Appendix 2

X-Ray Crystallography Reports Relevant to Chapter 2: Total Synthesis of (–)-Maoecrystal Z[†]

[†] The work disclosed in this appendix for the X-ray crystallographic analysis of **109** and **127** was completed entirely by Mr. Larry Henling and Dr. Michael Day in the Caltech X-ray crystallography lab.

A2.1 CRYSTAL STRUCTURE ANALYSIS OF SPIROLACTONE 109

Figure A2.1. Spirolactone **109**. 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 837088.

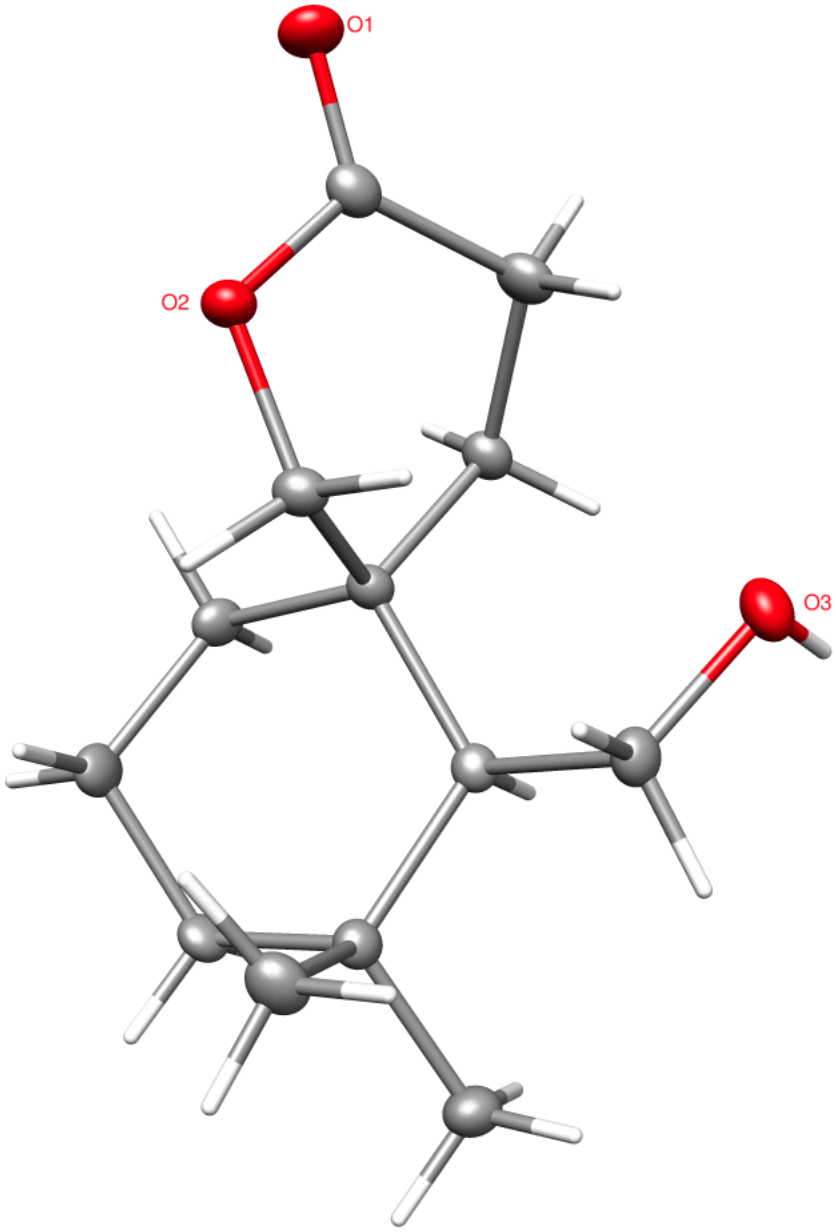


Table A2.1. Crystal data and structure refinement for spirolactone **109** (CCDC 837088).

