C(11)-C(12)-H(12A)	110.9(19)	H(16A)-C(16)-H(16B)	104(2)
C(8)-C(12)-H(12B)	106.1(17)	N(1)-C(16)-H(16C)	109(2)
C(11)-C(12)-H(12B)	113.2(17)	H(16A)-C(16)-H(16C)	108(3)
H(12A)-C(12)-H(12B)	113(2)	H(16B)-C(16)-H(16C)	111(3)
C(14)-C(13)-C(5)	106.0(2)	H(5A)-O(5)-H(5B)	117(4)

Table A4.4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for RUN03 (CCDC 821739). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	109(1)	272(2)	281(1)	-65(1)	38(1)	-28(1)
O(1)	253(12)	613(19)	192(12)	29(11)	-85(10)	-97(12)
O(2)	114(8)	342(14)	314(13)	-28(11)	11(9)	-29(8)
O(3)	157(9)	229(11)	189(9)	26(9)	-21(7)	5(9)
O(4)	160(10)	315(15)	334(12)	3(10)	88(8)	36(9)
N(1)	161(11)	186(15)	247(13)	41(11)	18(9)	18(10)
C(1)	87(12)	400(20)	230(16)	-42(15)	21(11)	1(13)
C(2)	144(14)	370(20)	268(18)	-132(15)	-8(13)	-41(14)
C(3)	142(13)	211(19)	281(16)	-68(13)	43(11)	0(12)
C(4)	142(11)	195(15)	211(14)	-37(12)	24(11)	-13(10)
C(5)	95(11)	195(14)	150(13)	-3(9)	5(10)	-6(11)
C(6)	93(12)	233(19)	172(15)	-19(13)	14(10)	41(12)
C(7)	123(13)	242(19)	142(13)	24(12)	-12(10)	18(11)
C(8)	100(11)	183(16)	142(14)	-6(12)	10(10)	26(10)
C(9)	93(10)	231(17)	146(12)	22(13)	41(9)	-17(12)
C(10)	138(10)	243(16)	191(14)	-22(14)	43(12)	21(10)
C(11)	121(12)	180(18)	268(17)	-23(14)	19(11)	31(12)
C(12)	108(12)	258(18)	173(15)	-18(12)	16(10)	19(12)
C(13)	136(12)	204(18)	161(14)	-20(13)	46(11)	13(12)
C(14)	180(14)	270(20)	214(16)	29(14)	37(11)	-3(12)
C(15)	181(14)	330(20)	159(15)	15(14)	-5(11)	-18(14)
C(16)	252(15)	240(20)	362(19)	67(15)	27(14)	18(14)
O(5)	298(12)	341(16)	210(13)	-13(11)	2(11)	106(10)

Table A4.5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for RUN03 (CCDC 821739).

	x	y	z	U_{iso}
H(4)	8310(50)	6090(30)	5680(20)	51(11)
H(2)	10340(40)	11260(20)	7496(16)	17(8)
H(3)	8440(30)	12040(20)	6632(14)	6(7)
H(6)	9290(40)	8530(20)	6709(16)	10(8)
H(9)	5160(40)	7890(20)	5518(16)	13(7)
H(10)	6110(20)	6077(14)	6215(9)	-32(3)
H(11A)	5520(40)	10830(20)	7208(15)	3(7)
H(11B)	4650(40)	11270(30)	6490(18)	27(10)
H(12A)	3500(40)	9440(20)	6959(18)	34(10)
H(12B)	3440(40)	9670(20)	6015(16)	24(8)
H(13A)	9740(50)	9080(30)	5390(19)	36(10)
H(13B)	7880(40)	8820(20)	5110(14)	9(7)
H(14A)	9380(50)	10810(30)	5064(16)	22(9)
H(14B)	7710(50)	10310(20)	4472(17)	33(8)
H(15A)	4910(50)	8260(30)	7966(19)	37(10)
H(15B)	5940(40)	9460(20)	8057(15)	12(7)
H(15C)	6790(40)	8260(20)	8268(15)	9(7)
H(16A)	7690(40)	12560(30)	5357(16)	39(9)
H(16B)	5630(50)	12360(30)	5580(20)	43(10)
H(16C)	6260(50)	12170(30)	4719(19)	39(10)
H(5A)	9120(50)	4730(30)	5900(20)	36(11)
H(5B)	9480(70)	4720(40)	5120(20)	68(16)

Table A4.6. Hydrogen bonds for RUN03 (CCDC 821739) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(4)-H(4)...O(5)	1.14(4)	1.55(4)	2.680(3)	173(3)
O(5)-H(5A)...O(1)#1	0.79(4)	2.05(4)	2.841(3)	177(4)
O(5)-H(5B)...N(1)#2	0.86(5)	2.06(5)	2.865(3)	157(4)

Symmetry transformations used to generate equivalent atoms:
#1 -x+2,y-1/2,-z+3/2
#2 x+1/2,-y+3/2,-z+1

A4.2. Crystal Structure analysis of ketal 238.

Figure A4.2. Ketal **238**. Crystallographic data have been deposited at the CCDC, 12 Union Road, Cambridge CB2 1EZ, UK and copies can be obtained on request, free of charge, by quoting the publication citation and the deposition number 845603.

