X-ray Structure Report

for

yamamoto_anada2

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Experimental

Data Collection

A colorless block crystal of $C_{21}H_{17}ClO$ having approximate dimensions of 0.259 x 0.230 x 0.093 mm was mounted on a glass fiber. All measurements were made on a Rigaku R-AXIS RAPID diffractometer using graphite monochromated Mo-Ka radiation.

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 = 5.6215(5) Å
b = 17.3962(16) Å b = 96.948(2)°
c = 8.3197(6) Å
V = 807.63(12) Å^3

For Z = 2 and F.W. = 320.82, the calculated density is 1.319 g/cm^3. Based on the reflection conditions 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 23 $\pm$ 1°C to a maximum 2q value of 54.9°. A total of 44 oscillation images were collected. A sweep of data was done using w scans from 130.0 to 190.0° in 5.00° step, at c=45.0° and f = 0.0°. The exposure rate was 120.0 [sec./°]. A second sweep was performed using w scans from 0.0 to 160.0° in 5.00° step, at c=45.0° and f = 180.0°. The exposure rate was 120.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 7808 reflections were collected, where 3650 were unique (R_{int} = 0.0289); equivalent reflections were merged.

The linear absorption coefficient, m, for Mo-Ka radiation is 2.380 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors

ranging from 0.783 to 0.978. 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. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[2] on F^2 was based on 3650 observed reflections and 208 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = S \; ||Fo| - |Fc|| \; / \; S \; |Fo| = 0.0328$$

$$wR2 = [S (w (Fo^2 - Fc^2)^2)/ S \; w(Fo^2)^2]^{1/2} = 0.0793$$

The goodness of fit[3] was 1.06. Unit weights were used. Plots of S w (|Fo| - |Fc|)^2 versus |Fo|, reflection order in data collection, sin q/l and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.16 and -0.17 $e^-/Å^3$, respectively. The final Flack parameter [4] was 0.03(3), indicating that the present absolute structure is correct. [5]

Neutral atom scattering factors were taken from International Tables for Crystallography (IT), Vol. C, Table 6.1.1.4 [6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Df' and Df'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL2013[11].

References

(1) SHELXS2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

(2) Least Squares function minimized: (SHELXL2013)

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

(3) Goodness of fit is defined as:

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

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

(4) Parsons, S. and Flack, H. (2004), Acta Cryst. A60, s61.

(5) Flack, H.D. and Bernardinelli (2000), J. Appl. Cryst. 33, 114-1148.

(6) International Tables for Crystallography, Vol.C (1992). Ed. A.J.C. Wilson, Kluwer Academic Publishers, Dordrecht, Netherlands, Table 6.1.1.4, pp. 572.

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

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 4.1: Crystal Structure Analysis Package, Rigaku Corporation (2000-2014). Tokyo 196-8666, Japan.

(11) SHELXL2013: Sheldrick, G. M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{21}H_{17}ClO$
Formula Weight	320.82
Crystal Color, Habit	colorless, block
Crystal Dimensions	0.259 X 0.230 X 0.093 mm
Crystal System	monoclinic
Lattice Type	Primitive
Lattice Parameters	a = 5.6215(5) Å

	b = 17.3962(16) Å c = 8.3197(6) Å b = 96.948(2) ^{o} V = 807.63(12) Å^{3}
Space Group	$P2_1$ (#4)
Z value	2
D_{calc}	1.319 g/cm^{3}
F_{000}	336.00
m(MoKa)	2.380 cm^{-1}

B. Intensity Measurements

Diffractometer	R-AXIS RAPID
Radiation	MoKa (l = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 40mA
Temperature	23.0^{o}C
Detector Aperture	460.0 x 256.0 mm
Data Images	44 exposures
w oscillation Range (c=45.0, f=0.0)	130.0 - 190.0^{o}
Exposure Rate	120.0 sec./^{o}
w oscillation Range (c=45.0, f=180.0)	0.0 - 160.0^{o}
Exposure Rate	120.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2q_{max}$	54.9^{o}
No. of Reflections Measured	Total: 7808 Unique: 3650 (R_{int} = 0.0289)

	Parsons quotients (Flack x parameter): 1031
Corrections	Lorentz-polarization Absorption (trans. factors: 0.783 - 0.978)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS2013)
Refinement	Full-matrix least-squares on F^2
Function Minimized	S w $(Fo^2 - Fc^2)^2$
Least Squares Weights	w = 1/ [$s^2(Fo^2)$ + $(0.0352 \cdot P)^2$ + 0.0000 · P] where P = $(Max(Fo^2,0) + 2Fc^2)/3$
$2q_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	3650
No. Variables	208
Reflection/Parameter Ratio	17.55
Residuals: R1 (I>2.00s(I))	0.0328
Residuals: R (All reflections)	0.0615
Residuals: wR2 (All reflections)	0.0793
Goodness of Fit Indicator	1.062
Flack parameter (Parsons' quotients = 1031)	0.03(3)
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.16 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.17 $e^-/Å^3$