Empirical formula	$C_{13}H_{22}O_3$
Formula weight	226.31
Crystallization Solvent	Isooctane/dichloromethane
Crystal Habit	Column
Crystal size	0.22 x 0.15 x 0.07 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 5677 reflections used in lattice determination	2.53 to 36.05°	
Unit cell dimensions	a = 8.3489(4) Å b = 7.3054(3) Å c = 9.9944(5) Å	α= 90° β= 105.627(3)° γ = 90°
Volume	587.05(5) $Å^3$	
Z	2	
Crystal system	Monoclinic	
Space group	P 2_1	
Density (calculated)	1.280 Mg/m^3	
F(000)	248	
Data collection program	Bruker APEX2 v2009.7-0	
θ range for data collection	2.12 to 36.35°	
Completeness to θ = 36.35°	99.6 %	
Index ranges	$-13 \leq h \leq 12$, $-12 \leq k \leq 12$, $-16 \leq l \leq 16$	
Data collection scan type	ω scans; 9 settings	
Data reduction program	Bruker SAINT-Plus v7.66A	
Reflections collected	17473	
Independent reflections	5474 [R_{int}= 0.0331]	
Absorption coefficient	0.089 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9938 and 0.9807	

Table A2.1 (cont.)

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	5474 / 1 / 233
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.709
Final R indices [I>2σ(I), 4661 reflections]	R1 = 0.0408, *w*R2 = 0.0498
R indices (all data)	R1 = 0.0512, *w*R2 = 0.0504
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 determination	Unable to reliably determine
Absolute structure parameter	-0.2(6)
Largest diff. peak and hole	0.380 and -0.314 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 A2.2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for spirolactone **109** (CCDC 837088). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	6715(1)	-573(1)	7277(1)	22(1)
O(2)	4260(1)	682(1)	7046(1)	18(1)
O(3)	4941(1)	6385(1)	5917(1)	21(1)
C(1)	5908(1)	820(1)	7204(1)	17(1)
C(2)	6586(1)	2720(1)	7332(1)	18(1)
C(3)	5836(1)	3874(1)	8286(1)	16(1)
C(4)	3897(1)	3793(1)	7901(1)	13(1)
C(5)	3322(1)	2395(1)	6732(1)	16(1)
C(6)	3432(1)	3115(2)	9204(1)	16(1)
C(7)	1601(1)	3218(1)	9127(1)	19(1)
C(8)	1023(1)	5195(1)	8884(1)	16(1)
C(9)	1284(1)	6031(1)	7544(1)	14(1)
C(10)	3106(1)	5731(1)	7462(1)	13(1)
C(11)	3280(1)	6376(1)	6038(1)	17(1)
C(12)	-36(1)	5250(2)	6284(1)	20(1)
C(13)	938(1)	8103(1)	7603(1)	21(1)

Table A2.3. Bond lengths [Å] and angles [°] for spirolactone **109** (CCDC 837088).