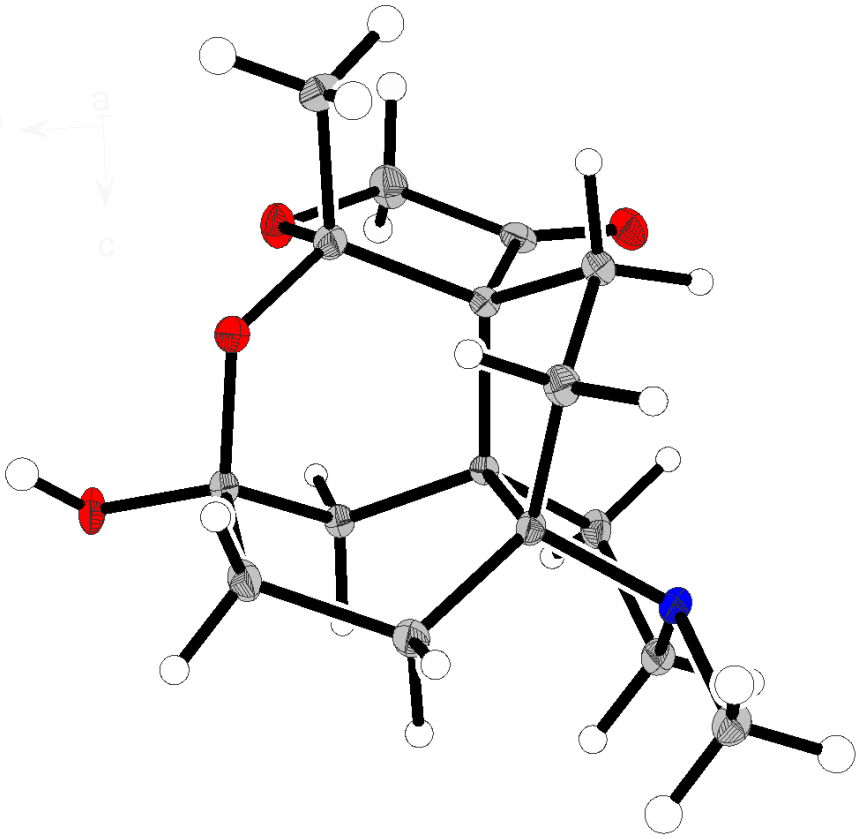
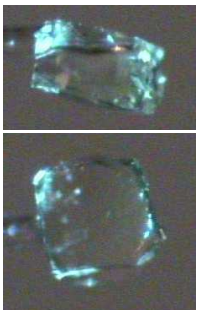


Table A4.7. Crystal data and structure refinement for ketal **238.**



Empirical formula	$C_{16}H_{23}NO_4$
Formula weight	293.35
Crystallization Solvent	Dichlromethane/iso-octane
Crystal Habit	Block
Crystal size	0.32 x 0.31 x 0.19 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9695 reflections used in lattice determination	2.51 to 41.09°	
Unit cell dimensions	a = 12.1329(5) Å b = 14.5974(6) Å c = 16.4163(7) Å	α= 90° β= 90° γ = 90°
Volume	2907.5(2) $Å^3$	
Z	8	
Crystal system	Orthorhombic	
Space group	$P\ 2_12_12_1$	
Density (calculated)	1.340 Mg/m^3	
F(000)	1264	
Data collection program	Bruker APEX2 v2009.7-0	
θ range for data collection	1.87 to 43.45°	
Completeness to θ = 43.45°	98.8 %	
Index ranges	$-23 \le h \le 23, -26 \le k \le 28, -30 \le l \le 31$	
Data collection scan type	ω scans; 10 settings	
Data reduction program	Bruker SAINT-Plus v7.68A	
Reflections collected	125921	
Independent reflections	21388 [R_{int}= 0.0565]	
Absorption coefficient	0.096 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9820 and 0.9700	

Table A4.7. (continued)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	21388 / 0 / 385
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.728
Final R indices [$I>2\sigma(I)$, 15751 reflections]	R1 = 0.0420, *w*R2 = 0.0639
R indices (all data)	R1 = 0.0649, *w*R2 = 0.0649
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Absolute structure parameter	-0.3(3)
Largest diff. peak and hole	0.496 and -0.336 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table A4.8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for RUN04 (CCDC 845603). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1A)	5554(1)	2458(1)	3635(1)	17(1)
O(2A)	5617(1)	4860(1)	3573(1)	16(1)
O(3A)	6856(1)	5367(1)	4536(1)	13(1)
O(4A)	5534(1)	6195(1)	5244(1)	15(1)
N(1A)	6999(1)	2778(1)	6210(1)	12(1)
C(1A)	5743(1)	3265(1)	3721(1)	12(1)
C(2A)	5213(1)	4024(1)	3234(1)	17(1)
C(3A)	6665(1)	4676(1)	3962(1)	12(1)
C(4A)	6553(1)	3693(1)	4317(1)	10(1)
C(5A)	7642(1)	3165(1)	4411(1)	12(1)
C(6A)	8139(1)	3580(1)	5187(1)	12(1)
C(7A)	7155(1)	3664(1)	5767(1)	10(1)
C(8A)	7224(1)	4482(1)	6354(1)	12(1)
C(9A)	7050(1)	5431(1)	5965(1)	13(1)
C(10A)	6201(1)	5414(1)	5272(1)	11(1)
C(11A)	5454(1)	4596(1)	5351(1)	10(1)
C(12A)	6098(1)	3720(1)	5213(1)	8(1)
C(13A)	5397(1)	2880(1)	5456(1)	12(1)
C(14A)	5810(1)	2651(1)	6303(1)	14(1)
C(15A)	7614(1)	2662(1)	6969(1)	18(1)
C(16A)	7580(1)	4788(1)	3341(1)	18(1)
O(1B)	2680(1)	7609(1)	2857(1)	29(1)
O(2B)	2933(1)	5215(1)	2843(1)	17(1)
O(3B)	1616(1)	4626(1)	3711(1)	14(1)
O(4B)	2919(1)	3806(1)	4444(1)	14(1)
N(1B)	1310(1)	7158(1)	5459(1)	12(1)
C(1B)	2641(1)	6797(1)	2993(1)	19(1)
C(2B)	3378(1)	6081(1)	2610(1)	24(1)
C(3B)	1831(1)	5337(1)	3167(1)	15(1)
C(4B)	1832(1)	6307(1)	3550(1)	14(1)
C(5B)	708(1)	6775(1)	3641(1)	17(1)
C(6B)	225(1)	6340(1)	4408(1)	16(1)
C(7B)	1197(1)	6278(1)	4998(1)	11(1)
C(8B)	1143(1)	5441(1)	5566(1)	15(1)
C(9B)	1342(1)	4518(1)	5139(1)	16(1)
C(10B)	2228(1)	4574(1)	4473(1)	12(1)
C(11B)	2944(1)	5407(1)	4592(1)	11(1)
C(12B)	2268(1)	6268(1)	4452(1)	10(1)
C(13B)	2931(1)	7117(1)	4725(1)	13(1)
C(14B)	2492(1)	7309(1)	5570(1)	14(1)
C(15B)	686(1)	7231(1)	6218(1)	17(1)
C(16B)	1021(1)	5208(1)	2474(1)	24(1)

Table A4.9. Bond lengths [Å] and angles [°] for RUN04 (CCDC 845603).

