APPENDIX THREE

X-Ray Crystallographic Data Relevant to Chapter Two

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Ketone viii (DCB03)

(CCDC 172324)

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Table 3. Selected bond distances and angles
Table 4. Anisotropic displacement parameters
Table 5. Hydrogen atomic coordinates

Figure A3.1 Representation of Ketone **viii**

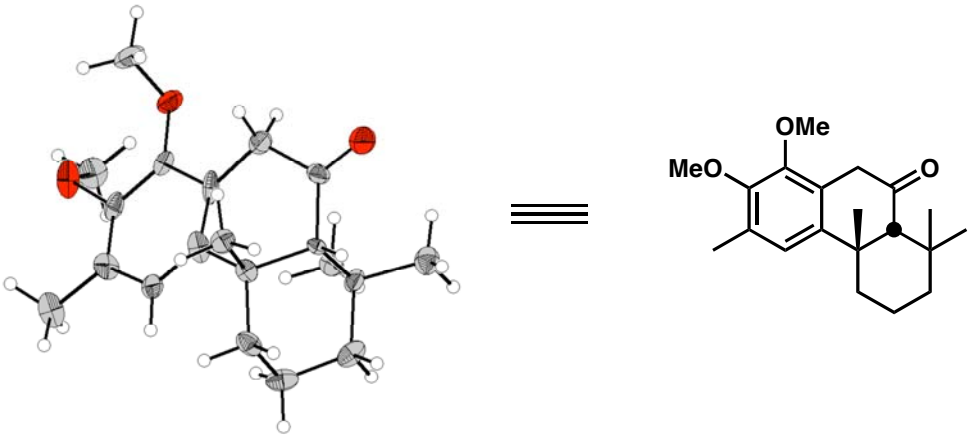

Table 1. Crystal data and structure refinement for DCB03 (CCDC 172324).

Empirical formula	$C_{20}H_{28}O_3$
Formula weight	316.42
Crystallization Solvent	Hexanes
Crystal Habit	Block
Crystal size	0.30 x 0.22 x 0.19 mm^3
Crystal color	Colorless

Data Collection

Preliminary Photos	Rotation
Type of diffractometer	CCD area detector
Wavelength	0.71073 Å MoKα
Data Collection Temperature	98(2) K
θ range for 9714 reflections used in lattice determination	2.60 to 28.29°
Unit cell dimensions	a = 7.900(3) Å b = 8.104(5) Å β= 97.212(6)° c = 13.664(4) Å
Volume	867.8(7) $Å^3$
Z	2
Crystal system	Monoclinic
Space group	$P2_1$
Density (calculated)	1.211 Mg/m^3
F(000)	344
Data collection program	Bruker SMART
θ range for data collection	2.93 to 28.38°
Completeness to θ = 28.38°	29.9 %
Index ranges	$-9 \le h \le 9$, $-10 \le k \le 10$, $-18 \le l \le 17$
Data collection scan type	ω scans at 7 ϕ settings
Data reduction program	Bruker SAINT v6.2
Reflections collected	4065
Independent reflections	4065 [R_{int}= 0.0000]
Absorption coefficient	0.080 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9854 and 0.9768

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	4065 / 1 / 215
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	0.983
Final R indices [$I>2\sigma(I)$, 2775 reflections]	R1 = 0.0513, *w*R2 = 0.0823
R indices (all data)	R1 = 0.0790, *w*R2 = 0.0900
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	2(3)
Largest diff. peak and hole	0.110 and -0.108 e.Å^{-3}

Special Refinement Details

The crystals are twinned. The twinning is non-merohedral and therefore (since the reciprocal lattices don't overlap exactly) the data required special handling. An orientation matrix was identified for each twin and each matrix was used to integrate the data for each twin component. The two resulting data sets were merged into one set that contained reflections unique to each twin component and reflections exactly overlapping between the two twin components. For component one there were 3057 exact overlaps, 14021 partial overlaps and 538 unique, for component two the corresponding numbers are 3060, 14136 and 505. The data set used for least squares refinement contained only 696 reflections after merging equivalents from both twin components into a single reflection..

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 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB03 (CCDC 172324). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	5945(12)	3813(7)	2029(5)	33(1)
O(2)	3847(9)	5388(4)	564(3)	34(1)
O(4)	4869(8)	-122(3)	4754(2)	37(1)
C(1)	4219(13)	3657(8)	2004(6)	16(2)
C(2)	3094(15)	4363(9)	1223(8)	24(2)
C(3)	1350(19)	4257(9)	1208(7)	26(2)
C(4)	745(12)	3298(5)	1930(5)	27(1)
C(5)	1789(17)	2637(11)	2680(7)	23(2)
C(6)	1034(11)	1640(7)	3497(5)	20(1)
C(7)	1112(10)	2803(4)	4392(3)	27(1)
C(8)	-797(10)	1109(7)	3252(4)	27(1)
C(9)	-1031(8)	-266(5)	2461(3)	30(1)
C(10)	64(12)	-1751(6)	2817(5)	29(2)
C(11)	1952(10)	-1365(6)	3057(4)	25(1)
C(12)	2789(11)	-2888(3)	3597(3)	31(1)
C(13)	2752(8)	-1097(5)	2147(3)	24(1)
C(14)	2119(9)	111(5)	3821(4)	18(1)
C(15)	3981(10)	547(6)	4091(4)	24(1)
C(16)	4684(10)	1943(5)	3536(5)	28(1)
C(17)	3524(18)	2647(10)	2719(7)	26(3)
C(18)	6548(9)	5292(5)	2521(5)	41(1)
C(19)	4648(11)	4484(7)	-129(3)	35(1)
C(20)	190(13)	5086(7)	378(4)	34(2)

Table 3. Bond lengths [Å] and angles [°] for DCB03 (CCDC 172324).

O(1)-C(1)	1.365(12)	C(20)-H(20A)	0.9800
O(1)-C(18)	1.426(7)	C(20)-H(20B)	0.9800
O(2)-C(2)	1.410(12)	C(20)-H(20C)	0.9800
O(2)-C(19)	1.409(8)		
O(4)-C(15)	1.203(7)	C(1)-O(1)-C(18)	111.3(7)
C(1)-C(2)	1.421(16)	C(2)-O(2)-C(19)	112.6(5)
C(1)-C(17)	1.435(14)	O(1)-C(1)-C(2)	120.7(9)
C(2)-C(3)	1.378(17)	O(1)-C(1)-C(17)	120.1(10)
C(3)-C(4)	1.387(13)	C(2)-C(1)-C(17)	119.0(10)
C(3)-C(20)	1.522(15)	C(3)-C(2)-O(2)	121.9(12)
C(4)-C(5)	1.343(15)	C(3)-C(2)-C(1)	121.1(11)
C(4)-H(4)	0.9500	O(2)-C(2)-C(1)	116.3(10)
C(5)-C(17)	1.365(19)	C(2)-C(3)-C(4)	117.2(12)
C(5)-C(6)	1.557(11)	C(2)-C(3)-C(20)	119.4(11)
C(6)-C(8)	1.506(11)	C(4)-C(3)-C(20)	123.3(12)
C(6)-C(7)	1.539(6)	C(5)-C(4)-C(3)	122.2(11)
C(6)-C(14)	1.539(9)	C(5)-C(4)-H(4)	118.9
C(7)-H(7A)	0.9800	C(3)-C(4)-H(4)	118.9
C(7)-H(7B)	0.9800	C(4)-C(5)-C(17)	123.0(11)
C(7)-H(7C)	0.9800	C(4)-C(5)-C(6)	120.0(10)
C(8)-C(9)	1.547(6)	C(17)-C(5)-C(6)	116.7(12)
C(8)-H(8A)	0.9900	C(8)-C(6)-C(7)	107.1(6)
C(8)-H(8B)	0.9900	C(8)-C(6)-C(14)	108.6(5)
C(9)-C(10)	1.525(9)	C(7)-C(6)-C(14)	107.4(5)
C(9)-H(9A)	0.9900	C(8)-C(6)-C(5)	115.6(8)
C(9)-H(9B)	0.9900	C(7)-C(6)-C(5)	105.7(5)
C(10)-C(11)	1.520(10)	C(14)-C(6)-C(5)	111.9(7)
C(10)-H(10A)	0.9900	C(6)-C(7)-H(7A)	109.5
C(10)-H(10B)	0.9900	C(6)-C(7)-H(7B)	109.5
C(11)-C(13)	1.479(8)	H(7A)-C(7)-H(7B)	109.5
C(11)-C(12)	1.544(7)	C(6)-C(7)-H(7C)	109.5
C(11)-C(14)	1.582(6)	H(7A)-C(7)-H(7C)	109.5
C(12)-H(12A)	0.9800	H(7B)-C(7)-H(7C)	109.5
C(12)-H(12B)	0.9800	C(6)-C(8)-C(9)	112.9(6)
C(12)-H(12C)	0.9800	C(6)-C(8)-H(8A)	109.0
C(13)-H(13A)	0.9800	C(9)-C(8)-H(8A)	109.0
C(13)-H(13B)	0.9800	C(6)-C(8)-H(8B)	109.0
C(13)-H(13C)	0.9800	C(9)-C(8)-H(8B)	109.0
C(14)-C(15)	1.513(9)	H(8A)-C(8)-H(8B)	107.8
C(14)-H(14)	1.0000	C(10)-C(9)-C(8)	109.4(5)
C(15)-C(16)	1.506(8)	C(10)-C(9)-H(9A)	109.8
C(16)-C(17)	1.468(14)	C(8)-C(9)-H(9A)	109.8
C(16)-H(16A)	0.9900	C(10)-C(9)-H(9B)	109.8
C(16)-H(16B)	0.9900	C(8)-C(9)-H(9B)	109.8
C(18)-H(18A)	0.9800	H(9A)-C(9)-H(9B)	108.2
C(18)-H(18B)	0.9800	C(11)-C(10)-C(9)	114.1(5)
C(18)-H(18C)	0.9800	C(11)-C(10)-H(10A)	108.7
C(19)-H(19A)	0.9800	C(9)-C(10)-H(10A)	108.7
C(19)-H(19B)	0.9800	C(11)-C(10)-H(10B)	108.7
C(19)-H(19C)	0.9800	C(9)-C(10)-H(10B)	108.7