O(1)-C(1)	1.2118(12)	C(2)-C(3)-C(4)	113.43(8)
O(2)-C(1)	1.3455(11)	C(2)-C(3)-H(3A)	108.7(7)
O(2)-C(5)	1.4640(12)	C(4)-C(3)-H(3A)	109.8(6)
O(3)-C(11)	1.4239(12)	C(2)-C(3)-H(3B)	107.5(6)
O(3)-H(3)	0.780(12)	C(4)-C(3)-H(3B)	108.2(6)
C(1)-C(2)	1.4917(14)	H(3A)-C(3)-H(3B)	109.2(8)
C(2)-C(3)	1.5280(14)	C(5)-C(4)-C(6)	109.43(8)
C(2)-H(2A)	0.972(10)	C(5)-C(4)-C(3)	107.76(8)
C(2)-H(2B)	0.946(10)	C(6)-C(4)-C(3)	106.58(7)
C(3)-C(4)	1.5608(13)	C(5)-C(4)-C(10)	111.49(7)
C(3)-H(3A)	0.954(10)	C(6)-C(4)-C(10)	110.09(8)
C(3)-H(3B)	0.966(10)	C(3)-C(4)-C(10)	111.34(8)
C(4)-C(5)	1.5288(13)	O(2)-C(5)-C(4)	111.57(7)
C(4)-C(6)	1.5377(13)	O(2)-C(5)-H(5A)	105.0(6)
C(4)-C(10)	1.5737(13)	C(4)-C(5)-H(5A)	109.2(6)
C(5)-H(5A)	0.989(11)	O(2)-C(5)-H(5B)	107.9(6)
C(5)-H(5B)	0.968(9)	C(4)-C(5)-H(5B)	111.2(6)
C(6)-C(7)	1.5115(14)	H(5A)-C(5)-H(5B)	111.8(8)
C(6)-H(6A)	0.971(10)	C(7)-C(6)-C(4)	115.05(8)
C(6)-H(6B)	0.967(10)	C(7)-C(6)-H(6A)	108.3(6)
C(7)-C(8)	1.5213(14)	C(4)-C(6)-H(6A)	108.2(6)
C(7)-H(7A)	1.022(10)	C(7)-C(6)-H(6B)	112.0(7)
C(7)-H(7B)	1.006(11)	C(4)-C(6)-H(6B)	108.1(7)
C(8)-C(9)	1.5394(13)	H(6A)-C(6)-H(6B)	104.6(8)
C(8)-H(8A)	0.974(10)	C(6)-C(7)-C(8)	109.17(9)
C(8)-H(8B)	0.921(11)	C(6)-C(7)-H(7A)	112.5(6)
C(9)-C(12)	1.5420(13)	C(8)-C(7)-H(7A)	111.7(5)
C(9)-C(13)	1.5449(14)	C(6)-C(7)-H(7B)	110.1(6)
C(9)-C(10)	1.5613(13)	C(8)-C(7)-H(7B)	108.9(6)
C(10)-C(11)	1.5425(12)	H(7A)-C(7)-H(7B)	104.2(8)
C(10)-H(10)	0.960(9)	C(7)-C(8)-C(9)	113.76(8)
C(11)-H(11A)	1.026(10)	C(7)-C(8)-H(8A)	108.8(6)
C(11)-H(11B)	1.036(10)	C(9)-C(8)-H(8A)	109.6(6)
C(12)-H(12A)	0.961(12)	C(7)-C(8)-H(8B)	111.0(6)
C(12)-H(12B)	0.960(10)	C(9)-C(8)-H(8B)	109.0(6)
C(12)-H(12C)	0.938(12)	H(8A)-C(8)-H(8B)	104.2(8)
C(13)-H(13A)	0.952(11)	C(8)-C(9)-C(12)	109.05(8)
C(13)-H(13B)	0.931(11)	C(8)-C(9)-C(13)	106.48(8)
C(13)-H(13C)	0.991(10)	C(12)-C(9)-C(13)	107.08(8)
		C(8)-C(9)-C(10)	110.98(7)
C(1)-O(2)-C(5)	115.62(8)	C(12)-C(9)-C(10)	113.40(8)
C(11)-O(3)-H(3)	105.0(10)	C(13)-C(9)-C(10)	109.54(8)
O(1)-C(1)-O(2)	118.63(9)	C(11)-C(10)-C(9)	109.93(7)
O(1)-C(1)-C(2)	125.72(9)	C(11)-C(10)-C(4)	113.29(8)
O(2)-C(1)-C(2)	115.60(9)	C(9)-C(10)-C(4)	116.95(8)
C(1)-C(2)-C(3)	110.91(9)	C(11)-C(10)-H(10)	107.4(6)
C(1)-C(2)-H(2A)	109.9(6)	C(9)-C(10)-H(10)	101.7(6)
C(3)-C(2)-H(2A)	109.8(6)	C(4)-C(10)-H(10)	106.4(5)
C(1)-C(2)-H(2B)	107.2(6)	O(3)-C(11)-C(10)	114.57(8)
C(3)-C(2)-H(2B)	111.1(6)	O(3)-C(11)-H(11A)	107.1(6)
H(2A)-C(2)-H(2B)	107.8(8)	C(10)-C(11)-H(11A)	108.5(6)