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

atom	x	y	z	B_{eq}
Cl24	0.55310(16)	0.40406(5)	0.64852(10)	5.18(2)
O11	0.9849(3)	0.76128(10)	0.7098(2)	3.04(3)
C2	0.5809(5)	0.92402(16)	0.9311(3)	3.47(5)
C3	0.6411(6)	1.00094(17)	0.9322(4)	4.04(6)
C4	0.7024(5)	0.87065(16)	0.8464(3)	2.86(5)
C5	0.8855(5)	0.89573(16)	0.7609(3)	2.91(5)
C6	0.9467(6)	0.97366(16)	0.7643(4)	3.68(6)
C7	0.8257(6)	1.02548(17)	0.8491(4)	4.11(6)
C8	0.6409(5)	0.78555(15)	0.8528(3)	2.69(5)
C9	0.7346(4)	0.74489(15)	0.7096(3)	2.83(5)
C10	1.0126(5)	0.83956(16)	0.6632(3)	3.27(5)
C12	0.7331(4)	0.75109(14)	1.0173(3)	2.48(4)
C13	0.5907(5)	0.69940(16)	1.0898(3)	3.13(5)
C14	0.6681(5)	0.66841(16)	1.2400(3)	3.58(6)
C15	0.8878(5)	0.68829(17)	1.3211(3)	3.34(5)
C16	1.0314(5)	0.73885(16)	1.2506(3)	3.32(5)
C17	0.9542(5)	0.77071(16)	1.0997(3)	3.13(5)
C18	0.6944(5)	0.65898(15)	0.7028(3)	2.75(5)
C19	0.4753(5)	0.63148(18)	0.6313(3)	3.42(5)
C20	0.4301(5)	0.55406(18)	0.6145(4)	3.73(6)
C21	0.6076(6)	0.50245(17)	0.6720(3)	3.40(5)
C22	0.8251(6)	0.52812(18)	0.7470(4)	3.87(6)
C23	0.8675(5)	0.60654(16)	0.7628(4)	3.46(5)

$B_{eq} = 8/3\ p^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos g + 2U_{13}(aa^*cc^*)\cos b + 2U_{23}(bb^*cc^*)\cos a)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H2	0.45743	0.90755	0.98771	4.161
H3	0.55797	1.03607	0.98857	4.844
H6	1.07059	0.99062	0.70858	4.415
H7	0.86804	1.07717	0.85054	4.926
H8	0.46585	0.78099	0.83857	3.227
H9	0.65008	0.76685	0.61016	3.390
H10A	0.95147	0.84525	0.54972	3.923
H10B	1.18201	0.85205	0.67512	3.923
H13	0.44182	0.68551	1.03673	3.762
H14	0.57082	0.63378	1.28683	4.297
H15	0.93830	0.66765	1.42267	4.003
H16	1.18102	0.75191	1.30388	3.983
H17	1.05182	0.80549	1.05368	3.758
H19	0.35582	0.66631	0.59373	4.104
H20	0.28259	0.53664	0.56525	4.476
H22	0.94279	0.49312	0.78678	4.643
H23	1.01365	0.62394	0.81432	4.148

