Supporting Material

X-ray diffraction study of pseudo single crystal prepared from crystal belonging to point group 2

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X-ray Structure Report

Experimental

Data Collection

A colorless block crystal of $C_{12}H_{22}O_{11}$ having approximate dimensions of 0.80 x 0.80 x 0.80 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 170 oscillations that were exposed for 100 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 7.7735(12) Å
b = 8.7169(13) Å β = 102.936(4)^o
c = 10.8765(17) Å
V = 718.29(19) Å^3

For Z = 2 and F.W. = 342.30, the calculated density is 1.583 g/cm^3. Based on the systematic absences of:

0k0: k ± 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$P2_1$ (#4)

The data were collected at a temperature of 20 ± 1^{o}C to a maximum 2θ value of 55.0^o. A total of 270 oscillation images were collected. A sweep of data was done using

ω scans from 80.0 to 260.0^{o} in 2.0^{o} step, at χ =45.0^{o} and ϕ = 0.0^{o}. The exposure rate was 50.0 [sec./^{o}]. A second sweep was performed using ω scans from 80.0 to 260.0^{o} in 2.0^{o} step, at χ =45.0^{o} and ϕ = 90.0^{o}. The exposure rate was 50.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^{o} in 2.0^{o} step, at χ =0.0^{o} and ϕ = 0.0^{o}. The exposure rate was 50.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 15524 reflections that were collected, 1747 were unique (R_{int} = 0.166); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-Kα radiation is 1.415 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.054 to 0.893. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 1730 observed reflections and 230 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \; ||Fo| - |Fc|| \; / \; \Sigma \; |Fo| = 0.0788$$

$$wR2 = [\Sigma \; (w (Fo^2 - Fc^2)^2) / \Sigma \; w(Fo^2)^2]^{1/2} = 0.1725$$

The standard deviation of an observation of unit weight[4] was 1.01. A Sheldrick weighting scheme was used. Plots of Σ w (|Fo| - |Fc|)^2 versus |Fo|, reflection order in data collection, sin θ /λ and various classes of indices showed no unusual trends. The

maximum and minimum peaks on the final difference Fourier map corresponded to 0.87 and -0.78 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δ f' and Δ f" were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$$\Sigma\ w(F_o^2-F_c^2)^2 \quad \text{where w = Least Squares weights.}$$

(4) Standard deviation of an observation of unit weight:

$$[\Sigma\ w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

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(9) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(10) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{12}H_{22}O_{11}$
Formula Weight	342.30
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.80 X 0.80 X 0.80 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	170 oscillations @ 100.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 7.7735(12) Å b = 8.7169(13) Å c = 10.8765(17) Å β = 102.936(4) ^{o} V = 718.29(19) Å^3
Space Group	$P2_1$ (#4)
Z value	2
D_{calc}	1.583 g/cm^3

F_{000}	364.00
μ (MoKα)	1.415 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	270 exposures
ω oscillation Range (χ =45.0, ϕ =0.0)	80.0 - 260.0^{o}
Exposure Rate	50.0 sec./^{o}
ω oscillation Range (χ =45.0, ϕ =90.0)	80.0 - 260.0^{o}
Exposure Rate	50.0 sec./^{o}
ω oscillation Range (χ =0.0, ϕ =0.0)	80.0 - 260.0^{o}
Exposure Rate	50.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 15524 Unique: 1730 (R_{int} = 0.166)
Corrections	Lorentz-polarization Absorption

(trans. factors: 0.054 - 0.893)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0004Fo^2+1.0000\sigma (Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	1730
No. Variables	230
Reflection/Parameter Ratio	7.52
Residuals: R1 ($I>2.00\sigma (I)$)	0.0788
Residuals: R (All reflections)	0.1896
Residuals: wR2 (All reflections)	0.1725
Goodness of Fit Indicator	1.009
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.87 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.78 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(1)	0.7461(9)	0.8285(9)	0.2283(6)	4.37(19)
O(2)	0.6192(8)	1.0306(8)	0.0320(6)	4.01(18)
O(3)	0.3914(9)	0.9212(7)	0.1713(6)	3.94(19)
O(4)	-0.0931(9)	1.1657(8)	-0.0170(6)	4.8(2)
O(5)	0.3142(9)	1.1739(9)	0.2121(6)	4.6(2)
O(6)	0.2828(9)	0.7237(9)	0.5800(7)	5.0(2)
O(7)	0.0428(9)	1.0289(9)	0.3237(6)	5.2(2)
O(8)	0.3707(9)	0.8661(7)	0.3784(6)	3.98(19)
O(9)	0.6975(10)	0.5227(8)	0.3042(7)	4.8(2)
O(10)	0.2053(8)	0.9446(7)	-0.0723(6)	4.5(2)
O(11)	0.3558(11)	0.4497(8)	0.3488(7)	5.7(2)
C(11)	0.1274(14)	1.2027(11)	0.1751(10)	3.9(2)
C(13)	0.6333(15)	0.7885(12)	0.3167(10)	4.0(3)
C(14)	0.2145(14)	1.0718(11)	0.0073(9)	3.5(2)
C(15)	0.5418(13)	1.1406(13)	0.1062(10)	4.7(3)
C(16)	0.3670(14)	1.0722(12)	0.1239(10)	3.6(2)
C(17)	0.5652(15)	0.6319(14)	0.2830(11)	4.4(3)
C(18)	0.0410(14)	1.1855(10)	0.2854(10)	4.1(3)
C(19)	0.0584(14)	1.1027(13)	0.0668(10)	4.2(2)
C(20)	0.2957(16)	0.7145(12)	0.3612(10)	4.2(3)
C(21)	0.4840(13)	0.9119(14)	0.2986(10)	4.3(3)
C(22)	0.1835(14)	0.6960(13)	0.4547(10)	4.6(3)
C(23)	0.4477(16)	0.5989(12)	0.3748(10)	4.5(3)