O(3)-C(11)-H(11B)	110.9(6)	H(12B)-C(12)-H(12C)	109.2(9)
C(10)-C(11)-H(11B)	108.1(5)	C(9)-C(13)-H(13A)	110.4(6)
H(11A)-C(11)-H(11B)	107.5(7)	C(9)-C(13)-H(13B)	111.3(6)
C(9)-C(12)-H(12A)	113.5(6)	H(13A)-C(13)-H(13B)	110.6(9)
C(9)-C(12)-H(12B)	108.9(6)	C(9)-C(13)-H(13C)	110.9(6)
H(12A)-C(12)-H(12B)	108.8(9)	H(13A)-C(13)-H(13C)	104.7(8)
C(9)-C(12)-H(12C)	107.1(6)	H(13B)-C(13)-H(13C)	108.8(9)
H(12A)-C(12)-H(12C)	109.2(9)		

Table A2.4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for spirolactone **109** (CCDC 837088). 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(1)	188(4)	184(4)	282(4)	-9(3)	54(3)	43(3)
O(2)	152(4)	148(4)	242(3)	-22(3)	72(3)	9(3)
O(3)	218(4)	219(4)	233(4)	-10(3)	125(3)	-28(3)
C(1)	161(5)	199(5)	147(4)	-20(4)	55(3)	-6(4)
C(2)	125(5)	214(6)	217(5)	-39(4)	51(4)	-6(4)
C(3)	128(5)	180(5)	165(4)	-17(4)	39(3)	-9(4)
C(4)	115(5)	142(4)	132(4)	-7(3)	31(3)	3(4)
C(5)	135(5)	153(5)	174(4)	-29(4)	37(4)	5(4)
C(6)	186(5)	155(5)	139(4)	23(4)	42(4)	24(4)
C(7)	213(5)	182(5)	194(4)	39(4)	106(4)	7(4)
C(8)	153(5)	182(5)	168(4)	-19(4)	74(4)	7(4)
C(9)	143(5)	150(4)	141(4)	2(4)	40(3)	0(4)
C(10)	133(5)	131(4)	113(4)	-23(3)	29(3)	-17(4)
C(11)	184(5)	173(5)	155(4)	20(4)	62(4)	-11(4)
C(12)	136(5)	248(6)	193(5)	6(4)	11(4)	0(4)
C(13)	198(6)	177(5)	240(5)	24(4)	63(4)	38(5)

Table A2.5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for spirolactone **109** (CCDC 837088).

	x	y	z	U_{iso}
H(3)	5343(16)	7264(17)	6308(13)	38(4)
H(2A)	6355(12)	3286(14)	6419(11)	18(3)
H(2B)	7754(13)	2629(13)	7687(10)	18(3)
H(3A)	6191(13)	5112(14)	8255(10)	19(3)
H(3B)	6263(12)	3407(14)	9218(11)	19(3)
H(5A)	2155(13)	2052(13)	6656(10)	16(3)
H(5B)	3458(12)	2861(13)	5863(10)	13(3)
H(6A)	4038(12)	3843(13)	9990(10)	16(3)
H(6B)	3868(13)	1889(14)	9408(11)	26(3)
H(7A)	898(12)	2361(13)	8392(10)	15(3)
H(7B)	1405(13)	2780(15)	10024(11)	28(3)
H(8A)	1609(12)	5927(15)	9680(10)	15(2)
H(8B)	-81(13)	5306(12)	8868(10)	12(3)
H(10)	3709(12)	6580(12)	8147(10)	10(3)
H(11A)	2608(12)	5509(14)	5287(10)	14(3)
H(11B)	2749(12)	7663(13)	5842(9)	14(3)
H(12A)	25(13)	3944(16)	6195(11)	27(3)
H(12B)	99(13)	5811(16)	5453(10)	20(3)
H(12C)	-1080(15)	5566(15)	6397(10)	27(3)
H(13A)	-115(13)	8302(14)	7776(10)	16(3)
H(13B)	990(13)	8688(14)	6789(11)	26(3)
H(13C)	1749(13)	8684(14)	8399(11)	22(3)

Table A2.6. Hydrogen bonds for spirolactone **109** (CCDC 837088) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3)-H(3)...O(1)#1	0.780(12)	2.037(12)	2.8100(10)	171.5(13)

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

A2.2 CRYSTAL STRUCTURE ANALYSIS OF ALDEHYDE 127

Figure A2.2. Aldehyde **127**. 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 830951.