H(10A)-C(10)-H(10B)	107.6	H(20B)-C(20)-H(20C)	109.5
C(13)-C(11)-C(10)	111.1(6)		
C(13)-C(11)-C(12)	108.5(5)		
C(10)-C(11)-C(12)	106.8(6)		
C(13)-C(11)-C(14)	115.8(5)		
C(10)-C(11)-C(14)	107.2(6)		
C(12)-C(11)-C(14)	107.1(4)		
C(11)-C(12)-H(12A)	109.5		
C(11)-C(12)-H(12B)	109.5		
H(12A)-C(12)-H(12B)	109.5		
C(11)-C(12)-H(12C)	109.5		
H(12A)-C(12)-H(12C)	109.5		
H(12B)-C(12)-H(12C)	109.5		
C(11)-C(13)-H(13A)	109.5		
C(11)-C(13)-H(13B)	109.5		
H(13A)-C(13)-H(13B)	109.5		
C(11)-C(13)-H(13C)	109.5		
H(13A)-C(13)-H(13C)	109.5		
H(13B)-C(13)-H(13C)	109.5		
C(15)-C(14)-C(6)	111.8(5)		
C(15)-C(14)-C(11)	109.6(5)		
C(6)-C(14)-C(11)	115.0(5)		
C(15)-C(14)-H(14)	106.7		
C(6)-C(14)-H(14)	106.7		
C(11)-C(14)-H(14)	106.7		
O(4)-C(15)-C(16)	120.0(7)		
O(4)-C(15)-C(14)	122.4(7)		
C(16)-C(15)-C(14)	117.5(6)		
C(17)-C(16)-C(15)	116.2(8)		
C(17)-C(16)-H(16A)	108.2		
C(15)-C(16)-H(16A)	108.2		
C(17)-C(16)-H(16B)	108.2		
C(15)-C(16)-H(16B)	108.2		
H(16A)-C(16)-H(16B)	107.4		
C(5)-C(17)-C(1)	116.4(13)		
C(5)-C(17)-C(16)	123.7(11)		
C(1)-C(17)-C(16)	118.9(11)		
O(1)-C(18)-H(18A)	109.5		
O(1)-C(18)-H(18B)	109.5		
H(18A)-C(18)-H(18B)	109.5		
O(1)-C(18)-H(18C)	109.5		
H(18A)-C(18)-H(18C)	109.5		
H(18B)-C(18)-H(18C)	109.5		
O(2)-C(19)-H(19A)	109.5		
O(2)-C(19)-H(19B)	109.5		
H(19A)-C(19)-H(19B)	109.5		
O(2)-C(19)-H(19C)	109.5		
H(19A)-C(19)-H(19C)	109.5		
H(19B)-C(19)-H(19C)	109.5		
C(3)-C(20)-H(20A)	109.5		
C(3)-C(20)-H(20B)	109.5		
H(20A)-C(20)-H(20B)	109.5		
C(3)-C(20)-H(20C)	109.5		
H(20A)-C(20)-H(20C)	109.5		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB03 (CCDC 172324). 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}
O(1)	230(30)	240(20)	520(30)	-2(18)	90(30)	-50(30)
O(2)	500(40)	275(16)	307(19)	80(13)	280(40)	130(30)
O(4)	370(30)	356(13)	372(15)	109(13)	-10(30)	-60(30)
C(1)	220(40)	150(20)	150(30)	-4(19)	170(30)	-20(30)
C(2)	310(50)	120(20)	330(40)	-10(20)	180(40)	20(30)
C(3)	350(50)	330(30)	70(30)	20(20)	-10(40)	10(40)
C(4)	230(30)	147(19)	420(30)	-56(19)	40(40)	40(30)
C(5)	350(50)	260(20)	130(40)	20(20)	170(40)	130(40)
C(6)	190(30)	220(30)	180(30)	-20(20)	70(40)	50(30)
C(7)	330(30)	224(18)	240(20)	-92(16)	-10(30)	-10(30)
C(8)	180(30)	360(30)	270(30)	60(20)	60(30)	60(30)
C(9)	240(30)	430(20)	250(20)	-58(18)	60(30)	-90(30)
C(10)	300(40)	320(30)	280(30)	40(20)	170(40)	-120(40)
C(11)	220(40)	130(20)	410(30)	-11(18)	60(40)	-10(30)
C(12)	380(40)	252(19)	300(20)	-23(15)	30(40)	10(30)
C(13)	240(30)	228(19)	236(18)	-72(15)	10(30)	30(30)
C(14)	150(30)	180(20)	190(30)	17(17)	-60(30)	0(30)
C(15)	200(30)	210(20)	280(30)	-62(19)	-60(40)	30(30)
C(16)	170(30)	190(20)	490(30)	-20(20)	100(40)	20(30)
C(17)	430(60)	110(20)	280(50)	0(20)	160(50)	110(40)
C(18)	240(30)	266(19)	720(30)	-120(20)	100(40)	-90(30)
C(19)	380(40)	400(20)	290(20)	24(16)	110(40)	0(40)
C(20)	440(50)	370(30)	220(30)	40(20)	140(40)	150(40)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for DCB03 (CCDC 172324).

	x	y	z	U_{iso}
H(4)	-447	3103	1893	32
H(7A)	796	2194	4961	40
H(7B)	316	3722	4237	40
H(7C)	2274	3235	4547	40
H(8A)	-1494	2076	3011	32
H(8B)	-1224	706	3860	32
H(9A)	-2246	-596	2340	36
H(9B)	-688	149	1833	36
H(10A)	-357	-2206	3414	35
H(10B)	-80	-2615	2301	35
H(12A)	2521	-3874	3193	46
H(12B)	2350	-3021	4232	46
H(12C)	4029	-2734	3710	46
H(13A)	2606	-2088	1734	36
H(13B)	3971	-872	2322	36
H(13C)	2207	-156	1784	36
H(14)	1703	-311	4436	21
H(16A)	5041	2838	4011	33
H(16B)	5719	1545	3270	33
H(18A)	6205	5309	3186	61
H(18B)	6058	6248	2149	61
H(18C)	7795	5335	2566	61
H(19A)	3814	3748	-498	53
H(19B)	5580	3827	216	53
H(19C)	5106	5247	-586	53
H(20A)	624	6194	264	51
H(20B)	-967	5170	562	51
H(20C)	167	4430	-226	51

CALIFORNIA INSTITUTE OF TECHNOLOGY

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Crystal Structure Analysis of:

Lactone 156 (DCB06)

(CCDC 175859)

Contents:

Table 1. Crystal data
Table 2. Atomic coordinates
Table 3. Full bond distances and angles (for deposit)
Table 4. Anisotropic displacement parameters
Table 5. Hydrogen atomic coordinates

Figure A3.2 Representation of Lactone **156**

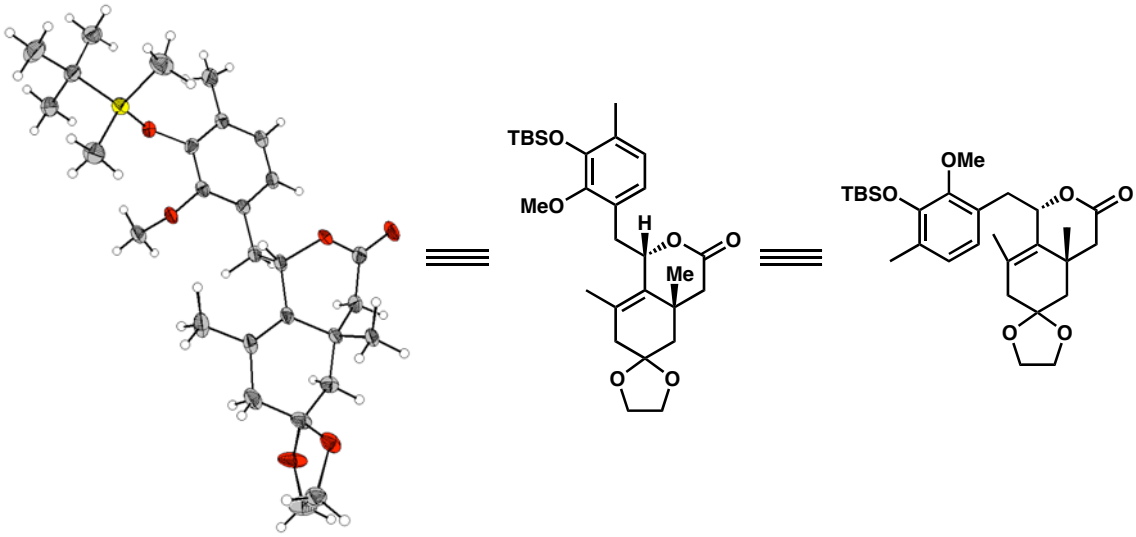

Table 1. Crystal data and structure refinement for DCB06_(CCDC_175859).