$$B_{eq} = 8/3\ \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.1103	1.3090	0.1457	4.52
H(2)	0.7028	0.7914	0.4038	4.85
H(3)	0.2279	1.1634	-0.0419	4.40
H(4)	0.6220	1.1586	0.1870	5.66
H(5)	0.5209	1.2377	0.0619	5.66
H(6)	0.4980	0.6262	0.1954	5.41
H(7)	-0.0790	1.2241	0.2638	4.91
H(8)	0.1066	1.2460	0.3552	4.92
H(9)	0.0223	1.0053	0.0983	4.91
H(10)	0.2206	0.7063	0.2762	5.09
H(11)	0.5345	1.0126	0.3257	5.22
H(12)	0.1349	0.5931	0.4489	5.66
H(13)	0.0860	0.7681	0.4355	5.66
H(14)	0.5188	0.6007	0.4615	5.58
H(15)	0.0149	1.0238	0.3920	6.49
H(16)	0.1042	0.9356	-0.1144	5.30
H(17)	0.8445	0.8528	0.2687	5.30
H(18)	0.7251	1.0230	0.0634	4.86
H(19)	-0.0580	1.2153	-0.0705	5.85
H(20)	0.3827	0.6894	0.5868	6.06
H(21)	0.7744	0.5484	0.3654	5.81
H(22)	0.4004	0.3987	0.3007	7.15