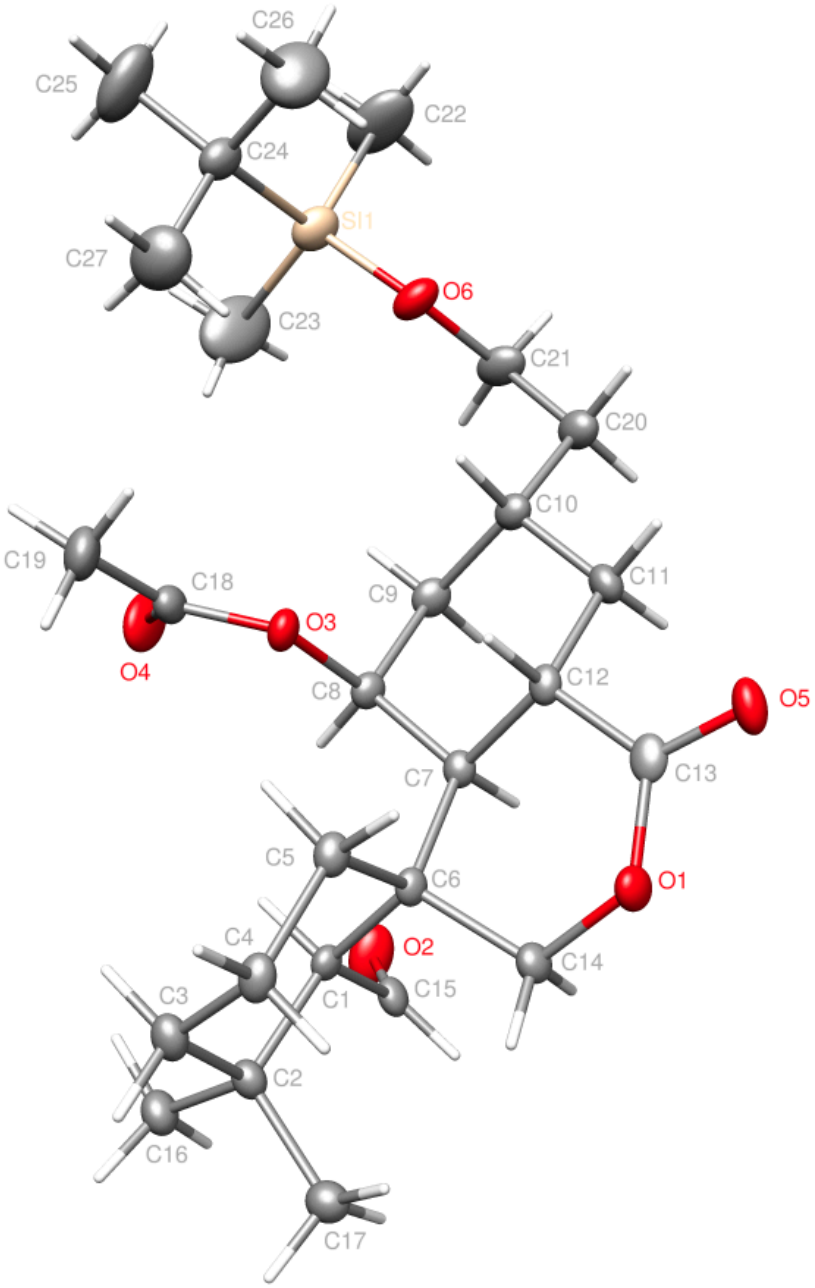
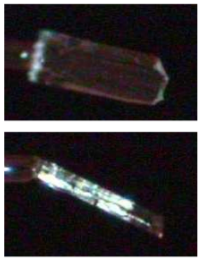


Table A2.7. Crystal data and structure refinement for aldehyde **127** (CCDC 830951).