Empirical formula	$C_{28}H_{42}O_6Si$
Formula weight	502.71
Crystallization Solvent	Hexanes
Crystal Habit	Block
Crystal size	0.33 x 0.17 x 0.14 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker P4
Wavelength	0.71073 Å MoKα
Data Collection Temperature	96(2) K
θ range for 8201 reflections used in lattice determination	2.79 to 26.49°
Unit cell dimensions	a = 29.220(3) Å b = 6.7215(8) Å β= 90.035(2)° c = 14.4249(17) Å
Volume	2833.0(6) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.179 Mg/m^3
F(000)	1088
Data collection program	Bruker SMART v5.054
θ range for data collection	1.39 to 28.38°
Completeness to θ = 28.38°	93.9 %
Index ranges	$-37 \le h \le 38$, $-8 \le k \le 8$, $-19 \le l \le 19$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.22
Reflections collected	38935
Independent reflections	6656 [R_{int}= 0.0985]
Absorption coefficient	0.120 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9829 and 0.9610

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	Bruker SHELXTL
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	Bruker SHELXTL
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	6656 / 0 / 484
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.403
Final R indices [I>2σ(I), 3988 reflections]	R1 = 0.0584, *w*R2 = 0.0804
R indices (all data)	R1 = 0.1062, *w*R2 = 0.0844
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
Largest diff. peak and hole	0.333 and -0.349 e.Å^{-3}

Special Refinement Details

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 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB06_(CCDC_175859). U(eq) the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Si(1)	6142(1)	3545(1)	4087(1)	25(1)
O(1)	6571(1)	5169(2)	4135(1)	23(1)
O(2)	7293(1)	2606(2)	3725(1)	23(1)
O(3)	8211(1)	1108(2)	6169(1)	28(1)
O(4)	8640(1)	2202(2)	7317(1)	44(1)
O(5)	9414(1)	-4435(2)	4438(1)	30(1)
O(6)	9725(1)	-2363(2)	3364(1)	45(1)
C(1)	5665(1)	5006(3)	3527(1)	27(1)
C(2)	5841(1)	5905(4)	2608(2)	35(1)
C(3)	5260(1)	3633(5)	3329(2)	43(1)
C(4)	5503(1)	6694(4)	4154(2)	36(1)
C(5)	6282(1)	1358(4)	3362(2)	34(1)
C(6)	5995(1)	2734(5)	5291(2)	39(1)
C(7)	6962(1)	5171(3)	4672(1)	21(1)
C(8)	6990(1)	6481(3)	5429(1)	24(1)
C(9)	6595(1)	7823(4)	5660(2)	37(1)
C(10)	7397(1)	6526(4)	5928(1)	27(1)
C(11)	7762(1)	5352(3)	5700(1)	26(1)
C(12)	7740(1)	4049(3)	4942(1)	22(1)
C(13)	7334(1)	3973(3)	4446(1)	20(1)
C(14)	7349(1)	3475(4)	2815(1)	32(1)
C(15)	8145(1)	2785(3)	4670(1)	22(1)
C(16)	8169(1)	761(3)	5164(1)	22(1)
C(17)	8623(1)	1598(3)	6522(1)	28(1)
C(18)	9030(1)	1238(4)	5922(2)	26(1)
C(19)	8979(1)	-684(3)	5354(1)	19(1)
C(20)	8948(1)	-2437(4)	6036(1)	24(1)
C(21)	9394(1)	-899(3)	4715(1)	24(1)
C(22)	9349(1)	-2544(3)	4006(1)	28(1)
C(23)	9638(1)	-5668(3)	3767(2)	29(1)
C(24)	9927(1)	-4215(4)	3241(2)	39(1)
C(25)	8901(1)	-2427(4)	3480(2)	33(1)
C(26)	8502(1)	-1441(3)	3960(1)	24(1)
C(27)	8074(1)	-1401(5)	3388(2)	35(1)
C(28)	8545(1)	-548(3)	4787(1)	19(1)

Table 3. Bond lengths [Å] and angles [°] for DCB06_(CCDC_175859).

Si(1)-O(1)	1.6629(15)	C(16)-C(28)	1.510(3)
Si(1)-C(5)	1.849(3)	C(16)-H(16)	0.995(16)
Si(1)-C(6)	1.872(2)	C(17)-C(18)	1.490(3)
Si(1)-C(1)	1.887(2)	C(18)-C(19)	1.538(3)
O(1)-C(7)	1.379(2)	C(18)-H(18A)	0.96(2)
O(2)-C(13)	1.392(2)	C(18)-H(18B)	0.96(2)
O(2)-C(14)	1.446(2)	C(19)-C(28)	1.512(3)
O(3)-C(17)	1.350(2)	C(19)-C(21)	1.531(3)
O(3)-C(16)	1.473(2)	C(19)-C(20)	1.538(3)
O(4)-C(17)	1.218(2)	C(20)-H(20A)	0.987(19)
O(5)-C(22)	1.428(2)	C(20)-H(20B)	1.016(18)
O(5)-C(23)	1.433(2)	C(20)-H(20C)	1.003(19)
O(6)-C(24)	1.389(3)	C(21)-C(22)	1.512(3)
O(6)-C(22)	1.443(2)	C(21)-H(21A)	0.99(2)
C(1)-C(4)	1.527(3)	C(21)-H(21B)	0.99(2)
C(1)-C(3)	1.528(3)	C(22)-C(25)	1.516(3)
C(1)-C(2)	1.545(3)	C(23)-C(24)	1.498(3)
C(2)-H(2A)	1.05(2)	C(23)-H(23A)	0.98(2)
C(2)-H(2B)	0.99(2)	C(23)-H(23B)	0.940(19)
C(2)-H(2C)	1.00(2)	C(24)-H(24A)	0.86(3)
C(3)-H(3A)	0.98(2)	C(24)-H(24B)	0.97(3)
C(3)-H(3B)	0.95(2)	C(25)-C(26)	1.509(3)
C(3)-H(3C)	1.04(2)	C(25)-H(25A)	0.99(2)
C(4)-H(4A)	0.99(2)	C(25)-H(25B)	0.98(2)
C(4)-H(4B)	0.99(2)	C(26)-C(28)	1.340(2)
C(4)-H(4C)	0.99(2)	C(26)-C(27)	1.498(3)
C(5)-H(5A)	0.97(2)	C(27)-H(27A)	0.95(2)
C(5)-H(5B)	1.01(2)	C(27)-H(27B)	0.97(2)
C(5)-H(5C)	0.98(2)	C(27)-H(27C)	1.06(2)
C(6)-H(6A)	1.03(3)		
C(6)-H(6B)	0.98(3)	O(1)-Si(1)-C(5)	112.26(10)
C(6)-H(6C)	0.98(2)	O(1)-Si(1)-C(6)	109.05(11)
C(7)-C(13)	1.392(3)	C(5)-Si(1)-C(6)	110.16(13)
C(7)-C(8)	1.405(3)	O(1)-Si(1)-C(1)	103.44(9)
C(8)-C(10)	1.389(3)	C(5)-Si(1)-C(1)	109.60(11)
C(8)-C(9)	1.503(3)	C(6)-Si(1)-C(1)	112.21(11)
C(9)-H(9A)	1.02(2)	C(7)-O(1)-Si(1)	130.34(12)
C(9)-H(9B)	0.92(2)	C(13)-O(2)-C(14)	113.73(17)
C(9)-H(9C)	0.95(2)	C(17)-O(3)-C(16)	118.89(15)
C(10)-C(11)	1.369(3)	C(22)-O(5)-C(23)	106.28(14)
C(10)-H(10)	0.98(2)	C(24)-O(6)-C(22)	109.25(17)
C(11)-C(12)	1.403(3)	C(4)-C(1)-C(3)	108.6(2)
C(11)-H(11)	0.965(19)	C(4)-C(1)-C(2)	108.7(2)
C(12)-C(13)	1.388(3)	C(3)-C(1)-C(2)	109.5(2)
C(12)-C(15)	1.508(3)	C(4)-C(1)-Si(1)	111.25(15)
C(14)-H(14A)	0.99(2)	C(3)-C(1)-Si(1)	109.77(18)
C(14)-H(14B)	1.00(2)	C(2)-C(1)-Si(1)	108.96(15)
C(14)-H(14C)	1.05(2)	C(1)-C(2)-H(2A)	113.2(11)
C(15)-C(16)	1.538(3)	C(1)-C(2)-H(2B)	109.5(13)
C(15)-H(15A)	1.047(18)	H(2A)-C(2)-H(2B)	106.4(17)
C(15)-H(15B)	0.950(18)	C(1)-C(2)-H(2C)	108.6(12)