O(1A)-C(1A)	1.2077(9)	C(2A)-O(2A)-C(3A)	108.34(5)
O(2A)-C(2A)	1.4278(9)	C(3A)-O(3A)-C(10A)	120.30(5)
O(2A)-C(3A)	1.4486(9)	C(15A)-N(1A)-C(14A)	113.62(6)
O(3A)-C(3A)	1.3986(9)	C(15A)-N(1A)-C(7A)	116.75(6)
O(3A)-C(10A)	1.4485(8)	C(14A)-N(1A)-C(7A)	106.52(5)
O(4A)-C(10A)	1.3994(9)	O(1A)-C(1A)-C(2A)	124.87(7)
N(1A)-C(15A)	1.4614(10)	O(1A)-C(1A)-C(4A)	126.79(7)
N(1A)-C(14A)	1.4631(10)	C(2A)-C(1A)-C(4A)	108.33(6)
N(1A)-C(7A)	1.4967(9)	O(2A)-C(2A)-C(1A)	105.95(6)
C(1A)-C(2A)	1.5100(11)	O(3A)-C(3A)-O(2A)	108.02(5)
C(1A)-C(4A)	1.5211(10)	O(3A)-C(3A)-C(16A)	104.69(6)
C(3A)-C(16A)	1.5170(10)	O(2A)-C(3A)-C(16A)	109.02(6)
C(3A)-C(4A)	1.5552(10)	O(3A)-C(3A)-C(4A)	115.33(6)
C(4A)-C(5A)	1.5368(10)	O(2A)-C(3A)-C(4A)	105.04(5)
C(4A)-C(12A)	1.5710(9)	C(16A)-C(3A)-C(4A)	114.53(6)
C(5A)-C(6A)	1.5331(10)	C(1A)-C(4A)-C(5A)	114.48(6)
C(6A)-C(7A)	1.5317(10)	C(1A)-C(4A)-C(3A)	101.21(5)
C(7A)-C(8A)	1.5366(10)	C(5A)-C(4A)-C(3A)	115.17(6)
C(7A)-C(12A)	1.5748(10)	C(1A)-C(4A)-C(12A)	112.67(6)
C(8A)-C(9A)	1.5409(10)	C(5A)-C(4A)-C(12A)	102.78(5)
C(9A)-C(10A)	1.5341(10)	C(3A)-C(4A)-C(12A)	110.93(6)
C(10A)-C(11A)	1.5041(10)	C(6A)-C(5A)-C(4A)	102.93(6)
C(11A)-C(12A)	1.5158(10)	C(7A)-C(6A)-C(5A)	103.98(6)
C(12A)-C(13A)	1.5453(10)	N(1A)-C(7A)-C(6A)	109.33(6)
C(13A)-C(14A)	1.5139(11)	N(1A)-C(7A)-C(8A)	111.91(5)
O(1B)-C(1B)	1.2068(10)	C(6A)-C(7A)-C(8A)	114.21(6)
O(2B)-C(2B)	1.4270(10)	N(1A)-C(7A)-C(12A)	102.88(5)
O(2B)-C(3B)	1.4499(9)	C(6A)-C(7A)-C(12A)	106.25(5)
O(3B)-C(3B)	1.3948(9)	C(8A)-C(7A)-C(12A)	111.51(6)
O(3B)-C(10B)	1.4555(8)	C(7A)-C(8A)-C(9A)	115.50(6)
O(4B)-C(10B)	1.4001(9)	C(10A)-C(9A)-C(8A)	112.63(6)
N(1B)-C(14B)	1.4623(10)	O(4A)-C(10A)-O(3A)	109.19(6)
N(1B)-C(15B)	1.4625(10)	O(4A)-C(10A)-C(11A)	107.44(5)
N(1B)-C(7B)	1.4965(10)	O(3A)-C(10A)-C(11A)	111.42(6)
C(1B)-C(2B)	1.5122(12)	O(4A)-C(10A)-C(9A)	113.55(6)
C(1B)-C(4B)	1.5198(11)	O(3A)-C(10A)-C(9A)	104.54(5)
C(3B)-C(16B)	1.5152(11)	C(11A)-C(10A)-C(9A)	110.74(6)
C(3B)-C(4B)	1.5493(11)	C(10A)-C(11A)-C(12A)	110.20(5)
C(4B)-C(5B)	1.5329(10)	C(11A)-C(12A)-C(13A)	110.34(6)
C(4B)-C(12B)	1.5728(10)	C(11A)-C(12A)-C(4A)	110.08(6)
C(5B)-C(6B)	1.5285(12)	C(13A)-C(12A)-C(4A)	114.53(6)
C(6B)-C(7B)	1.5291(10)	C(11A)-C(12A)-C(7A)	112.16(6)
C(7B)-C(8B)	1.5371(11)	C(13A)-C(12A)-C(7A)	104.90(5)
C(7B)-C(12B)	1.5795(10)	C(4A)-C(12A)-C(7A)	104.63(5)
C(8B)-C(9B)	1.5383(11)	C(14A)-C(13A)-C(12A)	103.35(6)
C(9B)-C(10B)	1.5349(10)	N(1A)-C(14A)-C(13A)	101.71(6)
C(10B)-C(11B)	1.5077(10)	C(2B)-O(2B)-C(3B)	109.79(6)
C(11B)-C(12B)	1.5181(10)	C(3B)-O(3B)-C(10B)	119.57(5)
C(12B)-C(13B)	1.5440(10)	C(14B)-N(1B)-C(15B)	112.94(6)
C(13B)-C(14B)	1.5123(11)	C(14B)-N(1B)-C(7B)	106.39(6)
		C(15B)-N(1B)-C(7B)	116.48(6)