Empirical formula	$C_{27}H_{46}O_6Si$
Formula weight	494.73
Crystallization Solvent	Ether
Crystal Habit	Plate
Crystal size	0.40 x 0.14 x 0.05 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 9883 reflections used in lattice determination	2.56 to 25.95°	
Unit cell dimensions	a = 7.0008(2) Å b = 14.2170(5) Å c = 28.7340(9) Å	α= 90° β= 90° γ = 90°
Volume	2859.91(16) $Å^3$	
Z	4	
Crystal system	Orthorhombic	
Space group	P $2_12_12_1$	
Density (calculated)	1.149 Mg/m^3	
F(000)	1080	
Data collection program	Bruker APEX2 v2009.7-0	
θ range for data collection	2.02 to 30.56°	
Completeness to θ = 30.56°	99.8 %	
Index ranges	$-10 \leq h \leq 6$, $-20 \leq k \leq 20$, $-41 \leq l \leq 39$	
Data collection scan type	ω scans; 9 settings	
Data reduction program	Bruker SAINT-Plus v7.66A	
Reflections collected	62150	
Independent reflections	8741 [R_{int}= 0.0589]	
Absorption coefficient	0.118 mm^{-1}	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7461 and 0.6633	

Table A2.7 (cont.)

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	8741 / 0 / 315
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.797
Final R indices [I>2σ(I), 7186 reflections]	R1 = 0.0453, *w*R2 = 0.0579
R indices (all data)	R1 = 0.0634, *w*R2 = 0.0594
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 determination	Unknown
Absolute structure parameter	0.07(7)
Largest diff. peak and hole	0.592 and -0.387 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 A2.8. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for aldehyde **127** (CCDC 830951). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Si(1)	14159(1)	5918(1)	7588(1)	29(1)
O(1)	5263(1)	6642(1)	9945(1)	20(1)
O(2)	11944(1)	5324(1)	10481(1)	33(1)
O(3)	9511(1)	4778(1)	9005(1)	19(1)
O(4)	12207(1)	3915(1)	8990(1)	38(1)
O(5)	4978(1)	7610(1)	9361(1)	23(1)
O(6)	12638(1)	6504(1)	7909(1)	27(1)
C(1)	8905(2)	4690(1)	10257(1)	15(1)
C(2)	7988(2)	3911(1)	10569(1)	18(1)
C(3)	6711(2)	3307(1)	10254(1)	21(1)
C(4)	5222(2)	3885(1)	9995(1)	20(1)
C(5)	6176(2)	4609(1)	9679(1)	18(1)
C(6)	7543(2)	5282(1)	9939(1)	14(1)
C(7)	8769(2)	5904(1)	9609(1)	15(1)
C(8)	10377(2)	5446(1)	9330(1)	16(1)
C(9)	11508(2)	6173(1)	9062(1)	20(1)
C(10)	10287(2)	6773(1)	8735(1)	19(1)
C(11)	8627(2)	7207(1)	9004(1)	19(1)
C(12)	7490(2)	6485(1)	9285(1)	16(1)
C(13)	5864(2)	6962(1)	9531(1)	18(1)
C(14)	6387(2)	5989(1)	10226(1)	18(1)
C(15)	10245(2)	5291(1)	10541(1)	18(1)
C(16)	9628(2)	3300(1)	10760(1)	24(1)
C(17)	6838(2)	4271(1)	10987(1)	23(1)
C(18)	10579(2)	4026(1)	8878(1)	24(1)
C(19)	9408(2)	3359(1)	8593(1)	34(1)
C(20)	11434(2)	7545(1)	8496(1)	25(1)
C(21)	13182(2)	7208(1)	8238(1)	29(1)
C(22)	15483(3)	6733(1)	7199(1)	63(1)
C(23)	15881(2)	5281(2)	7969(1)	68(1)
C(24)	12623(2)	5082(1)	7256(1)	28(1)
C(25)	13809(3)	4506(1)	6914(1)	66(1)
C(26)	11102(3)	5619(1)	6984(1)	62(1)
C(27)	11605(2)	4425(1)	7594(1)	56(1)

Table A2.9. Bond lengths [Å] and angles [°] for aldehyde **127** (CCDC 830951).