H(2A)-C(2)-H(2C)	108.2(18)	H(14A)-C(14)-H(14C)	108.8(17)
H(2B)-C(2)-H(2C)	110.9(18)	H(14B)-C(14)-H(14C)	114.6(17)
C(1)-C(3)-H(3A)	110.1(14)	C(12)-C(15)-C(16)	114.35(16)
C(1)-C(3)-H(3B)	110.8(14)	C(12)-C(15)-H(15A)	112.3(10)
H(3A)-C(3)-H(3B)	108.4(19)	C(16)-C(15)-H(15A)	104.7(10)
C(1)-C(3)-H(3C)	113.7(12)	C(12)-C(15)-H(15B)	109.6(11)
H(3A)-C(3)-H(3C)	108.5(18)	C(16)-C(15)-H(15B)	108.4(11)
H(3B)-C(3)-H(3C)	105.0(19)	H(15A)-C(15)-H(15B)	107.1(14)
C(1)-C(4)-H(4A)	109.9(14)	O(3)-C(16)-C(28)	112.77(15)
C(1)-C(4)-H(4B)	112.8(12)	O(3)-C(16)-C(15)	108.69(16)
H(4A)-C(4)-H(4B)	110.7(17)	C(28)-C(16)-C(15)	112.34(16)
C(1)-C(4)-H(4C)	115.2(12)	O(3)-C(16)-H(16)	101.4(9)
H(4A)-C(4)-H(4C)	104.1(17)	C(28)-C(16)-H(16)	113.5(10)
H(4B)-C(4)-H(4C)	103.8(18)	C(15)-C(16)-H(16)	107.4(10)
Si(1)-C(5)-H(5A)	113.1(14)	O(4)-C(17)-O(3)	118.11(19)
Si(1)-C(5)-H(5B)	112.4(13)	O(4)-C(17)-C(18)	124.7(2)
H(5A)-C(5)-H(5B)	108(2)	O(3)-C(17)-C(18)	117.06(17)
Si(1)-C(5)-H(5C)	110.5(14)	C(17)-C(18)-C(19)	111.65(18)
H(5A)-C(5)-H(5C)	106.6(19)	C(17)-C(18)-H(18A)	109.4(12)
H(5B)-C(5)-H(5C)	106.0(18)	C(19)-C(18)-H(18A)	112.2(12)
Si(1)-C(6)-H(6A)	110.3(13)	C(17)-C(18)-H(18B)	111.5(11)
Si(1)-C(6)-H(6B)	114.0(13)	C(19)-C(18)-H(18B)	111.1(12)
H(6A)-C(6)-H(6B)	107(2)	H(18A)-C(18)-H(18B)	100.5(16)
Si(1)-C(6)-H(6C)	107.1(13)	C(28)-C(19)-C(21)	110.20(15)
H(6A)-C(6)-H(6C)	111.0(19)	C(28)-C(19)-C(18)	108.56(17)
H(6B)-C(6)-H(6C)	108(2)	C(21)-C(19)-C(18)	108.89(16)
O(1)-C(7)-C(13)	120.97(17)	C(28)-C(19)-C(20)	110.06(16)
O(1)-C(7)-C(8)	119.01(17)	C(21)-C(19)-C(20)	111.11(17)
C(13)-C(7)-C(8)	119.93(18)	C(18)-C(19)-C(20)	107.94(16)
C(10)-C(8)-C(7)	117.79(19)	C(19)-C(20)-H(20A)	111.0(10)
C(10)-C(8)-C(9)	121.9(2)	C(19)-C(20)-H(20B)	108.3(10)
C(7)-C(8)-C(9)	120.24(19)	H(20A)-C(20)-H(20B)	107.8(14)
C(8)-C(9)-H(9A)	109.2(13)	C(19)-C(20)-H(20C)	110.5(11)
C(8)-C(9)-H(9B)	112.5(13)	H(20A)-C(20)-H(20C)	104.4(15)
H(9A)-C(9)-H(9B)	111.6(18)	H(20B)-C(20)-H(20C)	114.8(14)
C(8)-C(9)-H(9C)	112.0(13)	C(22)-C(21)-C(19)	113.99(17)
H(9A)-C(9)-H(9C)	106.5(18)	C(22)-C(21)-H(21A)	107.4(12)
H(9B)-C(9)-H(9C)	104.8(19)	C(19)-C(21)-H(21A)	112.5(11)
C(11)-C(10)-C(8)	122.0(2)	C(22)-C(21)-H(21B)	107.6(10)
C(11)-C(10)-H(10)	120.2(12)	C(19)-C(21)-H(21B)	110.2(10)
C(8)-C(10)-H(10)	117.8(12)	H(21A)-C(21)-H(21B)	104.7(16)
C(10)-C(11)-C(12)	120.8(2)	O(5)-C(22)-O(6)	104.75(15)
C(10)-C(11)-H(11)	120.4(11)	O(5)-C(22)-C(21)	110.15(16)
C(12)-C(11)-H(11)	118.8(11)	O(6)-C(22)-C(21)	107.79(17)
C(13)-C(12)-C(11)	117.63(19)	O(5)-C(22)-C(25)	112.29(19)
C(13)-C(12)-C(15)	121.07(18)	O(6)-C(22)-C(25)	109.41(17)
C(11)-C(12)-C(15)	121.30(19)	C(21)-C(22)-C(25)	112.09(19)
C(12)-C(13)-C(7)	121.79(18)	O(5)-C(23)-C(24)	102.91(18)
C(12)-C(13)-O(2)	118.78(17)	O(5)-C(23)-H(23A)	110.8(11)
C(7)-C(13)-O(2)	119.39(17)	C(24)-C(23)-H(23A)	113.5(12)
O(2)-C(14)-H(14A)	107.7(11)	O(5)-C(23)-H(23B)	109.3(12)
O(2)-C(14)-H(14B)	103.7(13)	C(24)-C(23)-H(23B)	112.7(12)
H(14A)-C(14)-H(14B)	111.7(17)	H(23A)-C(23)-H(23B)	107.6(17)
O(2)-C(14)-H(14C)	109.9(11)	O(6)-C(24)-C(23)	106.3(2)

O(6)-C(24)-H(24A)	111(2)
C(23)-C(24)-H(24A)	121(2)
O(6)-C(24)-H(24B)	107.4(15)
C(23)-C(24)-H(24B)	109.6(15)
H(24A)-C(24)-H(24B)	101(2)
C(26)-C(25)-C(22)	117.35(18)
C(26)-C(25)-H(25A)	105.2(13)
C(22)-C(25)-H(25A)	103.6(13)
C(26)-C(25)-H(25B)	109.0(12)
C(22)-C(25)-H(25B)	108.3(12)
H(25A)-C(25)-H(25B)	113.4(18)
C(28)-C(26)-C(27)	124.0(2)
C(28)-C(26)-C(25)	122.27(19)
C(27)-C(26)-C(25)	113.50(18)
C(26)-C(27)-H(27A)	111.8(14)
C(26)-C(27)-H(27B)	110.0(13)
H(27A)-C(27)-H(27B)	108.2(19)
C(26)-C(27)-H(27C)	118.2(11)
H(27A)-C(27)-H(27C)	102.5(18)
H(27B)-C(27)-H(27C)	105.5(18)
C(26)-C(28)-C(16)	120.96(18)
C(26)-C(28)-C(19)	122.09(18)
C(16)-C(28)-C(19)	116.83(16)

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Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for DCB06_(CCDC_175859). 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}
Si(1)	252(3)	279(4)	224(3)	4(3)	10(3)	-5(3)
O(1)	223(8)	280(9)	195(7)	-17(6)	-35(6)	7(6)
O(2)	277(8)	286(9)	134(7)	-31(6)	-19(6)	-16(7)
O(3)	292(9)	444(10)	110(7)	15(7)	31(6)	67(7)
O(4)	623(12)	535(12)	148(8)	-121(7)	-86(7)	211(9)
O(5)	483(10)	221(9)	186(7)	34(7)	67(7)	24(7)
O(6)	607(11)	324(10)	426(9)	130(8)	373(8)	165(8)
C(1)	206(12)	327(14)	273(12)	19(10)	-8(10)	-14(10)
C(2)	339(16)	452(18)	257(13)	51(12)	-44(12)	55(14)
C(3)	243(15)	500(19)	547(18)	-31(17)	-58(13)	-29(14)
C(4)	287(15)	436(17)	370(15)	43(13)	11(12)	69(14)
C(5)	344(16)	311(15)	366(15)	-48(12)	17(12)	-74(14)
C(6)	465(18)	396(17)	313(14)	73(13)	77(13)	51(15)
C(7)	190(12)	250(13)	199(11)	50(9)	-14(9)	-7(10)
C(8)	254(12)	283(13)	190(10)	6(10)	-4(9)	29(10)
C(9)	400(17)	389(17)	309(15)	-133(14)	-63(12)	122(14)
C(10)	315(14)	336(14)	170(11)	-59(11)	-37(10)	-4(11)
C(11)	274(14)	329(14)	165(11)	3(10)	-41(10)	-10(11)
C(12)	225(12)	279(13)	143(10)	36(9)	23(9)	1(9)
C(13)	237(12)	217(13)	151(10)	13(9)	9(9)	-59(10)
C(14)	336(16)	453(16)	160(11)	-4(12)	-1(10)	-19(14)
C(15)	208(13)	273(13)	170(11)	13(10)	-15(9)	-33(10)
C(16)	202(12)	323(14)	128(10)	10(9)	-18(9)	-19(10)
C(17)	328(14)	308(14)	207(11)	3(10)	-45(10)	114(11)
C(18)	266(14)	297(15)	202(11)	-55(11)	-68(10)	5(11)
C(19)	191(11)	245(12)	126(10)	-11(9)	3(8)	-18(9)
C(20)	228(14)	356(15)	125(11)	21(11)	-11(10)	15(11)
C(21)	253(13)	218(14)	240(12)	33(10)	54(10)	-15(10)
C(22)	378(14)	250(13)	209(11)	52(10)	128(10)	45(11)
C(23)	364(15)	201(13)	292(13)	-18(11)	14(12)	51(12)
C(24)	351(17)	247(15)	576(18)	-68(13)	127(15)	-24(12)
C(25)	501(16)	330(16)	164(12)	6(12)	1(11)	53(13)
C(26)	318(13)	239(12)	150(10)	21(10)	-44(9)	-16(10)
C(27)	458(17)	310(16)	269(13)	-22(13)	-157(12)	-2(13)
C(28)	208(12)	244(13)	125(10)	42(9)	2(9)	-23(9)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for DCB06_(CCDC_175859).