$$B_{eq} = 8/3\ \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	0.036(4)	0.076(5)	0.055(5)	0.009(4)	0.014(4)	0.008(4)
O(2)	0.033(4)	0.064(4)	0.056(4)	-0.008(4)	0.013(3)	-0.003(4)
O(3)	0.046(5)	0.047(4)	0.054(5)	0.011(3)	0.005(4)	0.001(4)
O(4)	0.076(5)	0.057(4)	0.053(5)	0.014(4)	0.019(4)	0.005(4)
O(5)	0.038(4)	0.068(5)	0.063(5)	-0.000(4)	-0.001(3)	-0.016(4)
O(6)	0.046(5)	0.080(5)	0.065(5)	0.003(4)	0.018(4)	0.016(5)
O(7)	0.065(5)	0.087(5)	0.054(5)	-0.006(5)	0.032(4)	0.011(5)
O(8)	0.053(5)	0.044(4)	0.063(5)	0.001(3)	0.031(4)	0.004(4)
O(9)	0.079(6)	0.043(4)	0.062(5)	0.007(4)	0.018(4)	-0.004(4)
O(10)	0.035(4)	0.073(5)	0.059(5)	-0.013(4)	0.005(3)	-0.021(4)
O(11)	0.108(7)	0.038(4)	0.080(6)	-0.024(5)	0.042(5)	-0.014(4)
C(11)	0.043(7)	0.041(7)	0.060(7)	0.001(5)	0.002(6)	0.009(6)
C(13)	0.034(7)	0.079(9)	0.040(7)	-0.001(6)	0.009(6)	-0.005(6)
C(14)	0.050(7)	0.041(6)	0.048(7)	0.007(5)	0.027(6)	0.007(5)
C(15)	0.041(7)	0.069(8)	0.069(8)	0.001(7)	0.015(6)	0.002(7)
C(16)	0.039(7)	0.050(7)	0.051(7)	-0.013(5)	0.020(6)	0.008(6)
C(17)	0.053(8)	0.078(8)	0.040(7)	0.010(7)	0.019(6)	0.009(6)
C(18)	0.057(8)	0.036(6)	0.063(8)	-0.001(6)	0.015(6)	0.006(6)
C(19)	0.045(7)	0.067(7)	0.044(6)	-0.001(6)	0.003(5)	0.010(6)
C(20)	0.062(8)	0.059(7)	0.040(7)	-0.005(7)	0.011(6)	0.018(6)
C(21)	0.043(7)	0.080(8)	0.042(7)	0.003(6)	0.011(6)	-0.016(6)
C(22)	0.064(8)	0.052(7)	0.063(8)	0.014(6)	0.021(7)	0.007(6)
C(23)	0.062(8)	0.063(7)	0.052(8)	0.021(7)	0.025(6)	0.004(6)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(1)	C(13)	1.481(15)	O(2)	C(15)	1.466(14)
O(3)	C(16)	1.411(12)	O(3)	C(21)	1.412(12)
O(4)	C(19)	1.428(12)	O(5)	C(11)	1.439(12)
O(5)	C(16)	1.432(14)	O(6)	C(22)	1.428(12)
O(7)	C(18)	1.426(12)	O(8)	C(20)	1.440(12)
O(8)	C(21)	1.424(14)	O(9)	C(17)	1.382(14)
O(10)	C(14)	1.398(12)	O(11)	C(23)	1.481(13)
C(11)	C(18)	1.508(17)	C(11)	C(19)	1.467(15)
C(13)	C(17)	1.480(16)	C(13)	C(21)	1.562(16)
C(14)	C(16)	1.530(13)	C(14)	C(19)	1.522(17)
C(15)	C(16)	1.535(16)	C(17)	C(23)	1.524(18)
C(20)	C(22)	1.489(18)	C(20)	C(23)	1.535(16)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
O(1)	H(17)	0.820	O(2)	H(18)	0.820
O(4)	H(19)	0.820	O(6)	H(20)	0.820
O(7)	H(15)	0.820	O(9)	H(21)	0.820
O(10)	H(16)	0.820	O(11)	H(22)	0.820
C(11)	H(1)	0.980	C(13)	H(2)	0.980
C(14)	H(3)	0.980	C(15)	H(4)	0.970
C(15)	H(5)	0.970	C(17)	H(6)	0.980
C(18)	H(7)	0.970	C(18)	H(8)	0.970
C(19)	H(9)	0.980	C(20)	H(10)	0.980
C(21)	H(11)	0.980	C(22)	H(12)	0.970
C(22)	H(13)	0.970	C(23)	H(14)	0.980

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(16)	O(3)	C(21)	114.2(8)	C(11)	O(5)	C(16)	110.1(7)
C(20)	O(8)	C(21)	117.7(8)	O(5)	C(11)	C(18)	111.2(8)
O(5)	C(11)	C(19)	106.6(9)	C(18)	C(11)	C(19)	115.6(9)
O(1)	C(13)	C(17)	106.7(9)	O(1)	C(13)	C(21)	106.7(8)
C(17)	C(13)	C(21)	112.9(9)	O(10)	C(14)	C(16)	116.0(8)
O(10)	C(14)	C(19)	117.9(8)	C(16)	C(14)	C(19)	101.0(8)
O(2)	C(15)	C(16)	107.4(8)	O(3)	C(16)	O(5)	111.6(8)
O(3)	C(16)	C(14)	108.5(7)	O(3)	C(16)	C(15)	110.9(8)
O(5)	C(16)	C(14)	105.8(8)	O(5)	C(16)	C(15)	103.8(8)
C(14)	C(16)	C(15)	116.1(9)	O(9)	C(17)	C(13)	112.5(9)
O(9)	C(17)	C(23)	107.1(9)	C(13)	C(17)	C(23)	104.2(10)
O(7)	C(18)	C(11)	110.5(8)	O(4)	C(19)	C(11)	112.7(9)
O(4)	C(19)	C(14)	114.8(9)	C(11)	C(19)	C(14)	105.3(8)
O(8)	C(20)	C(22)	107.0(9)	O(8)	C(20)	C(23)	108.0(9)
C(22)	C(20)	C(23)	114.8(9)	O(3)	C(21)	O(8)	111.4(7)
O(3)	C(21)	C(13)	111.4(9)	O(8)	C(21)	C(13)	105.9(9)
O(6)	C(22)	C(20)	111.0(9)	O(11)	C(23)	C(17)	112.0(9)
O(11)	C(23)	C(20)	103.3(8)	C(17)	C(23)	C(20)	112.0(9)