O(1B)-C(1B)-C(2B)	125.30(8)	N(1B)-C(7B)-C(12B)	102.70(6)
O(1B)-C(1B)-C(4B)	126.79(8)	C(6B)-C(7B)-C(12B)	105.98(5)
C(2B)-C(1B)-C(4B)	107.88(7)	C(8B)-C(7B)-C(12B)	111.83(6)
O(2B)-C(2B)-C(1B)	106.06(7)	C(7B)-C(8B)-C(9B)	114.43(6)
O(3B)-C(3B)-O(2B)	108.45(6)	C(10B)-C(9B)-C(8B)	112.82(6)
O(3B)-C(3B)-C(16B)	105.52(6)	O(4B)-C(10B)-O(3B)	108.59(6)
O(2B)-C(3B)-C(16B)	107.94(6)	O(4B)-C(10B)-C(11B)	107.73(5)
O(3B)-C(3B)-C(4B)	114.84(6)	O(3B)-C(10B)-C(11B)	111.32(6)
O(2B)-C(3B)-C(4B)	105.07(6)	O(4B)-C(10B)-C(9B)	113.59(6)
C(16B)-C(3B)-C(4B)	114.75(6)	O(3B)-C(10B)-C(9B)	104.90(5)
C(1B)-C(4B)-C(5B)	115.01(7)	C(11B)-C(10B)-C(9B)	110.73(6)
C(1B)-C(4B)-C(3B)	100.74(6)	C(10B)-C(11B)-C(12B)	109.66(5)
C(5B)-C(4B)-C(3B)	116.50(6)	C(11B)-C(12B)-C(13B)	109.81(6)
C(1B)-C(4B)-C(12B)	111.49(6)	C(11B)-C(12B)-C(4B)	110.76(6)
C(5B)-C(4B)-C(12B)	102.93(6)	C(13B)-C(12B)-C(4B)	114.82(6)
C(3B)-C(4B)-C(12B)	110.44(6)	C(11B)-C(12B)-C(7B)	111.47(6)
C(6B)-C(5B)-C(4B)	103.66(6)	C(13B)-C(12B)-C(7B)	104.82(6)
C(5B)-C(6B)-C(7B)	104.52(6)	C(4B)-C(12B)-C(7B)	104.94(5)
N(1B)-C(7B)-C(6B)	109.86(6)	C(14B)-C(13B)-C(12B)	103.41(6)
N(1B)-C(7B)-C(8B)	112.33(6)	N(1B)-C(14B)-C(13B)	101.70(6)
C(6B)-C(7B)-C(8B)	113.44(6)		

Table A4.10. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for RUN04 (CCDC 845603). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1A)	216(3)	132(3)	168(3)	-42(2)	-12(2)	-11(2)
O(2A)	200(3)	117(2)	152(2)	0(2)	-63(2)	22(2)
O(3A)	158(2)	98(2)	123(2)	-3(2)	16(2)	-17(2)
O(4A)	155(3)	68(2)	213(3)	19(2)	14(2)	16(2)
N(1A)	156(3)	98(3)	101(3)	17(2)	-12(2)	13(2)
C(1A)	131(3)	137(3)	96(3)	-24(3)	13(3)	12(3)
C(2A)	214(4)	152(4)	148(3)	-30(3)	-62(3)	16(3)
C(3A)	153(3)	102(3)	97(3)	3(3)	-10(2)	7(3)
C(4A)	122(3)	86(3)	89(3)	-5(3)	11(2)	3(3)
C(5A)	125(3)	106(3)	119(3)	-1(3)	20(3)	24(3)
C(6A)	113(3)	116(3)	138(3)	-4(3)	-6(3)	16(3)
C(7A)	120(3)	78(3)	95(3)	8(2)	-11(2)	11(3)
C(8A)	155(3)	109(3)	105(3)	-9(3)	-19(3)	8(3)
C(9A)	179(3)	90(3)	133(3)	-19(3)	-18(3)	2(3)
C(10A)	140(3)	79(3)	111(3)	4(3)	17(2)	21(3)
C(11A)	113(3)	87(3)	107(3)	2(3)	9(2)	11(3)
C(12A)	91(3)	77(3)	85(3)	1(3)	5(2)	1(2)
C(13A)	112(3)	84(3)	157(3)	5(3)	15(3)	-4(2)
C(14A)	172(4)	107(3)	142(3)	19(3)	43(3)	8(3)
C(15A)	259(4)	143(4)	127(3)	12(3)	-40(3)	50(3)
C(16A)	248(4)	157(4)	135(3)	22(3)	50(3)	-14(3)
O(1B)	519(4)	124(3)	222(3)	45(2)	109(3)	30(3)
O(2B)	238(3)	116(3)	164(2)	-1(2)	44(2)	26(2)
O(3B)	165(2)	99(2)	152(2)	-7(2)	-46(2)	-8(2)
O(4B)	157(2)	69(2)	200(3)	-13(2)	-25(2)	12(2)
N(1B)	128(3)	105(3)	121(3)	-23(2)	3(2)	9(2)
C(1B)	320(5)	131(4)	119(3)	8(3)	27(3)	18(3)
C(2B)	359(5)	136(4)	219(4)	21(3)	98(4)	4(3)
C(3B)	188(4)	115(3)	133(3)	-12(3)	-26(3)	35(3)
C(4B)	185(4)	98(3)	125(3)	12(3)	-19(3)	31(3)
C(5B)	216(4)	144(4)	164(4)	-28(3)	-74(3)	76(3)
C(6B)	123(3)	162(4)	208(4)	-53(3)	-38(3)	31(3)
C(7B)	110(3)	88(3)	133(3)	-9(3)	1(3)	7(3)
C(8B)	160(3)	119(3)	163(3)	10(3)	32(3)	0(3)
C(9B)	186(4)	100(3)	193(4)	16(3)	25(3)	-12(3)
C(10B)	132(3)	78(3)	138(3)	7(3)	-24(3)	8(3)
C(11B)	110(3)	86(3)	127(3)	6(3)	-7(2)	9(3)
C(12B)	106(3)	72(3)	116(3)	4(3)	1(2)	1(3)
C(13B)	132(3)	89(3)	176(3)	0(3)	8(3)	-7(3)
C(14B)	153(4)	113(3)	164(4)	-23(3)	-26(3)	-8(3)
C(15B)	198(4)	169(4)	147(4)	-13(3)	24(3)	28(3)
C(16B)	327(5)	203(4)	181(4)	-43(3)	-97(3)	70(4)