Si(1)-O(6)	1.6355(9)	C(15)-C(1)-C(6)	112.88(10)
Si(1)-C(22)	1.8584(17)	C(2)-C(1)-C(6)	117.75(10)
Si(1)-C(23)	1.8635(17)	C(17)-C(2)-C(3)	110.03(10)
Si(1)-C(24)	1.8647(15)	C(17)-C(2)-C(16)	107.40(10)
O(1)-C(13)	1.3433(14)	C(3)-C(2)-C(16)	109.25(10)
O(1)-C(14)	1.4589(13)	C(17)-C(2)-C(1)	115.34(10)
O(2)-C(15)	1.2024(13)	C(3)-C(2)-C(1)	107.34(10)
O(3)-C(18)	1.3539(15)	C(16)-C(2)-C(1)	107.34(10)
O(3)-C(8)	1.4648(14)	C(4)-C(3)-C(2)	112.66(11)
O(4)-C(18)	1.1946(15)	C(3)-C(4)-C(5)	110.86(10)
O(5)-C(13)	1.2125(14)	C(4)-C(5)-C(6)	113.57(10)
O(6)-C(21)	1.4286(15)	C(14)-C(6)-C(5)	109.84(9)
C(1)-C(15)	1.5095(17)	C(14)-C(6)-C(7)	104.33(10)
C(1)-C(2)	1.5634(17)	C(5)-C(6)-C(7)	113.46(10)
C(1)-C(6)	1.5654(16)	C(14)-C(6)-C(1)	111.19(10)
C(2)-C(17)	1.5346(16)	C(5)-C(6)-C(1)	109.00(10)
C(2)-C(3)	1.5335(17)	C(7)-C(6)-C(1)	108.98(9)
C(2)-C(16)	1.5411(17)	C(8)-C(7)-C(12)	109.97(9)
C(3)-C(4)	1.5223(17)	C(8)-C(7)-C(6)	119.00(11)
C(4)-C(5)	1.5269(17)	C(12)-C(7)-C(6)	110.76(9)
C(5)-C(6)	1.5472(16)	O(3)-C(8)-C(9)	109.54(9)
C(6)-C(14)	1.5310(16)	O(3)-C(8)-C(7)	107.82(9)
C(6)-C(7)	1.5545(16)	C(9)-C(8)-C(7)	111.20(10)
C(7)-C(8)	1.5272(16)	C(8)-C(9)-C(10)	113.64(10)
C(7)-C(12)	1.5333(16)	C(20)-C(10)-C(9)	112.59(10)
C(8)-C(9)	1.5131(17)	C(20)-C(10)-C(11)	109.77(11)
C(9)-C(10)	1.5287(16)	C(9)-C(10)-C(11)	109.88(10)
C(10)-C(20)	1.5253(17)	C(12)-C(11)-C(10)	113.04(10)
C(10)-C(11)	1.5266(16)	C(13)-C(12)-C(11)	109.82(10)
C(11)-C(12)	1.5286(17)	C(13)-C(12)-C(7)	113.61(10)
C(12)-C(13)	1.5021(17)	C(11)-C(12)-C(7)	112.23(9)
C(18)-C(19)	1.4975(19)	O(5)-C(13)-O(1)	117.02(11)
C(20)-C(21)	1.5089(18)	O(5)-C(13)-C(12)	122.82(12)
C(24)-C(27)	1.523(2)	O(1)-C(13)-C(12)	120.06(11)
C(24)-C(26)	1.526(2)	O(1)-C(14)-C(6)	113.88(9)
C(24)-C(25)	1.525(2)	O(2)-C(15)-C(1)	124.07(12)
		O(4)-C(18)-O(3)	124.03(13)
O(6)-Si(1)-C(22)	110.23(7)	O(4)-C(18)-C(19)	125.78(13)
O(6)-Si(1)-C(23)	109.73(7)	O(3)-C(18)-C(19)	110.17(12)
C(22)-Si(1)-C(23)	109.49(9)	C(21)-C(20)-C(10)	114.84(11)
O(6)-Si(1)-C(24)	103.71(6)	O(6)-C(21)-C(20)	109.37(10)
C(22)-Si(1)-C(24)	112.19(7)	C(27)-C(24)-C(26)	107.83(13)
C(23)-Si(1)-C(24)	111.36(8)	C(27)-C(24)-C(25)	109.58(14)
C(13)-O(1)-C(14)	122.50(9)	C(26)-C(24)-C(25)	108.56(13)
C(18)-O(3)-C(8)	117.04(9)	C(27)-C(24)-Si(1)	109.61(10)
C(21)-O(6)-Si(1)	123.77(8)	C(26)-C(24)-Si(1)	110.22(11)
C(15)-C(1)-C(2)	110.26(9)	C(25)-C(24)-Si(1)	110.97(11)