Table 7. Bond angles involving hydrogens (^{o})

atom	atom	atom	angle	atom	atom	atom	angle
C(13)	O(1)	H(17)	109.2	C(15)	O(2)	H(18)	108.7
C(19)	O(4)	H(19)	107.3	C(22)	O(6)	H(20)	108.9
C(18)	O(7)	H(15)	109.2	C(17)	O(9)	H(21)	109.2
C(14)	O(10)	H(16)	109.3	C(23)	O(11)	H(22)	110.1
O(5)	C(11)	H(1)	108.1	C(18)	C(11)	H(1)	107.6
C(19)	C(11)	H(1)	107.5	O(1)	C(13)	H(2)	110.1
C(17)	C(13)	H(2)	110.6	C(21)	C(13)	H(2)	109.7
O(10)	C(14)	H(3)	107.7	C(16)	C(14)	H(3)	107.1
C(19)	C(14)	H(3)	106.3	O(2)	C(15)	H(4)	110.2
O(2)	C(15)	H(5)	110.0	C(16)	C(15)	H(4)	110.8
C(16)	C(15)	H(5)	110.3	H(4)	C(15)	H(5)	108.1
O(9)	C(17)	H(6)	110.0	C(13)	C(17)	H(6)	111.8
C(23)	C(17)	H(6)	111.1	O(7)	C(18)	H(7)	110.4
O(7)	C(18)	H(8)	108.9	C(11)	C(18)	H(7)	110.4
C(11)	C(18)	H(8)	108.6	H(7)	C(18)	H(8)	108.0
O(4)	C(19)	H(9)	107.2	C(11)	C(19)	H(9)	108.4
C(14)	C(19)	H(9)	108.2	O(8)	C(20)	H(10)	109.1
C(22)	C(20)	H(10)	108.7	C(23)	C(20)	H(10)	109.0
O(3)	C(21)	H(11)	109.0	O(8)	C(21)	H(11)	109.1
C(13)	C(21)	H(11)	110.0	O(6)	C(22)	H(12)	109.7
O(6)	C(22)	H(13)	108.9	C(20)	C(22)	H(12)	109.5
C(20)	C(22)	H(13)	109.4	H(12)	C(22)	H(13)	108.1
O(11)	C(23)	H(14)	110.0	C(17)	C(23)	H(14)	109.6
C(20)	C(23)	H(14)	109.8				