Table A4.11. Hydrogen bonds for RUN04 (CCDC 845603) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(4A)-H(4A)...N(1B)#1	0.84	2.02	2.8290(8)	160.1
O(4B)-H(4B)...N(1A)#2	0.84	1.96	2.7835(8)	166.4

Symmetry transformations used to generate equivalent atoms:
#1 x+1/2,-y+3/2,-z+1
#2 x-1/2,-y+1/2,-z+1

A4.3. Crystal Structure analysis of hexacycle 255.

Figure A4.2. Hexacycle **255**.

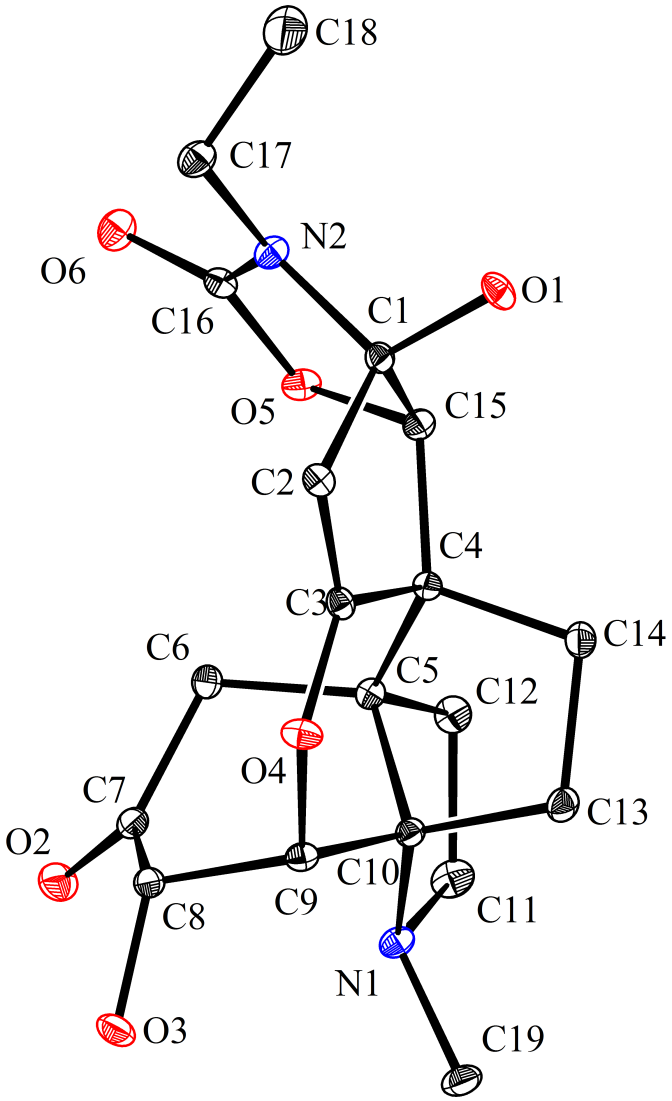


Table A4.12. Crystal Data and Structure Analysis Details for hexacycle **255.**

Empirical formula	C20 H25 Cl3 N2 O6
Formula weight	495.77
Crystallization solvent	chloroform
Crystal shape	blade
Crystal color	colourless
Crystal size	0.07 x 0.10 x 0.38 mm

Data Collection

Preliminary photograph(s)	rotation	
Type of diffractometer	Bruker APEX-II CCD	
Wavelength	0.71073 Å MoK	
Data collection temperature	100 K	
Theta range for 9903 reflections used in lattice determination	2.37 to 34.99°	
Unit cell dimensions	a = 6.7127(3) Å b = 14.4750(7) Å c = 10.7522(6) Å	a= 90° b= 95.003(3)° g = 90°
Volume	1040.77(9) $Å^3$	
Z	2	
Crystal system	monoclinic	
Space group	P 1 21 1 (# 4)	
Density (calculated)	1.582 g/cm^3	
F(000)	516	
Theta range for data collection	1.9 to 37.1°	
Completeness to theta = 25.000°	99.9%	
Index ranges	-10 £ h £ 11, -24 £ k £ 24, -17 £ l £ 17	
Data collection scan type	and scans	
Reflections collected	42130	
Independent reflections	9868 [R_{int}= 0.0488]	
Reflections > 2s(I)	8802	
Average s(I)/(net I)	0.0474	
Absorption coefficient	0.48 mm^{-1}	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.0000 and 0.9024	

Table A4.12. (continued)

Structure Solution and Refinement

Primary solution method	dual
Secondary solution method	?
Hydrogen placement	difmap
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	9868 / 1 / 380
Treatment of hydrogen atoms	refall
Goodness-of-fit on F^2	1.20
Final R indices [I>2s(I), 8802 reflections]	R1 = 0.0370, wR2 = 0.0744
R indices (all data)	R1 = 0.0459, wR2 = 0.0770
Type of weighting scheme used	calc
Weighting scheme used	w=1/[^2^(Fo^2^)+(0.0300P)^2^] where P=(Fo^2^+2Fc^2^)/3
Max shift/error	0.001
Average shift/error	0.000
Absolute structure parameter	0.046(15)
Extinction coefficient	n/a
Largest diff. peak and hole	0.51 and -0.27 e·Å^{-3}