	x	y	z	U_{iso}
H(2A)	6133(7)	6800(30)	2693(13)	38(6)
H(2B)	5928(7)	4820(40)	2180(15)	49(7)
H(2C)	5594(8)	6750(40)	2334(15)	49(7)
H(3A)	5005(8)	4400(40)	3059(15)	54(8)
H(3B)	5343(7)	2620(40)	2906(15)	47(8)
H(3C)	5141(7)	2890(30)	3909(15)	48(7)
H(4A)	5371(7)	6140(40)	4734(15)	53(7)
H(4B)	5749(7)	7660(30)	4298(13)	29(6)
H(4C)	5256(7)	7520(30)	3897(14)	38(6)
H(5A)	6544(8)	620(40)	3588(15)	55(8)
H(5B)	6018(8)	410(40)	3306(15)	53(7)
H(5C)	6356(8)	1770(40)	2728(16)	53(7)
H(6A)	5864(8)	3910(40)	5662(16)	68(8)
H(6B)	5767(8)	1660(40)	5317(15)	60(8)
H(6C)	6278(8)	2230(30)	5577(15)	50(7)
H(9A)	6319(8)	6970(30)	5851(14)	47(7)
H(9B)	6667(7)	8740(30)	6106(14)	36(7)
H(9C)	6499(7)	8590(30)	5140(16)	48(7)
H(10)	7416(7)	7440(30)	6454(14)	36(6)
H(11)	8045(6)	5450(30)	6044(12)	27(6)
H(14A)	7663(7)	4000(30)	2774(12)	28(6)
H(14B)	7302(7)	2330(40)	2385(15)	49(7)
H(14C)	7118(7)	4650(30)	2727(13)	39(6)
H(15A)	8144(6)	2430(30)	3962(13)	27(5)
H(15B)	8421(6)	3490(30)	4792(11)	16(5)
H(16)	7859(6)	150(20)	5125(10)	8(4)
H(18A)	9081(6)	2380(30)	5535(13)	31(6)
H(18B)	9308(7)	1230(30)	6276(13)	31(6)
H(20A)	8897(6)	-3700(30)	5704(12)	18(5)
H(20B)	9252(6)	-2550(30)	6377(12)	21(5)
H(20C)	8673(7)	-2290(30)	6446(12)	26(5)
H(21A)	9683(7)	-1130(30)	5063(13)	34(6)
H(21B)	9448(6)	360(30)	4373(12)	24(5)
H(23A)	9815(7)	-6710(30)	4072(13)	34(6)
H(23B)	9417(6)	-6290(30)	3393(13)	24(6)
H(24A)	9995(11)	-4420(50)	2670(20)	112(14)
H(24B)	10229(9)	-4160(40)	3514(16)	64(9)
H(25A)	8972(7)	-1560(30)	2948(15)	51(7)
H(25B)	8812(7)	-3770(30)	3298(14)	39(7)
H(27A)	8088(8)	-420(40)	2917(16)	56(8)
H(27B)	8027(7)	-2690(40)	3096(15)	48(7)
H(27C)	7762(7)	-1060(30)	3722(13)	43(7)

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X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Acid 161•$CHCl_3$ (DCB05)

(CCDC 175588)

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Table 1. Crystal data
Table 2. Atomic coordinates
Table 3. Full bond distances and angles (for deposit)
Table 4. Anisotropic displacement parameters
Table 5. Hydrogen atomic coordinates
Table 6. Hydrogen bonds

Figure A3.3 Representation of Acid **161•$CHCl_3$**

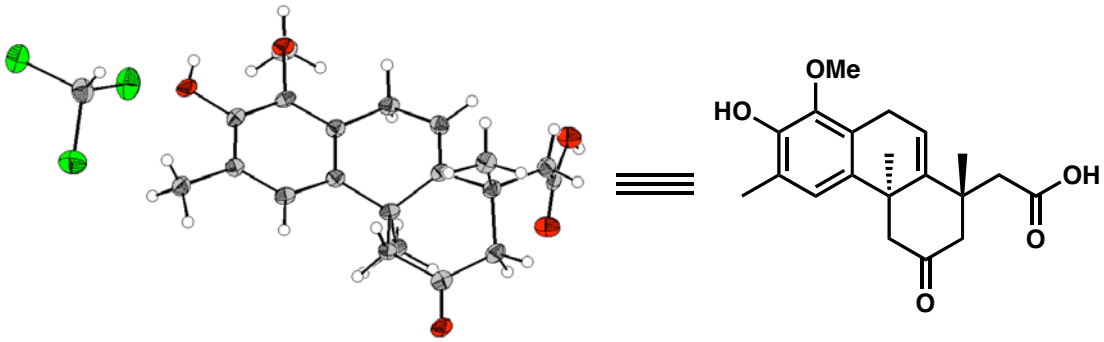

Table 1. Crystal data and structure refinement for DCB05 (CCDC 175588).

Empirical formula	$C_{20}H_{24}O_5 \cdot CHCl_3$
Formula weight	463.76
Crystallization Solvent	Chloroform
Crystal Habit	Fragment
Crystal size	0.22 x 0.15 x 0.15 mm^3
Crystal color	Colorless

Data Collection

Preliminary Photos	Rotation
Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	98(2) K
θ range for 4336 reflections used in lattice determination	2.47 to 25.80°
Unit cell dimensions	a = 11.137(3) Å b = 13.282(3) Å β= 98.762(4)° c = 15.008(4) Å
Volume	2194.3(10) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/n$
Density (calculated)	1.404 Mg/m^3
F(000)	968
Data collection program	Bruker SMART v5.054
θ range for data collection	2.06 to 28.36°
Completeness to θ = 28.36°	93.7 %
Index ranges	$-14 \le h \le 14$, $-17 \le k \le 17$, $-19 \le l \le 19$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.22
Reflections collected	32070
Independent reflections	5144 [R_{int}= 0.1503]
Absorption coefficient	0.447 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9368 and 0.9072

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	5144 / 0 / 362
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.064
Final R indices [$I>2\sigma(I)$, 2718 reflections]	R1 = 0.0468, *w*R2 = 0.0744
R indices (all data)	R1 = 0.1218, *w*R2 = 0.0862
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Largest diff. peak and hole	0.402 and -0.348 e.Å^{-3}

Special Refinement Details

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 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB05 (CCDC 175588). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	2996(2)	1246(2)	424(1)	23(1)
O(2)	3667(2)	289(1)	2011(1)	23(1)
O(3)	10816(2)	767(1)	809(1)	24(1)
O(4)	10340(2)	21(1)	3988(1)	28(1)
O(5)	9289(2)	-1153(1)	4583(1)	27(1)
C(1)	4196(2)	1102(2)	735(2)	20(1)
C(2)	4579(2)	649(2)	1547(2)	19(1)
C(3)	5789(2)	489(2)	1874(2)	19(1)
C(4)	6178(2)	-65(2)	2727(2)	22(1)
C(5)	7352(2)	-608(2)	2734(2)	20(1)
C(6)	8184(2)	-314(2)	2243(2)	18(1)
C(7)	9343(2)	-929(2)	2231(2)	20(1)
C(8)	9093(3)	-1750(2)	1504(2)	25(1)
C(9)	9748(3)	-1452(2)	3131(2)	22(1)
C(10)	9824(2)	-789(2)	3926(2)	21(1)
C(11)	10377(2)	-254(2)	2003(2)	23(1)
C(12)	10036(2)	410(2)	1214(2)	21(1)
C(13)	8738(2)	652(2)	926(2)	19(1)
C(14)	7993(2)	658(2)	1710(2)	18(1)
C(15)	8424(3)	1530(2)	2337(2)	22(1)
C(16)	6654(2)	805(2)	1345(2)	18(1)
C(17)	6244(2)	1260(2)	531(2)	19(1)
C(18)	5037(2)	1421(2)	199(2)	19(1)
C(19)	4615(3)	1892(3)	-688(2)	24(1)
C(20)	3502(3)	888(3)	2774(2)	28(1)
C(21)	1118(3)	3109(2)	603(2)	31(1)
Cl(1)	1475(1)	3147(1)	1778(1)	41(1)
Cl(2)	2089(1)	3910(1)	117(1)	45(1)
Cl(3)	-397(1)	3458(1)	263(1)	39(1)

Table 3. Bond lengths [Å] and angles [°] for DCB05 (CCDC 175588).