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(16)	O(3)	C(21)	O(8)	109.5(10)	C(16)	O(3)	C(21)	C(13)	-132.6(9)
C(21)	O(3)	C(16)	O(5)	-43.8(11)	C(21)	O(3)	C(16)	C(14)	-160.0(9)
C(21)	O(3)	C(16)	C(15)	71.4(11)	C(11)	O(5)	C(16)	O(3)	-102.1(9)
C(11)	O(5)	C(16)	C(14)	15.6(11)	C(11)	O(5)	C(16)	C(15)	138.3(8)
C(16)	O(5)	C(11)	C(18)	132.6(8)	C(16)	O(5)	C(11)	C(19)	5.8(11)
C(20)	O(8)	C(21)	O(3)	65.1(11)	C(20)	O(8)	C(21)	C(13)	-56.1(10)
C(21)	O(8)	C(20)	C(22)	-179.8(6)	C(21)	O(8)	C(20)	C(23)	56.1(11)
O(5)	C(11)	C(18)	O(7)	-68.6(10)	O(5)	C(11)	C(19)	O(4)	-150.8(9)
O(5)	C(11)	C(19)	C(14)	-25.0(11)	C(18)	C(11)	C(19)	O(4)	85.1(11)
C(18)	C(11)	C(19)	C(14)	-149.1(8)	C(19)	C(11)	C(18)	O(7)	53.1(11)
O(1)	C(13)	C(17)	O(9)	65.6(11)	O(1)	C(13)	C(17)	C(23)	-178.8(7)
O(1)	C(13)	C(21)	O(3)	55.2(10)	O(1)	C(13)	C(21)	O(8)	176.4(7)
C(17)	C(13)	C(21)	O(3)	-61.7(13)	C(17)	C(13)	C(21)	O(8)	59.5(11)
C(21)	C(13)	C(17)	O(9)	-177.5(9)	C(21)	C(13)	C(17)	C(23)	-61.9(11)
O(10)	C(14)	C(16)	O(3)	-38.4(13)	O(10)	C(14)	C(16)	O(5)	-158.3(8)
O(10)	C(14)	C(16)	C(15)	87.3(11)	O(10)	C(14)	C(19)	O(4)	-75.0(11)
O(10)	C(14)	C(19)	C(11)	160.5(8)	C(16)	C(14)	C(19)	O(4)	157.6(8)
C(16)	C(14)	C(19)	C(11)	33.0(10)	C(19)	C(14)	C(16)	O(3)	90.3(9)
C(19)	C(14)	C(16)	O(5)	-29.5(10)	C(19)	C(14)	C(16)	C(15)	-144.0(9)
O(2)	C(15)	C(16)	O(3)	51.5(10)	O(2)	C(15)	C(16)	O(5)	171.5(7)
O(2)	C(15)	C(16)	C(14)	-72.9(10)	O(9)	C(17)	C(23)	O(11)	-65.1(10)
O(9)	C(17)	C(23)	C(20)	179.4(8)	C(13)	C(17)	C(23)	O(11)	175.6(8)
C(13)	C(17)	C(23)	C(20)	60.1(10)	O(8)	C(20)	C(22)	O(6)	-55.9(10)
O(8)	C(20)	C(23)	O(11)	-177.0(8)	O(8)	C(20)	C(23)	C(17)	-56.3(11)
C(22)	C(20)	C(23)	O(11)	63.6(11)	C(22)	C(20)	C(23)	C(17)	-175.6(9)
C(23)	C(20)	C(22)	O(6)	64.0(11)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(1)	O(7)[1)]	2.894(10)	O(1)	C(14)[2)]	3.467(13)
O(2)	O(4)[1)]	2.684(10)	O(2)	C(17)[3)]	3.518(13)
O(2)	C(19)[1)]	3.408(13)	O(3)	O(4)[4)]	3.378(9)
O(4)	O(2)[5)]	2.684(10)	O(4)	O(3)[6)]	3.378(9)
O(4)	O(10)[6)]	2.827(10)	O(4)	C(15)[5)]	3.405(14)
O(5)	O(6)[7)]	3.456(9)	O(5)	O(11)[8)]	2.807(10)
O(6)	O(5)[9)]	3.456(9)	O(6)	O(7)[10)]	3.402(11)
O(6)	O(9)[7)]	2.884(10)	O(6)	O(11)[7)]	3.377(10)
O(6)	C(15)[9)]	3.457(13)	O(6)	C(18)[10)]	3.200(15)
O(6)	C(21)[9)]	3.369(13)	O(7)	O(1)[5)]	2.894(10)
O(7)	O(6)[11)]	3.402(11)	O(7)	C(22)[11)]	3.595(14)
O(8)	O(11)[7)]	3.322(10)	O(8)	C(23)[7)]	3.409(12)
O(9)	O(6)[9)]	2.884(10)	O(9)	O(10)[2)]	2.873(11)
O(10)	O(4)[4)]	2.827(10)	O(10)	O(9)[3)]	2.873(11)
O(10)	C(11)[4)]	3.330(12)	O(10)	C(15)[2)]	3.369(13)
O(10)	C(17)[3)]	3.596(15)	O(10)	C(18)[4)]	3.486(11)
O(11)	O(5)[12)]	2.807(10)	O(11)	O(6)[9)]	3.377(10)
O(11)	O(8)[9)]	3.322(10)	O(11)	C(11)[12)]	3.139(12)
O(11)	C(18)[12)]	3.318(12)	C(11)	O(10)[6)]	3.330(12)
C(11)	O(11)[8)]	3.139(12)	C(14)	O(1)[3)]	3.467(13)
C(15)	O(4)[1)]	3.405(14)	C(15)	O(6)[7)]	3.457(13)
C(15)	O(10)[3)]	3.369(13)	C(17)	O(2)[2)]	3.518(13)
C(17)	O(10)[2)]	3.596(15)	C(18)	O(6)[11)]	3.200(15)
C(18)	O(10)[6)]	3.486(11)	C(18)	O(11)[8)]	3.318(12)
C(19)	O(2)[5)]	3.408(13)	C(21)	O(6)[7)]	3.369(13)
C(22)	O(7)[10)]	3.595(14)	C(23)	O(8)[9)]	3.409(12)

Symmetry Operators:

(1) X+1,Y,Z
(2) -X+1,Y+1/2-1,-Z
(3) -X+1,Y+1/2,-Z
(4) -X,Y+1/2-1,-Z
(5) X-1,Y,Z
(6) -X,Y+1/2,-Z
(7) -X+1,Y+1/2,-Z+1
(8) X,Y+1,Z
(9) -X+1,Y+1/2-1,-Z+1
(10) -X,Y+1/2-1,-Z+1