Programs Used

Cell refinement	SAINT V8.32B (Bruker-AXS, 2007)
Data collection	APEX2 2013.2-0 (Bruker-AXS, 2007)
Data reduction	SAINT V8.32B (Bruker-AXS, 2007)
Structure solution	SHELXT (Sheldrick, 2012)
Structure refinement	SHELXL-2013/2 (Sheldrick, 2013)
Graphics	DIAMOND 3 (Crystal Impact, 1999)

References

Special Refinement Details

Table A4.13. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for run08. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	5492(2)	10893(1)	6327(1)	11(1)
O(2)	-1017(2)	7011(1)	8955(1)	14(1)
O(3)	2649(2)	6411(1)	9950(1)	13(1)
O(4)	5085(2)	8311(1)	8663(1)	10(1)
O(5)	691(2)	10026(1)	6578(1)	10(1)
O(6)	-791(2)	11151(1)	7604(1)	13(1)
N(1)	2162(2)	6446(1)	6944(2)	9(1)
N(2)	2603(2)	11166(1)	7397(1)	9(1)
C(1)	4063(2)	10485(1)	7032(2)	8(1)
C(2)	4938(2)	9904(1)	8105(2)	8(1)
C(3)	4478(2)	9016(1)	7889(2)	8(1)
C(4)	3356(2)	8845(1)	6616(2)	7(1)
C(5)	1859(2)	8058(1)	6755(2)	8(1)
C(6)	416(2)	8230(1)	7782(2)	9(1)
C(7)	441(2)	7447(1)	8698(2)	9(1)
C(8)	2499(2)	7289(1)	9377(2)	9(1)
C(9)	4159(2)	7404(1)	8484(2)	8(1)
C(10)	3376(2)	7282(1)	7116(2)	8(1)
C(11)	783(3)	6592(1)	5813(2)	13(1)
C(12)	728(2)	7644(1)	5571(2)	11(1)
C(13)	5071(2)	7415(1)	6236(2)	10(1)
C(14)	4881(2)	8425(1)	5776(2)	10(1)
C(15)	2689(2)	9817(1)	6235(2)	8(1)
C(16)	735(2)	10824(1)	7238(2)	9(1)
C(17)	3148(3)	11961(1)	8193(2)	12(1)
C(18)	3986(4)	12748(2)	7479(3)	29(1)
C(19)	3298(3)	5585(1)	6923(2)	13(1)
Cl(1)	2513(1)	9965(1)	10419(1)	18(1)
Cl(2)	1398(1)	9278(1)	12784(1)	25(1)
Cl(3)	-1371(1)	9156(1)	10564(1)	16(1)
C(20)	527(3)	9823(1)	11365(2)	12(1)

Table A4.14. Bond lengths [Å] and angles [°] for hexacycle **253**.

O(1)-H(1)	0.87(3)
O(1)-C(1)	1.403(2)
O(2)-C(7)	1.216(2)
O(3)-H(3)	0.84(3)
O(3)-C(8)	1.412(2)
O(4)-C(3)	1.357(2)
O(4)-C(9)	1.459(2)
O(5)-C(15)	1.4535(19)
O(5)-C(16)	1.355(2)
O(6)-C(16)	1.223(2)
N(1)-C(10)	1.462(2)
N(1)-C(11)	1.478(2)
N(1)-C(19)	1.462(2)
N(2)-C(1)	1.467(2)
N(2)-C(16)	1.346(2)
N(2)-C(17)	1.462(2)
C(1)-C(2)	1.505(2)
C(1)-C(15)	1.544(2)
C(2)-H(2)	0.96(3)
C(2)-C(3)	1.338(2)
C(3)-C(4)	1.524(2)
C(4)-C(5)	1.535(2)
C(4)-C(14)	1.547(2)
C(4)-C(15)	1.523(2)
C(5)-C(6)	1.551(2)
C(5)-C(10)	1.543(2)
C(5)-C(12)	1.545(2)
C(6)-H(6A)	0.92(3)
C(6)-H(6B)	0.97(3)
C(6)-C(7)	1.501(2)
C(7)-C(8)	1.523(2)
C(8)-H(8)	0.92(3)
C(8)-C(9)	1.542(2)
C(9)-H(9)	0.94(2)
C(9)-C(10)	1.528(2)
C(10)-C(13)	1.554(2)
C(11)-H(11A)	0.98(3)
C(11)-H(11B)	0.95(2)
C(11)-C(12)	1.544(2)
C(12)-H(12A)	0.96(3)
C(12)-H(12B)	0.89(2)
C(13)-H(13A)	0.90(3)
C(13)-H(13B)	0.92(2)
C(13)-C(14)	1.544(2)
C(14)-H(14A)	0.96(3)
C(14)-H(14B)	0.92(2)
C(15)-H(15)	1.00(2)
C(17)-H(17A)	0.91(3)
C(17)-H(17B)	0.95(2)
C(17)-C(18)	1.509(3)
C(18)-H(18A)	0.89(4)

C(18)-H(18B)	0.97(3)
C(18)-H(18C)	1.01(5)
C(19)-H(19A)	1.00(3)
C(19)-H(19B)	0.91(3)
C(19)-H(19C)	0.97(3)
Cl(1)-C(20)	1.7580(18)
Cl(2)-C(20)	1.7706(19)
Cl(3)-C(20)	1.7626(19)
C(20)-H(20)	0.88(3)