O(1)-C(1)	1.361(3)	C(21)-Cl(3)	1.749(3)
O(1)-H(1)	0.72(3)	C(21)-Cl(1)	1.749(3)
O(2)-C(2)	1.401(3)	C(21)-Cl(2)	1.753(3)
O(2)-C(20)	1.428(3)	C(21)-H(21)	0.91(3)
O(3)-C(12)	1.230(3)		
O(4)-C(10)	1.216(3)	C(1)-O(1)-H(1)	107(3)
O(5)-C(10)	1.319(3)	C(2)-O(2)-C(20)	113.6(2)
O(5)-H(5A)	0.97(4)	C(10)-O(5)-H(5A)	111.0(18)
C(1)-C(2)	1.368(4)	O(1)-C(1)-C(2)	121.6(2)
C(1)-C(18)	1.390(3)	O(1)-C(1)-C(18)	118.2(2)
C(2)-C(3)	1.378(3)	C(2)-C(1)-C(18)	120.3(2)
C(3)-C(16)	1.403(3)	C(1)-C(2)-C(3)	122.7(2)
C(3)-C(4)	1.483(4)	C(1)-C(2)-O(2)	116.2(2)
C(4)-C(5)	1.492(4)	C(3)-C(2)-O(2)	120.9(2)
C(4)-H(4A)	0.94(3)	C(2)-C(3)-C(16)	118.1(2)
C(4)-H(4B)	0.96(3)	C(2)-C(3)-C(4)	121.7(2)
C(5)-C(6)	1.328(3)	C(16)-C(3)-C(4)	120.1(2)
C(5)-H(5)	0.97(3)	C(3)-C(4)-C(5)	112.8(2)
C(6)-C(14)	1.516(4)	C(3)-C(4)-H(4A)	108.3(16)
C(6)-C(7)	1.529(3)	C(5)-C(4)-H(4A)	109.2(15)
C(7)-C(9)	1.525(4)	C(3)-C(4)-H(4B)	108.8(16)
C(7)-C(8)	1.538(4)	C(5)-C(4)-H(4B)	107.1(16)
C(7)-C(11)	1.539(3)	H(4A)-C(4)-H(4B)	111(2)
C(8)-H(8A)	0.95(3)	C(6)-C(5)-C(4)	122.6(3)
C(8)-H(8B)	1.02(3)	C(6)-C(5)-H(5)	119.0(14)
C(8)-H(8C)	1.03(3)	C(4)-C(5)-H(5)	118.4(14)
C(9)-C(10)	1.475(4)	C(5)-C(6)-C(14)	119.3(2)
C(9)-H(9A)	0.92(3)	C(5)-C(6)-C(7)	120.7(2)
C(9)-H(9B)	0.93(2)	C(14)-C(6)-C(7)	119.97(19)
C(11)-C(12)	1.478(4)	C(9)-C(7)-C(6)	111.69(19)
C(11)-H(11A)	0.99(3)	C(9)-C(7)-C(8)	107.6(2)
C(11)-H(11B)	1.00(3)	C(6)-C(7)-C(8)	109.0(2)
C(12)-C(13)	1.480(4)	C(9)-C(7)-C(11)	109.4(2)
C(13)-C(14)	1.541(3)	C(6)-C(7)-C(11)	110.7(2)
C(13)-H(13A)	0.94(2)	C(8)-C(7)-C(11)	108.4(2)
C(13)-H(13B)	1.00(2)	C(7)-C(8)-H(8A)	108.3(15)
C(14)-C(16)	1.520(4)	C(7)-C(8)-H(8B)	107.9(16)
C(14)-C(15)	1.523(4)	H(8A)-C(8)-H(8B)	109(2)
C(15)-H(15A)	1.00(2)	C(7)-C(8)-H(8C)	113.1(14)
C(15)-H(15B)	1.00(2)	H(8A)-C(8)-H(8C)	106(2)
C(15)-H(15C)	1.01(3)	H(8B)-C(8)-H(8C)	113(2)
C(16)-C(17)	1.378(4)	C(10)-C(9)-C(7)	114.7(2)
C(17)-C(18)	1.377(4)	C(10)-C(9)-H(9A)	111.5(15)
C(17)-H(17)	0.97(2)	C(7)-C(9)-H(9A)	114.9(16)
C(18)-C(19)	1.481(4)	C(10)-C(9)-H(9B)	105.6(15)
C(19)-H(19A)	0.94(3)	C(7)-C(9)-H(9B)	111.0(15)
C(19)-H(19B)	0.92(3)	H(9A)-C(9)-H(9B)	97(2)
C(19)-H(19C)	1.03(2)	O(4)-C(10)-O(5)	122.0(2)
C(20)-H(20A)	1.00(3)	O(4)-C(10)-C(9)	123.9(2)
C(20)-H(20B)	0.95(2)	O(5)-C(10)-C(9)	114.2(2)
C(20)-H(20C)	0.99(3)	C(12)-C(11)-C(7)	114.6(2)

C(12)-C(11)-H(11A)	106.0(16)
C(7)-C(11)-H(11A)	112.5(15)
C(12)-C(11)-H(11B)	113.7(14)
C(7)-C(11)-H(11B)	108.6(15)
H(11A)-C(11)-H(11B)	101(2)
O(3)-C(12)-C(11)	120.7(2)
O(3)-C(12)-C(13)	120.3(2)
C(11)-C(12)-C(13)	119.0(2)
C(12)-C(13)-C(14)	113.2(2)
C(12)-C(13)-H(13A)	109.8(13)
C(14)-C(13)-H(13A)	108.8(12)
C(12)-C(13)-H(13B)	110.1(13)
C(14)-C(13)-H(13B)	109.3(12)
H(13A)-C(13)-H(13B)	105.3(19)
C(6)-C(14)-C(16)	110.5(2)
C(6)-C(14)-C(15)	108.5(2)
C(16)-C(14)-C(15)	108.9(2)
C(6)-C(14)-C(13)	110.5(2)
C(16)-C(14)-C(13)	109.8(2)
C(15)-C(14)-C(13)	108.6(2)
C(14)-C(15)-H(15A)	109.5(14)
C(14)-C(15)-H(15B)	111.6(14)
H(15A)-C(15)-H(15B)	107.9(18)
C(14)-C(15)-H(15C)	116.1(16)
H(15A)-C(15)-H(15C)	111(2)
H(15B)-C(15)-H(15C)	100(2)
C(17)-C(16)-C(3)	118.0(2)
C(17)-C(16)-C(14)	123.4(2)
C(3)-C(16)-C(14)	118.6(2)
C(18)-C(17)-C(16)	124.3(2)
C(18)-C(17)-H(17)	117.2(15)
C(16)-C(17)-H(17)	118.6(15)
C(17)-C(18)-C(1)	116.7(2)
C(17)-C(18)-C(19)	123.3(2)
C(1)-C(18)-C(19)	120.0(2)
C(18)-C(19)-H(19A)	114.1(17)
C(18)-C(19)-H(19B)	111.4(17)
H(19A)-C(19)-H(19B)	109(2)
C(18)-C(19)-H(19C)	109.9(15)
H(19A)-C(19)-H(19C)	104(2)
H(19B)-C(19)-H(19C)	107(2)
O(2)-C(20)-H(20A)	111.9(14)
O(2)-C(20)-H(20B)	104.7(14)
H(20A)-C(20)-H(20B)	108(2)
O(2)-C(20)-H(20C)	112.1(16)
H(20A)-C(20)-H(20C)	108(2)
H(20B)-C(20)-H(20C)	111(2)
Cl(3)-C(21)-Cl(1)	110.32(16)
Cl(3)-C(21)-Cl(2)	110.32(17)
Cl(1)-C(21)-Cl(2)	109.98(17)
Cl(3)-C(21)-H(21)	109.2(19)
Cl(1)-C(21)-H(21)	105.5(19)
Cl(2)-C(21)-H(21)	111.4(16)

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for DCB05 (CCDC 175588). 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}
O(1)	149(10)	334(13)	235(11)	33(9)	80(9)	-25(9)
O(2)	182(10)	311(11)	210(10)	7(9)	113(8)	-10(8)
O(3)	154(10)	348(11)	253(11)	24(9)	102(8)	-2(8)
O(4)	324(11)	318(12)	214(10)	-30(9)	98(9)	-60(9)
O(5)	325(11)	325(12)	182(11)	-13(10)	95(9)	-42(9)
C(1)	142(14)	230(15)	221(15)	-30(12)	23(11)	8(12)
C(2)	142(14)	248(15)	205(15)	-29(12)	82(11)	-27(11)
C(3)	174(15)	228(15)	167(14)	-8(12)	58(11)	4(11)
C(4)	183(15)	279(17)	222(16)	16(14)	92(12)	-9(13)
C(5)	223(15)	228(16)	165(15)	17(13)	41(12)	4(12)
C(6)	155(14)	222(15)	159(14)	-23(11)	31(11)	-4(11)
C(7)	164(14)	230(15)	198(15)	8(12)	36(11)	1(11)
C(8)	240(17)	298(18)	208(16)	-20(14)	47(13)	18(14)
C(9)	190(16)	242(16)	242(16)	20(13)	49(12)	24(14)
C(10)	148(14)	267(17)	208(15)	27(13)	-21(12)	41(12)
C(11)	150(15)	294(17)	243(16)	-13(14)	53(12)	15(13)
C(12)	216(15)	196(15)	230(16)	-72(12)	56(12)	-7(12)
C(13)	175(15)	235(16)	172(15)	24(13)	71(12)	9(12)
C(14)	158(14)	222(15)	186(14)	-19(12)	92(11)	-10(11)
C(15)	192(15)	254(16)	217(16)	-18(13)	85(12)	-7(13)
C(16)	169(14)	203(14)	180(14)	-3(11)	79(11)	13(11)
C(17)	176(14)	229(15)	194(15)	-22(12)	92(12)	-2(12)
C(18)	184(14)	216(15)	191(14)	-5(12)	67(11)	22(11)
C(19)	181(16)	298(18)	228(16)	44(15)	29(13)	-2(14)
C(20)	237(18)	400(20)	250(17)	-48(15)	140(15)	-19(15)
C(21)	345(18)	274(18)	296(17)	-15(15)	10(14)	23(15)
Cl(1)	443(5)	519(5)	245(4)	-55(4)	17(3)	122(4)
Cl(2)	415(5)	447(5)	491(5)	63(4)	117(4)	-38(4)
Cl(3)	331(4)	475(5)	339(4)	-10(4)	21(3)	69(4)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for DCB05 (CCDC 175588).