(11) -X,Y+1/2,-Z+1

(12) X,Y-1,Z

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(3)[1)]	2.531	O(1)	H(5)[1)]	3.459
O(1)	H(9)[2)]	3.216	O(1)	H(13)[2)]	3.110
O(1)	H(15)[2)]	2.961	O(1)	H(19)[1)]	3.414
O(2)	H(3)[1)]	3.408	O(2)	H(5)[1)]	2.872
O(2)	H(6)[3)]	2.576	O(2)	H(7)[2)]	3.472
O(2)	H(9)[2)]	3.062	O(2)	H(19)[2)]	3.374
O(3)	H(5)[1)]	3.198	O(3)	H(19)[4)]	3.142
O(4)	H(1)[4)]	3.400	O(4)	H(4)[5)]	3.469
O(4)	H(5)[5)]	3.359	O(4)	H(6)[6)]	3.325
O(4)	H(9)[6)]	3.173	O(4)	H(10)[6)]	2.794
O(4)	H(16)[6)]	2.765	O(4)	H(18)[5)]	2.204
O(5)	H(14)[7)]	3.554	O(5)	H(20)[7)]	2.836
O(5)	H(22)[8)]	2.220	O(6)	H(4)[9)]	2.540
O(6)	H(7)[10)]	2.570	O(6)	H(8)[10)]	3.263
O(6)	H(11)[9)]	2.409	O(6)	H(15)[10)]	2.966
O(6)	H(21)[7)]	2.947	O(6)	H(22)[7)]	2.941
O(7)	H(4)[5)]	3.466	O(7)	H(12)[11)]	3.142
O(7)	H(17)[5)]	2.163	O(7)	H(18)[5)]	3.315
O(7)	H(21)[7)]	3.366	O(8)	H(14)[7)]	2.699
O(8)	H(20)[7)]	3.382	O(8)	H(22)[7)]	3.557
O(9)	H(3)[1)]	3.274	O(9)	H(4)[12)]	3.422
O(9)	H(7)[13)]	3.215	O(9)	H(12)[2)]	3.467
O(9)	H(15)[9)]	3.551	O(9)	H(16)[1)]	2.939
O(9)	H(20)[9)]	3.250	O(10)	H(1)[4)]	2.679
O(10)	H(4)[1)]	3.213	O(10)	H(5)[1)]	2.773
O(10)	H(6)[3)]	3.317	O(10)	H(7)[4)]	2.842
O(10)	H(19)[4)]	2.918	O(10)	H(21)[3)]	3.351
O(11)	H(1)[12)]	2.853	O(11)	H(2)[9)]	3.146
O(11)	H(8)[12)]	2.640	O(11)	H(11)[9)]	3.494
O(11)	H(20)[9)]	3.022	C(11)	H(16)[6)]	2.696
C(11)	H(18)[5)]	3.465	C(11)	H(22)[8)]	2.827
C(13)	H(3)[1)]	3.568	C(13)	H(13)[2)]	3.471
C(13)	H(15)[2)]	3.548	C(14)	H(1)[4)]	3.537
C(14)	H(6)[3)]	3.505	C(14)	H(19)[4)]	3.462
C(15)	H(6)[3)]	3.227	C(15)	H(7)[2)]	3.145

C(15)	H(20)[7)]	3.286	C(15)	H(22)[8)]	3.434
C(16)	H(20)[7)]	3.463	C(16)	H(22)[8)]	3.412

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(17)	H(3)[1)]	3.380	C(18)	H(4)[5)]	3.202
C(18)	H(12)[11)]	3.555	C(18)	H(13)[11)]	3.471
C(18)	H(16)[6)]	2.924	C(18)	H(17)[5)]	3.264
C(18)	H(18)[5)]	3.348	C(18)	H(22)[8)]	3.329
C(19)	H(1)[4)]	3.501	C(19)	H(16)[6)]	3.253
C(19)	H(18)[5)]	2.675	C(19)	H(19)[4)]	3.378
C(20)	H(19)[4)]	3.286	C(21)	H(14)[7)]	3.089
C(21)	H(20)[7)]	2.811	C(22)	H(7)[10)]	3.348
C(22)	H(8)[10)]	3.412	C(22)	H(11)[9)]	3.275
C(22)	H(15)[10)]	2.927	C(22)	H(17)[5)]	3.242
C(22)	H(21)[5)]	3.366	C(23)	H(11)[9)]	3.315
H(1)	O(4)[6)]	3.400	H(1)	O(10)[6)]	2.679
H(1)	O(11)[8)]	2.853	H(1)	C(14)[6)]	3.537
H(1)	C(19)[6)]	3.501	H(1)	H(9)[6)]	3.131
H(1)	H(16)[6)]	1.965	H(1)	H(18)[3)]	3.405
H(1)	H(22)[8)]	2.615	H(2)	O(11)[7)]	3.146
H(2)	H(8)[9)]	2.735	H(2)	H(12)[7)]	3.189
H(2)	H(13)[2)]	2.928	H(2)	H(15)[2)]	3.185
H(2)	H(20)[7)]	3.539	H(3)	O(1)[3)]	2.531
H(3)	O(2)[3)]	3.408	H(3)	O(9)[3)]	3.274
H(3)	C(13)[3)]	3.568	H(3)	C(17)[3)]	3.380
H(3)	H(6)[3)]	3.004	H(3)	H(9)[6)]	3.537
H(3)	H(17)[3)]	2.916	H(3)	H(18)[3)]	3.171
H(4)	O(4)[2)]	3.469	H(4)	O(6)[7)]	2.540
H(4)	O(7)[2)]	3.466	H(4)	O(9)[8)]	3.422
H(4)	O(10)[3)]	3.213	H(4)	C(18)[2)]	3.202
H(4)	H(7)[2)]	2.357	H(4)	H(15)[2)]	3.554
H(4)	H(16)[3)]	3.427	H(4)	H(20)[7)]	2.483
H(4)	H(22)[8)]	3.135	H(5)	O(1)[3)]	3.459
H(5)	O(2)[3)]	2.872	H(5)	O(3)[3)]	3.198
H(5)	O(4)[2)]	3.359	H(5)	O(10)[3)]	2.773
H(5)	H(6)[3)]	2.935	H(5)	H(7)[2)]	3.384
H(5)	H(16)[3)]	3.324	H(5)	H(18)[3)]	3.245
H(5)	H(22)[8)]	3.269	H(6)	O(2)[1)]	2.576