C(1)-O(1)-H(1)	105.7(19)
C(8)-O(3)-H(3)	108(2)
C(3)-O(4)-C(9)	119.62(13)
C(16)-O(5)-C(15)	109.38(12)
C(10)-N(1)-C(11)	106.73(13)
C(10)-N(1)-C(19)	114.96(13)
C(19)-N(1)-C(11)	113.54(14)
C(16)-N(2)-C(1)	110.85(14)
C(16)-N(2)-C(17)	123.14(14)
C(17)-N(2)-C(1)	123.15(13)
O(1)-C(1)-N(2)	111.46(13)
O(1)-C(1)-C(2)	114.21(13)
O(1)-C(1)-C(15)	111.52(14)
N(2)-C(1)-C(2)	113.29(14)
N(2)-C(1)-C(15)	100.87(12)
C(2)-C(1)-C(15)	104.46(13)
C(1)-C(2)-H(2)	123.4(15)
C(3)-C(2)-C(1)	109.59(15)
C(3)-C(2)-H(2)	127.0(15)
O(4)-C(3)-C(4)	121.84(14)
C(2)-C(3)-O(4)	124.29(16)
C(2)-C(3)-C(4)	113.64(14)
C(3)-C(4)-C(5)	108.04(13)
C(3)-C(4)-C(14)	106.72(12)
C(5)-C(4)-C(14)	103.76(13)
C(15)-C(4)-C(3)	101.67(13)
C(15)-C(4)-C(5)	122.16(12)
C(15)-C(4)-C(14)	113.59(14)
C(4)-C(5)-C(6)	113.87(13)
C(4)-C(5)-C(10)	98.11(12)
C(4)-C(5)-C(12)	119.09(14)
C(10)-C(5)-C(6)	112.16(14)
C(10)-C(5)-C(12)	101.03(13)
C(12)-C(5)-C(6)	110.91(13)
C(5)-C(6)-H(6A)	110.2(15)
C(5)-C(6)-H(6B)	109.2(15)
H(6A)-C(6)-H(6B)	105(2)
C(7)-C(6)-C(5)	111.92(13)
C(7)-C(6)-H(6A)	107.0(16)
C(7)-C(6)-H(6B)	113.1(15)
O(2)-C(7)-C(6)	125.29(15)
O(2)-C(7)-C(8)	121.84(16)
C(6)-C(7)-C(8)	112.70(14)
O(3)-C(8)-C(7)	111.83(13)

O(3)-C(8)-H(8)	108.8(16)
O(3)-C(8)-C(9)	109.91(13)
C(7)-C(8)-H(8)	107.7(15)
C(7)-C(8)-C(9)	111.12(14)
C(9)-C(8)-H(8)	107.4(14)
O(4)-C(9)-C(8)	109.72(13)
O(4)-C(9)-H(9)	104.8(14)
O(4)-C(9)-C(10)	109.74(13)
C(8)-C(9)-H(9)	106.4(14)
C(10)-C(9)-C(8)	112.32(13)
C(10)-C(9)-H(9)	113.5(15)
N(1)-C(10)-C(5)	102.84(12)
N(1)-C(10)-C(9)	111.01(13)
N(1)-C(10)-C(13)	117.09(14)
C(5)-C(10)-C(13)	104.98(13)
C(9)-C(10)-C(5)	108.84(13)
C(9)-C(10)-C(13)	111.27(13)
N(1)-C(11)-H(11A)	103.4(18)
N(1)-C(11)-H(11B)	114.0(15)
N(1)-C(11)-C(12)	106.48(13)
H(11A)-C(11)-H(11B)	106(2)
C(12)-C(11)-H(11A)	115.2(17)
C(12)-C(11)-H(11B)	111.5(15)
C(5)-C(12)-H(12A)	111.4(15)
C(5)-C(12)-H(12B)	109.2(16)
C(11)-C(12)-C(5)	103.93(14)
C(11)-C(12)-H(12A)	110.8(15)
C(11)-C(12)-H(12B)	112.3(16)
H(12A)-C(12)-H(12B)	109(2)
C(10)-C(13)-H(13A)	108.3(18)
C(10)-C(13)-H(13B)	112.3(15)
H(13A)-C(13)-H(13B)	106(2)
C(14)-C(13)-C(10)	105.56(13)
C(14)-C(13)-H(13A)	112.7(18)
C(14)-C(13)-H(13B)	112.3(16)
C(4)-C(14)-H(14A)	111.1(15)
C(4)-C(14)-H(14B)	110.0(15)
C(13)-C(14)-C(4)	103.00(13)
C(13)-C(14)-H(14A)	113.6(16)
C(13)-C(14)-H(14B)	116.4(15)
H(14A)-C(14)-H(14B)	103(2)
O(5)-C(15)-C(1)	104.40(13)
O(5)-C(15)-C(4)	112.34(13)
O(5)-C(15)-H(15)	110.7(13)
C(1)-C(15)-H(15)	110.1(14)
C(4)-C(15)-C(1)	106.38(13)
C(4)-C(15)-H(15)	112.5(15)
O(6)-C(16)-O(5)	121.13(15)
O(6)-C(16)-N(2)	127.91(16)
N(2)-C(16)-O(5)	110.95(14)
N(2)-C(17)-H(17A)	110(2)
N(2)-C(17)-H(17B)	107.1(16)
N(2)-C(17)-C(18)	112.43(17)
H(17A)-C(17)-H(17B)	104(3)

C(18)-C(17)-H(17A)	110(2)
C(18)-C(17)-H(17B)	112.2(15)
C(17)-C(18)-H(18A)	112(2)
C(17)-C(18)-H(18B)	108.9(19)
C(17)-C(18)-H(18C)	109(3)
H(18A)-C(18)-H(18B)	117(3)
H(18A)-C(18)-H(18C)	104(3)
H(18B)-C(18)-H(18C)	105(3)
N(1)-C(19)-H(19A)	112.3(15)
N(1)-C(19)-H(19B)	108.8(16)
N(1)-C(19)-H(19C)	108.5(15)
H(19A)-C(19)-H(19B)	107(2)
H(19A)-C(19)-H(19C)	109(2)
H(19B)-C(19)-H(19C)	111(2)
Cl(1)-C(20)-Cl(2)	109.88(9)
Cl(1)-C(20)-Cl(3)	109.45(10)
Cl(1)-C(20)-H(20)	107.4(17)
Cl(2)-C(20)-H(20)	107.8(18)
Cl(3)-C(20)-Cl(2)	110.31(10)
Cl(3)-C(20)-H(20)	111.9(16)

Table A4.15. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for run08. The anisotropic displacement factor exponent takes the form: $-2p^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$.