	x	y	z	U_{iso}
H(1)	2670(30)	960(20)	710(20)	37(11)
H(4A)	6260(20)	400(20)	3203(17)	23(7)
H(4B)	5570(20)	-560(20)	2799(17)	29(7)
H(5)	7500(20)	-1210(20)	3098(17)	29(8)
H(5A)	9470(30)	-750(30)	5120(20)	60(10)
H(8A)	8480(20)	-2180(20)	1656(18)	30(8)
H(8B)	9870(30)	-2160(20)	1514(18)	40(8)
H(8C)	8770(20)	-1467(19)	875(19)	30(7)
H(9A)	9340(20)	-2040(20)	3214(16)	19(7)
H(9B)	10520(20)	-1729(19)	3152(16)	22(7)
H(11A)	10710(20)	190(20)	2508(19)	39(8)
H(11B)	11100(20)	-680(20)	1959(16)	29(7)
H(13A)	8666(18)	1287(17)	642(14)	3(6)
H(13B)	8370(20)	165(18)	456(16)	16(7)
H(15A)	8294(19)	2176(18)	1999(15)	11(6)
H(15B)	7970(20)	1565(18)	2856(17)	22(7)
H(15C)	9280(30)	1480(20)	2670(19)	39(8)
H(17)	6830(20)	1466(18)	154(16)	23(7)
H(19A)	4240(20)	2520(20)	-652(18)	31(8)
H(19B)	4110(20)	1470(20)	-1056(19)	33(8)
H(19C)	5350(20)	2042(19)	-1011(17)	32(7)
H(20A)	4260(30)	910(20)	3234(18)	29(8)
H(20B)	2880(20)	553(17)	3031(15)	14(6)
H(20C)	3270(20)	1590(20)	2602(19)	38(9)
H(21)	1220(20)	2450(20)	447(19)	39(9)

Table 6. Hydrogen bonds for DCB05 (CCDC 175588) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1)...O(3)#1	0.72(3)	2.10(3)	2.656(2)	135(3)
O(1)-H(1)...O(2)	0.72(3)	2.28(3)	2.703(3)	119(3)
O(5)-H(5A)...O(4)#2	0.97(4)	1.64(4)	2.600(3)	175(3)

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

CALIFORNIA INSTITUTE OF TECHNOLOGY

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X-RAY CRYSTALLOGRAPHY LABORATORY

Crystal Structure Analysis of:

Diketone 175 (DCB11)

(CCDC 201187)

Contents:

Table 1. Crystal data
Table 2. Atomic coordinates
Table 3. Full bond distances and angles (for deposit)
Table 4. Anisotropic displacement parameters
Table 5. Hydrogen atomic coordinates

Figure A3.4 Representation of Diketone **175**.

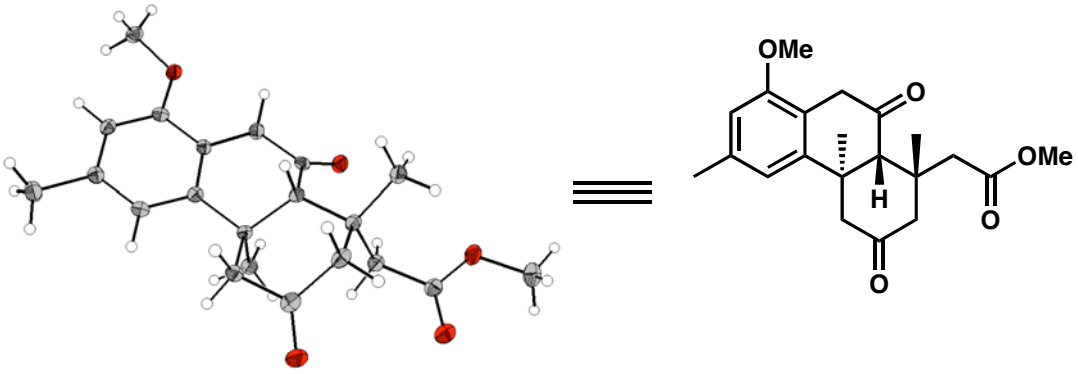

Table 1. Crystal data and structure refinement for DCB11 (CCDC 201187).

Empirical formula	$C_{21}H_{26}O_5$
Formula weight	358.42
Crystallization Solvent	Acetone/heptane
Crystal Habit	Fragment
Crystal size	0.26 x 0.22 x 0.17 mm^3
Crystal color	Colorless

Data Collection

Preliminary Photos	Rotation
Type of diffractometer	Bruker SMART 1000
Wavelength	0.71073 Å MoKα
Data Collection Temperature	98(2) K
θ range for 11980 reflections used in lattice determination	2.28 to 28.32°
Unit cell dimensions	a = 9.0211(6) Å b = 11.3617(7) Å β= 97.5510(10)° c = 17.9596(12) Å
Volume	1824.8(2) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/n$
Density (calculated)	1.305 Mg/m^3
F(000)	768
Data collection program	Bruker SMART v5.054
θ range for data collection	2.13 to 28.32°
Completeness to θ = 28.32°	93.0 %
Index ranges	$-11 \le h \le 11$, $-14 \le k \le 14$, $-23 \le l \le 23$
Data collection scan type	ω scans at 5 ϕ settings
Data reduction program	Bruker SAINT v6.022
Reflections collected	25862
Independent reflections	4226 [R_{int}= 0.0517]
Absorption coefficient	0.092 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9845 and 0.9764

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	4226 / 0 / 339
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	2.153
Final R indices [$I>2\sigma(I)$, 3426 reflections]	R1 = 0.0404, *w*R2 = 0.0704
R indices (all data)	R1 = 0.0511, *w*R2 = 0.0715
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
Largest diff. peak and hole	0.326 and -0.254 e.Å^{-3}

Special Refinement Details

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 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DCB11 (CCDC 201187). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
O(1)	7731(1)	4507(1)	529(1)	19(1)
O(2)	4749(1)	8200(1)	472(1)	21(1)
O(3)	9216(1)	11345(1)	2166(1)	25(1)
O(4)	3377(1)	11397(1)	1288(1)	24(1)
O(5)	5418(1)	12446(1)	1710(1)	29(1)
C(1)	10631(1)	7158(1)	1330(1)	16(1)
C(2)	11220(1)	6047(1)	1256(1)	16(1)
C(3)	12869(2)	5814(1)	1455(1)	23(1)
C(4)	10262(1)	5133(1)	986(1)	16(1)
C(5)	8757(1)	5346(1)	789(1)	15(1)
C(6)	8217(2)	3305(1)	562(1)	20(1)
C(7)	8162(1)	6478(1)	851(1)	14(1)
C(8)	6510(1)	6650(1)	623(1)	18(1)
C(9)	5914(1)	7834(1)	811(1)	15(1)
C(10)	6851(1)	8484(1)	1442(1)	14(1)
C(11)	6098(1)	9579(1)	1751(1)	15(1)
C(12)	4682(1)	9175(1)	2071(1)	19(1)
C(13)	5708(1)	10548(1)	1148(1)	17(1)
C(14)	4865(1)	11569(1)	1417(1)	18(1)
C(15)	2478(2)	12330(1)	1540(1)	31(1)
C(16)	7204(1)	10084(1)	2400(1)	18(1)
C(17)	8678(1)	10359(1)	2139(1)	18(1)
C(18)	9450(1)	9319(1)	1844(1)	18(1)
C(19)	8466(1)	8626(1)	1220(1)	14(1)
C(20)	8500(2)	9259(1)	463(1)	19(1)
C(21)	9104(1)	7387(1)	1138(1)	14(1)

Table 3. Bond lengths [Å] and angles [°] for DCB11 (CCDC 201187).

O(1)-C(5)	1.3673(13)	C(20)-H(20A)	0.996(14)
O(1)-C(6)	1.4332(14)	C(20)-H(20B)	0.987(13)
O(2)-C(9)	1.2162(13)	C(20)-H(20C)	0.985(13)
O(3)-C(17)	1.2186(13)		
O(4)-C(14)	1.3457(14)	C(5)-O(1)-C(6)	117.41(9)
O(4)-C(15)	1.4438(16)	C(14)-O(4)-C(15)	115.32(10)
O(5)-C(14)	1.2036(14)	C(2)-C(1)-C(21)	121.54(11)
C(1)-C(2)	1.3828(16)	C(2)-C(1)-H(1)	118.7(7)
C(1)-C(21)	1.3997(16)	C(21)-C(1)-H(1)	119.8(7)
C(1)-H(1)	1.006(12)	C(1)-C(2)-C(4)	118.97(11)
C(2)-C(4)	1.3963(16)	C(1)-C(2)-C(3)	121.17(11)
C(2)-C(3)	1.5069(17)	C(4)-C(2)-C(3)	119.86(11)
C(3)-H(3A)	0.967(14)	C(2)-C(3)-H(3A)	109.9(8)
C(3)-H(3B)	0.963(16)	C(2)-C(3)-H(3B)	111.4(9)
C(3)-H(3C)	0.980(15)	H(3A)-C(3)-H(3B)	109.7(12)
C(4)-C(5)	1.3783(16)	C(2)-C(3)-H(3C)	111.9(9)
C(4)-H(4)	0.958(11)	H(3A)-C(3)-H(3C)	108.7(12)
C(5)-C(7)	1.4037(15)	H(3B)-C(3)-H(3C)	105.1(12)
C(6)-H(6A)	0.978(12)	C(5)-C(4)-C(2)	120.19(11)
C(6)-H(6B)	0.973(12)	C(5)-C(4)-H(4)	119.0(7)
C(6)-H(6C)	0.999(13)	C(2)-C(4)-H(4)	120.8(7)
C(7)-C(21)	1.3926(16)	O(1)-C(5)-C(4)	124.44(10)
C(7)-C(8)	1.5051(16)	O(1)-C(5)-C(7)	114.64(10)
C(8)-C(9)	1.5034(16)	C(4)-C(5)-C(7)	120.92(11)
C(8)-H(8A)	0.993(13)	O(1)-C(6)-H(6A)	105.1(7)
C(8)-H(8B)	0.996(13)	O(1)-C(6)-H(6B)	110.4(7)
C(9)-C(10)	1.5144(16)	H(6A)-C(6)-H(6B)	110.7(10)
C(10)-C(11)	1.5546(16)	O(1)-C(6)-H(6C)	111.8(7)
C(10)-C(19)	1.5679(16)	H(6A)-C(6)-H(6C)	110.0(10)
C(10)-H(10)	0.982(11)	H(6B)-C(6)-H(6C)	108.8(10)
C(11)-C(12)	1.5377(16)	C(5)-C(7)-C(21)	119.20(11)
C(11)-C(13)	1.5525(16)	C(5)-C(7)-C(8)	118.20(10)
C(11)-C(16)	1.5420(16)	C(21)-C(7)-C(8)	122.58(10)
C(12)-H(12A)	0.986(12)	C(7)-C(8)-C(9)	115.21(10)
C(12)-H(12B)	1.025(12)	C(7)-C(8)-H(8A)	112.9(8)
C(12)-H(12C)	0.993(12)	C(9)-C(8)-H(8A)	106.4(7)
C(13)-C(14)	1.5018(16)	C(7)-C(8)-H(8B)	109.6(8)
C(13)-H(13A)	0.986(12)	C(9)-C(8)-H(8B)	105.9(8)
C(13)-H(13B)	0.957(11)	H(8A)-C(8)-H(8B)	106.3(11)
C(15)-H(15A)	0.969(14)	O(2)-C(9)-C(10)	124.61(11)
C(15)-H(15B)	0.980(15)	O(2)-C(9)-C(8)	120.35(11)
C(15)-H(15C)	0.989(15)	C(10)-C(9)-C(8)	115.03(10)
C(16)-C(17)	1.4999(17)	C(9)-C(10)-C(11)	115.52(10)
C(16)-H(16A)	0.980(12)	C(9)-C(10)-C(19)	107.82(9)
C(16)-H(16B)	0.982(12)	C(11)-C(10)-C(19)	118.21(9)
C(17)-C(18)	1.5039(17)	C(9)-C(10)-H(10)	104.1(6)
C(18)-C(19)	1.5492(16)	C(11)-C(10)-H(10)	105.7(6)
C(18)-H(18A)	0.966(12)	C(19)-C(10)-H(10)	103.8(6)
C(18)-H(18B)	0.996(12)	C(12)-C(11)-C(10)	108.53(9)
C(19)-C(21)	1.5357(15)	C(12)-C(11)-C(13)	110.46(10)
C(19)-C(20)	1.5424(16)	C(10)-C(11)-C(13)	112.81(9)