H(6)	O(4)[4)]	3.325	H(6)	O(10)[1)]	3.317
H(6)	C(14)[1)]	3.505	H(6)	C(15)[1)]	3.227
H(6)	H(3)[1)]	3.004	H(6)	H(5)[1)]	2.935

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(6)	H(18)[1)]	3.091	H(6)	H(19)[4)]	3.474
H(7)	O(2)[5)]	3.472	H(7)	O(6)[11)]	2.570
H(7)	O(9)[14)]	3.215	H(7)	O(10)[6)]	2.842
H(7)	C(15)[5)]	3.145	H(7)	C(22)[11)]	3.348
H(7)	H(4)[5)]	2.357	H(7)	H(5)[5)]	3.384
H(7)	H(12)[11)]	3.445	H(7)	H(13)[11)]	3.305
H(7)	H(16)[6)]	2.436	H(7)	H(17)[5)]	3.293
H(7)	H(18)[5)]	2.942	H(7)	H(20)[11)]	3.166
H(7)	H(21)[14)]	3.328	H(8)	O(6)[11)]	3.263
H(8)	O(11)[8)]	2.640	H(8)	C(22)[11)]	3.412
H(8)	H(2)[7)]	2.735	H(8)	H(12)[8)]	3.184
H(8)	H(12)[11)]	3.411	H(8)	H(13)[11)]	2.997
H(8)	H(14)[7)]	3.386	H(8)	H(16)[6)]	3.218
H(8)	H(21)[7)]	3.435	H(8)	H(22)[8)]	2.819
H(9)	O(1)[5)]	3.216	H(9)	O(2)[5)]	3.062
H(9)	O(4)[4)]	3.173	H(9)	H(1)[4)]	3.131
H(9)	H(3)[4)]	3.537	H(9)	H(17)[5)]	2.874
H(9)	H(18)[5)]	2.261	H(9)	H(19)[4)]	2.569
H(10)	O(4)[4)]	2.794	H(10)	H(17)[5)]	3.174
H(10)	H(19)[4)]	2.311	H(11)	O(6)[7)]	2.409
H(11)	O(11)[7)]	3.494	H(11)	C(22)[7)]	3.275
H(11)	C(23)[7)]	3.315	H(11)	H(12)[7)]	3.207
H(11)	H(14)[7)]	2.559	H(11)	H(20)[7)]	1.850
H(11)	H(22)[8)]	3.516	H(12)	O(7)[10)]	3.142
H(12)	O(9)[5)]	3.467	H(12)	C(18)[10)]	3.555
H(12)	H(2)[9)]	3.189	H(12)	H(7)[10)]	3.445
H(12)	H(8)[12)]	3.184	H(12)	H(8)[10)]	3.411
H(12)	H(11)[9)]	3.207	H(12)	H(15)[10)]	2.371
H(12)	H(17)[5)]	3.475	H(12)	H(21)[5)]	2.773
H(13)	O(1)[5)]	3.110	H(13)	C(13)[5)]	3.471
H(13)	C(18)[10)]	3.471	H(13)	H(2)[5)]	2.928
H(13)	H(7)[10)]	3.305	H(13)	H(8)[10)]	2.997
H(13)	H(15)[10)]	3.054	H(13)	H(17)[5)]	2.416
H(13)	H(21)[5)]	3.047	H(13)	H(21)[7)]	3.284

H(14)	O(5)[9)]	3.554	H(14)	O(8)[9)]	2.699
H(14)	C(21)[9)]	3.089	H(14)	H(8)[9)]	3.386
H(14)	H(11)[9)]	2.559	H(15)	O(1)[5)]	2.961