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	77(5)	134(5)	118(6)	35(5)	29(4)	-23(4)
O(2)	111(5)	131(6)	177(7)	24(5)	43(5)	-12(4)
O(3)	132(5)	119(5)	131(6)	60(5)	23(5)	16(4)
O(4)	112(5)	76(5)	103(6)	4(4)	-36(4)	-22(4)
O(5)	68(4)	86(5)	132(6)	3(4)	-13(4)	2(4)
O(6)	79(5)	148(6)	156(7)	-13(5)	15(4)	7(4)
N(1)	88(5)	73(6)	107(7)	-21(5)	-2(5)	-6(4)
N(2)	77(5)	82(6)	110(7)	-19(5)	2(5)	-9(4)
C(1)	75(6)	83(6)	87(7)	1(5)	18(5)	-7(5)
C(2)	66(5)	99(6)	84(7)	6(6)	-4(5)	-9(5)
C(3)	62(5)	101(7)	79(7)	9(5)	11(5)	-2(5)
C(4)	68(5)	72(6)	66(7)	0(5)	-2(5)	-2(5)
C(5)	71(6)	80(6)	77(7)	-9(5)	1(5)	-3(5)
C(6)	76(6)	98(6)	90(7)	-4(5)	9(5)	-1(5)
C(7)	93(6)	83(6)	95(7)	-7(5)	12(5)	10(5)
C(8)	109(6)	81(6)	73(7)	-1(5)	4(5)	7(5)
C(9)	80(6)	69(6)	99(7)	-2(5)	1(5)	-1(5)
C(10)	75(6)	70(6)	84(7)	-6(5)	11(5)	-1(5)
C(11)	130(6)	113(7)	133(9)	-33(6)	-29(6)	-14(6)
C(12)	106(6)	123(7)	82(8)	-12(6)	-27(6)	-1(5)
C(13)	82(6)	104(7)	124(8)	-7(6)	28(5)	5(5)
C(14)	104(6)	115(7)	97(8)	-4(6)	26(5)	0(5)
C(15)	70(5)	85(6)	95(7)	-3(5)	3(5)	5(5)
C(16)	97(6)	87(6)	86(7)	17(5)	3(5)	0(5)
C(17)	122(7)	101(7)	134(8)	-27(6)	8(6)	-9(5)
C(18)	425(13)	123(8)	348(14)	-77(9)	235(12)	-107(9)
C(19)	151(7)	74(7)	176(9)	-11(6)	34(6)	14(5)
Cl(1)	179(2)	181(2)	200(2)	-37(2)	87(2)	-28(2)
Cl(2)	263(2)	343(3)	134(2)	53(2)	-6(2)	119(2)
Cl(3)	167(2)	154(2)	171(2)	-22(2)	25(2)	-30(2)
C(20)	142(7)	120(7)	112(8)	-5(6)	20(6)	19(6)

Table A4.16. Hydrogen coordinates (x 10^3) and isotropic displacement parameters (Å^{2}x 10^3) for hexacycle **253**.

	x	y	z	U_{iso}
H(1)	658(4)	1093(2)	683(3)	22(7)
H(3)	176(4)	638(2)	1046(3)	26(7)
H(2)	565(4)	1016(2)	884(2)	12(6)
H(6A)	79(4)	875(2)	823(2)	13(6)
H(6B)	-91(4)	836(2)	739(2)	14(6)
H(8)	270(3)	774(2)	998(2)	7(5)
H(9)	517(3)	698(2)	875(2)	5(5)
H(11A)	-47(4)	632(2)	604(3)	28(8)
H(11B)	114(3)	626(2)	510(2)	8(5)
H(12A)	138(4)	779(2)	483(3)	12(6)
H(12B)	-51(4)	786(2)	549(2)	11(6)
H(13A)	625(4)	730(2)	667(3)	21(7)
H(13B)	499(3)	700(2)	559(2)	8(5)
H(14A)	442(4)	848(2)	491(3)	16(6)
H(14B)	604(3)	877(2)	584(2)	10(6)
H(15)	279(3)	994(2)	532(2)	10(5)
H(17A)	404(5)	1179(2)	884(3)	32(8)
H(17B)	199(3)	1214(2)	858(2)	9(5)
H(18A)	494(5)	1256(2)	701(3)	39(9)
H(18B)	430(5)	1326(2)	805(3)	32(8)
H(18C)	291(7)	1300(4)	686(5)	85(16)
H(19A)	422(4)	558(2)	624(3)	17(6)
H(19B)	243(3)	511(2)	678(2)	14(6)
H(19C)	408(4)	552(2)	772(3)	11(6)
H(20)	10(4)	1038(2)	1155(3)	13(6)

Table A4.17. Hydrogen bonds for run08 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(6)#1	0.87(3)	1.91(3)	2.7662(18)	167(3)
O(3)-H(3)...O(6)#2	0.84(3)	2.26(3)	3.031(2)	152(3)
C(2)-H(2)...O(3)#3	0.96(3)	2.46(3)	3.341(2)	153(2)
C(2)-H(2)...Cl(3)#1	0.96(3)	2.98(2)	3.6276(17)	125.9(18)
C(9)-H(9)...O(2)#1	0.94(2)	2.55(2)	3.283(2)	135.2(18)
C(13)-H(13B)...O(1)#4	0.92(2)	2.61(3)	3.523(2)	176(2)
C(15)-H(15)...Cl(2)#5	1.00(2)	2.97(2)	3.8169(18)	143.1(18)
C(17)-H(17A)...O(3)#3	0.91(3)	2.54(3)	3.406(2)	159(3)
C(18)-H(18A)...O(1)	0.89(4)	2.56(4)	3.160(3)	125(3)
C(18)-H(18B)...Cl(3)#6	0.97(3)	2.88(3)	3.508(2)	123(2)
C(19)-H(19B)...Cl(2)#2	0.91(3)	2.91(2)	3.7129(18)	148(2)
C(20)-H(20)...O(2)#6	0.88(3)	2.52(3)	3.206(2)	136(2)

Symmetry transformations used to generate equivalent atoms:
#1 x+1,y,z #2 -x,y-1/2,-z+2 #3 -x+1,y+1/2,-z+2
#4 -x+1,y-1/2,-z+1 #5 x,y,z-1 #6 -x,y+1/2,-z+2