Table A2.10. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for aldehyde **127** (CCDC 830951). 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}
Si(1)	247(2)	349(3)	278(2)	-58(2)	22(2)	4(2)
O(1)	170(5)	187(5)	247(5)	9(4)	21(4)	55(4)
O(2)	152(5)	329(6)	522(7)	-116(5)	-16(5)	-9(4)
O(3)	197(5)	142(5)	223(5)	-44(4)	13(4)	13(4)
O(4)	351(6)	327(7)	466(6)	-82(5)	-71(5)	188(5)
O(5)	211(5)	137(5)	352(5)	8(4)	-52(4)	25(4)
O(6)	278(5)	303(6)	223(5)	-96(5)	37(4)	-44(4)
C(1)	150(6)	133(6)	166(6)	-20(6)	22(5)	13(5)
C(2)	190(7)	141(7)	204(7)	16(6)	6(6)	-21(5)
C(3)	222(7)	148(7)	274(7)	5(6)	22(6)	-32(6)
C(4)	177(7)	150(7)	266(7)	-43(6)	14(6)	-36(5)
C(5)	160(7)	155(7)	216(7)	-26(6)	-17(5)	-1(5)
C(6)	122(6)	132(7)	180(6)	-14(6)	-2(5)	9(5)
C(7)	137(6)	129(6)	176(6)	-15(6)	-11(5)	-17(5)
C(8)	146(6)	166(7)	180(6)	-21(6)	6(5)	6(5)
C(9)	159(7)	215(8)	214(7)	-27(6)	15(6)	-25(5)
C(10)	231(7)	153(7)	177(6)	-25(6)	20(6)	-44(6)
C(11)	249(7)	133(7)	199(7)	18(6)	-22(6)	-5(5)
C(12)	153(6)	121(7)	192(6)	-29(6)	-23(6)	-14(5)
C(13)	143(7)	146(7)	254(7)	-48(6)	-59(6)	-34(6)
C(14)	167(7)	158(7)	200(7)	17(6)	-4(5)	23(6)
C(15)	188(7)	127(7)	215(7)	22(6)	-26(6)	14(6)
C(16)	286(8)	174(7)	264(7)	11(6)	-12(6)	-3(6)
C(17)	258(7)	213(8)	227(7)	3(6)	26(6)	-44(6)
C(18)	333(9)	164(8)	207(7)	16(6)	41(6)	64(6)
C(19)	502(10)	177(8)	340(8)	-71(7)	15(8)	13(7)
C(20)	355(8)	187(8)	221(7)	-19(6)	30(6)	-62(6)
C(21)	356(9)	283(9)	239(8)	-24(7)	79(7)	-139(7)
C(22)	701(13)	639(13)	554(11)	-180(10)	350(10)	-266(11)
C(23)	465(11)	926(16)	653(12)	-139(12)	-236(10)	253(12)
C(24)	328(8)	275(9)	231(7)	-46(7)	-3(7)	46(7)
C(25)	713(13)	542(14)	728(13)	-340(11)	146(11)	-5(11)
C(26)	682(12)	647(15)	547(11)	-108(10)	-309(10)	67(11)
C(27)	716(12)	432(12)	530(11)	-51(9)	30(10)	-201(9)