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(15)	O(6)[11)]	2.966	H(15)	O(9)[7)]	3.551
H(15)	C(13)[5)]	3.548	H(15)	C(22)[11)]	2.927
H(15)	H(2)[5)]	3.185	H(15)	H(4)[5)]	3.554
H(15)	H(12)[11)]	2.371	H(15)	H(13)[11)]	3.054
H(15)	H(17)[5)]	2.231	H(15)	H(20)[11)]	3.465
H(15)	H(21)[7)]	2.785	H(16)	O(4)[4)]	2.765
H(16)	O(9)[3)]	2.939	H(16)	C(11)[4)]	2.696
H(16)	C(18)[4)]	2.924	H(16)	C(19)[4)]	3.253
H(16)	H(1)[4)]	1.965	H(16)	H(4)[1)]	3.427
H(16)	H(5)[1)]	3.324	H(16)	H(7)[4)]	2.436
H(16)	H(8)[4)]	3.218	H(16)	H(19)[4)]	2.862
H(16)	H(21)[3)]	3.234	H(17)	O(7)[2)]	2.163
H(17)	C(18)[2)]	3.264	H(17)	C(22)[2)]	3.242
H(17)	H(3)[1)]	2.916	H(17)	H(7)[2)]	3.293
H(17)	H(9)[2)]	2.874	H(17)	H(10)[2)]	3.174
H(17)	H(12)[2)]	3.475	H(17)	H(13)[2)]	2.416
H(17)	H(15)[2)]	2.231	H(17)	H(19)[1)]	3.231
H(18)	O(4)[2)]	2.204	H(18)	O(7)[2)]	3.315
H(18)	C(11)[2)]	3.465	H(18)	C(18)[2)]	3.348
H(18)	C(19)[2)]	2.675	H(18)	H(1)[1)]	3.405
H(18)	H(3)[1)]	3.171	H(18)	H(5)[1)]	3.245
H(18)	H(6)[3)]	3.091	H(18)	H(7)[2)]	2.942
H(18)	H(9)[2)]	2.261	H(18)	H(19)[2)]	2.979
H(19)	O(1)[3)]	3.414	H(19)	O(2)[5)]	3.374
H(19)	O(3)[6)]	3.142	H(19)	O(10)[6)]	2.918
H(19)	C(14)[6)]	3.462	H(19)	C(19)[6)]	3.378
H(19)	C(20)[6)]	3.286	H(19)	H(6)[6)]	3.474
H(19)	H(9)[6)]	2.569	H(19)	H(10)[6)]	2.311
H(19)	H(16)[6)]	2.862	H(19)	H(17)[3)]	3.231
H(19)	H(18)[5)]	2.979	H(20)	O(5)[9)]	2.836
H(20)	O(8)[9)]	3.382	H(20)	O(9)[7)]	3.250
H(20)	O(11)[7)]	3.022	H(20)	C(15)[9)]	3.286
H(20)	C(16)[9)]	3.463	H(20)	C(21)[9)]	2.811
H(20)	H(2)[9)]	3.539	H(20)	H(4)[9)]	2.483

H(20)	H(7)[10)]	3.166	H(20)	H(11)[9)]	1.850
H(20)	H(15)[10)]	3.465	H(20)	H(21)[7)]	3.441
H(20)	H(22)[7)]	2.597	H(21)	O(6)[9)]	2.947

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(21)	O(7)[9)]	3.366	H(21)	O(10)[1)]	3.351
H(21)	C(22)[2)]	3.366	H(21)	H(7)[13)]	3.328
H(21)	H(8)[9)]	3.435	H(21)	H(12)[2)]	2.773
H(21)	H(13)[2)]	3.047	H(21)	H(13)[9)]	3.284
H(21)	H(15)[9)]	2.785	H(21)	H(16)[1)]	3.234
H(21)	H(20)[9)]	3.441	H(22)	O(5)[12)]	2.220
H(22)	O(6)[9)]	2.941	H(22)	O(8)[9)]	3.557
H(22)	C(11)[12)]	2.827	H(22)	C(15)[12)]	3.434
H(22)	C(16)[12)]	3.412	H(22)	C(18)[12)]	3.329
H(22)	H(1)[12)]	2.615	H(22)	H(4)[12)]	3.135
H(22)	H(5)[12)]	3.269	H(22)	H(8)[12)]	2.819
H(22)	H(11)[12)]	3.516	H(22)	H(20)[9)]	2.597

Symmetry Operators:

(1) -X+1,Y+1/2-1,-Z
(2) X+1,Y,Z
(3) -X+1,Y+1/2,-Z
(4) -X,Y+1/2-1,-Z
(5) X-1,Y,Z
(6) -X,Y+1/2,-Z
(7) -X+1,Y+1/2,-Z+1
(8) X,Y+1,Z
(9) -X+1,Y+1/2-1,-Z+1
(10) -X,Y+1/2-1,-Z+1
(11) -X,Y+1/2,-Z+1
(12) X,Y-1,Z
(13) X+1,Y-1,Z
(14) X-1,Y+1,Z