C(12)-C(11)-C(16)	108.40(10)
C(10)-C(11)-C(16)	107.36(9)
C(13)-C(11)-C(16)	109.15(10)
C(11)-C(12)-H(12A)	109.5(7)
C(11)-C(12)-H(12B)	110.2(7)
H(12A)-C(12)-H(12B)	109.2(9)
C(11)-C(12)-H(12C)	109.5(7)
H(12A)-C(12)-H(12C)	108.7(10)
H(12B)-C(12)-H(12C)	109.7(9)
C(14)-C(13)-C(11)	113.64(10)
C(14)-C(13)-H(13A)	104.7(7)
C(11)-C(13)-H(13A)	110.9(7)
C(14)-C(13)-H(13B)	108.1(7)
C(11)-C(13)-H(13B)	109.4(7)
H(13A)-C(13)-H(13B)	110.0(10)
O(5)-C(14)-O(4)	122.87(11)
O(5)-C(14)-C(13)	125.55(12)
O(4)-C(14)-C(13)	111.58(10)
O(4)-C(15)-H(15A)	107.3(8)
O(4)-C(15)-H(15B)	109.3(9)
H(15A)-C(15)-H(15B)	110.0(12)
O(4)-C(15)-H(15C)	110.6(8)
H(15A)-C(15)-H(15C)	111.5(12)
H(15B)-C(15)-H(15C)	108.1(12)
C(17)-C(16)-C(11)	110.67(10)
C(17)-C(16)-H(16A)	108.5(7)
C(11)-C(16)-H(16A)	111.5(7)
C(17)-C(16)-H(16B)	108.4(7)
C(11)-C(16)-H(16B)	107.5(7)
H(16A)-C(16)-H(16B)	110.2(9)
O(3)-C(17)-C(18)	122.40(11)
O(3)-C(17)-C(16)	122.92(11)
C(18)-C(17)-C(16)	114.67(11)
C(17)-C(18)-C(19)	113.93(10)
C(17)-C(18)-H(18A)	109.0(7)
C(19)-C(18)-H(18A)	110.2(7)
C(17)-C(18)-H(18B)	105.8(7)
C(19)-C(18)-H(18B)	109.9(7)
H(18A)-C(18)-H(18B)	107.8(9)
C(21)-C(19)-C(20)	106.90(9)
C(21)-C(19)-C(18)	110.43(9)
C(20)-C(19)-C(18)	108.92(10)
C(21)-C(19)-C(10)	107.65(9)
C(20)-C(19)-C(10)	113.48(10)
C(18)-C(19)-C(10)	109.42(9)
C(19)-C(20)-H(20A)	108.7(7)
C(19)-C(20)-H(20B)	111.1(7)
H(20A)-C(20)-H(20B)	107.8(10)
C(19)-C(20)-H(20C)	112.4(7)
H(20A)-C(20)-H(20C)	107.0(10)
H(20B)-C(20)-H(20C)	109.7(10)
C(1)-C(21)-C(7)	119.14(11)
C(1)-C(21)-C(19)	121.02(10)
C(7)-C(21)-C(19)	119.82(10)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DCB11 (CCDC 201187). 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}
O(1)	179(5)	129(4)	246(5)	-34(4)	5(4)	-8(3)
O(2)	175(5)	198(5)	252(5)	-10(4)	-23(4)	19(4)
O(3)	239(5)	164(5)	340(5)	-50(4)	-12(4)	-28(4)
O(4)	181(5)	202(5)	316(5)	-22(4)	1(4)	57(4)
O(5)	270(5)	197(5)	396(6)	-85(4)	44(4)	-6(4)
C(1)	170(6)	162(6)	152(6)	-5(5)	26(5)	-28(5)
C(2)	160(6)	188(7)	140(6)	12(5)	29(5)	5(5)
C(3)	165(7)	220(8)	302(8)	-28(7)	7(6)	13(6)
C(4)	189(7)	140(6)	158(6)	10(5)	43(5)	31(5)
C(5)	176(6)	159(6)	118(6)	-7(5)	29(5)	-31(5)
C(6)	223(8)	142(7)	237(7)	-9(6)	13(6)	-2(6)
C(7)	145(6)	157(6)	123(6)	7(5)	31(5)	4(5)
C(8)	162(7)	168(7)	209(7)	-33(6)	8(5)	0(5)
C(9)	142(6)	161(6)	161(6)	30(5)	48(5)	-18(5)
C(10)	151(6)	128(6)	143(6)	26(5)	21(5)	7(5)
C(11)	173(6)	128(6)	153(6)	1(5)	32(5)	14(5)
C(12)	202(7)	167(7)	216(7)	13(6)	73(6)	27(6)
C(13)	185(7)	159(7)	171(7)	4(5)	29(5)	13(5)
C(14)	209(7)	164(7)	163(6)	30(5)	19(5)	19(5)
C(15)	218(8)	287(9)	419(10)	-13(7)	64(7)	94(7)
C(16)	241(7)	145(7)	153(7)	-7(5)	26(5)	29(5)
C(17)	203(7)	172(7)	130(6)	-11(5)	-53(5)	9(5)
C(18)	158(7)	159(7)	206(7)	-1(5)	4(5)	-17(5)
C(19)	139(6)	125(6)	166(6)	0(5)	15(5)	-6(5)
C(20)	196(7)	182(7)	201(7)	18(5)	58(6)	0(6)
C(21)	162(6)	150(6)	114(6)	8(5)	37(5)	3(5)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for DCB11 (CCDC 201187).

	x	y	z	U_{iso}
H(1)	11324(13)	7813(10)	1530(6)	16(3)
H(3A)	13385(15)	6538(12)	1604(7)	35(4)
H(3B)	13289(17)	5469(13)	1039(9)	50(5)
H(3C)	13068(16)	5243(13)	1865(9)	48(5)
H(4)	10635(12)	4351(10)	941(6)	13(3)
H(6A)	7338(14)	2849(10)	357(7)	21(3)
H(6B)	8556(13)	3077(10)	1078(7)	18(3)
H(6C)	9048(14)	3171(10)	254(7)	19(3)
H(8A)	6202(14)	6529(11)	77(8)	29(4)
H(8B)	5944(15)	6062(11)	886(7)	33(4)
H(10)	6980(11)	7914(9)	1856(6)	9(3)
H(12A)	4310(12)	9821(10)	2363(6)	17(3)
H(12B)	3867(14)	8942(10)	1643(7)	22(3)
H(12C)	4927(13)	8491(11)	2410(7)	20(3)
H(13A)	6623(14)	10898(10)	997(6)	18(3)
H(13B)	5112(12)	10214(10)	719(6)	12(3)
H(15A)	1440(17)	12099(12)	1419(8)	38(4)
H(15B)	2664(16)	13060(13)	1277(8)	43(4)
H(15C)	2744(16)	12466(12)	2086(9)	44(4)
H(16A)	6816(13)	10804(10)	2604(6)	16(3)
H(16B)	7366(13)	9478(10)	2792(7)	18(3)
H(18A)	10357(14)	9583(10)	1663(6)	16(3)
H(18B)	9741(13)	8799(10)	2285(7)	22(3)
H(20A)	9509(15)	9153(10)	304(7)	26(3)
H(20B)	7759(14)	8917(10)	69(7)	22(3)
H(20C)	8331(13)	10112(11)	498(6)	21(3)