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cl24	0.0863(6)	0.0410(4)	0.0687(5)	-0.0030(4)	0.0056(4)	-0.0111(4)
O11	0.0327(9)	0.0402(11)	0.0433(10)	0.0040(8)	0.0075(7)	0.0051(9)
C2	0.0470(17)	0.0420(17)	0.0438(15)	0.0024(13)	0.0093(13)	0.0035(13)
C3	0.066(2)	0.0389(17)	0.0493(17)	0.0081(16)	0.0094(15)	0.0016(14)
C4	0.0359(15)	0.0355(14)	0.0357(14)	0.0027(11)	-0.0011(11)	0.0048(12)
C5	0.0331(14)	0.0392(15)	0.0374(14)	-0.0001(12)	0.0002(11)	0.0111(13)
C6	0.0464(18)	0.0441(18)	0.0488(17)	-0.0024(13)	0.0037(14)	0.0126(14)
C7	0.068(2)	0.0316(16)	0.0554(18)	-0.0026(14)	0.0017(16)	0.0060(14)
C8	0.0284(12)	0.0378(15)	0.0358(13)	0.0014(11)	0.0028(11)	0.0047(11)
C9	0.0320(13)	0.0384(15)	0.0367(13)	0.0019(12)	0.0029(10)	0.0029(12)
C10	0.0371(15)	0.0443(17)	0.0432(15)	0.0008(12)	0.0061(12)	0.0097(13)
C12	0.0321(13)	0.0301(13)	0.0327(12)	0.0014(11)	0.0075(10)	-0.0009(11)
C13	0.0372(14)	0.0429(16)	0.0393(14)	-0.0055(12)	0.0059(11)	-0.0004(13)
C14	0.0535(18)	0.0466(17)	0.0377(15)	-0.0082(14)	0.0124(13)	0.0071(13)
C15	0.0538(17)	0.0439(17)	0.0297(13)	0.0047(13)	0.0075(12)	0.0010(12)
C16	0.0405(15)	0.0460(17)	0.0380(14)	-0.0022(13)	-0.0018(11)	-0.0011(13)
C17	0.0358(14)	0.0425(16)	0.0403(14)	-0.0055(12)	0.0029(11)	0.0075(13)
C18	0.0364(14)	0.0394(15)	0.0294(13)	0.0027(11)	0.0069(11)	0.0007(11)
C19	0.0378(15)	0.0464(17)	0.0445(17)	0.0049(13)	-0.0002(12)	0.0026(13)
C20	0.0438(16)	0.0471(18)	0.0493(18)	-0.0035(14)	-0.0005(13)	-0.0046(14)
C21	0.0544(17)	0.0375(15)	0.0380(15)	-0.0014(14)	0.0081(13)	-0.0051(13)
C22	0.0473(19)	0.0447(18)	0.0537(18)	0.0120(14)	0.0008(14)	0.0017(15)

C23	0.0375(15)	0.0449(17)	0.0471(17)	0.0027(13)	-0.0023(13) 0.0004(14)

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

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Cl24	C21	1.746(3)	O11	C9	1.435(3)
O11	C10	1.430(3)	C2	C3	1.380(4)
C2	C4	1.393(4)	C3	C7	1.382(5)
C4	C5	1.390(4)	C4	C8	1.523(4)
C5	C6	1.398(4)	C5	C10	1.506(4)
C6	C7	1.375(4)	C8	C9	1.533(4)
C8	C12	1.527(3)	C9	C18	1.512(4)
C12	C13	1.389(4)	C12	C17	1.387(3)
C13	C14	1.382(4)	C14	C15	1.378(4)
C15	C16	1.373(4)	C16	C17	1.393(4)
C18	C19	1.387(4)	C18	C23	1.382(4)
C19	C20	1.375(4)	C20	C21	1.384(4)
C21	C22	1.378(4)	C22	C23	1.388(4)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C2	H2	0.930	C3	H3	0.930
C6	H6	0.930	C7	H7	0.930
C8	H8	0.980	C9	H9	0.980
C10	H10A	0.970	C10	H10B	0.970

C13	H13	0.930	C14	H14	0.930
C15	H15	0.930	C16	H16	0.930
C17	H17	0.930	C19	H19	0.930
C20	H20	0.930	C22	H22	0.930
C23	H23	0.930			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C9	O11	C10	109.15(19)	C3	C2	C4	121.0(3)
C2	C3	C7	119.6(3)	C2	C4	C5	119.2(3)
C2	C4	C8	120.2(2)	C5	C4	C8	120.5(2)
C4	C5	C6	119.3(3)	C4	C5	C10	120.1(2)
C6	C5	C10	120.6(3)	C5	C6	C7	120.7(3)
C3	C7	C6	120.2(3)	C4	C8	C9	108.7(2)
C4	C8	C12	111.0(2)	C9	C8	C12	113.9(2)
O11	C9	C8	109.64(19)	O11	C9	C18	109.8(2)
C8	C9	C18	114.9(2)	O11	C10	C5	113.4(2)
C8	C12	C13	119.5(2)	C8	C12	C17	122.2(2)
C13	C12	C17	118.3(2)	C12	C13	C14	120.7(2)
C13	C14	C15	120.7(3)	C14	C15	C16	119.4(2)
C15	C16	C17	120.3(2)	C12	C17	C16	120.7(2)
C9	C18	C19	118.6(2)	C9	C18	C23	122.9(2)
C19	C18	C23	118.5(3)	C18	C19	C20	121.7(3)
C19	C20	C21	118.9(3)	Cl24	C21	C20	119.2(2)
Cl24	C21	C22	120.2(2)	C20	C21	C22	120.6(3)
C21	C22	C23	119.6(3)	C18	C23	C22	120.6(3)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C3	C2	H2	119.5	C4	C2	H2	119.5
C2	C3	H3	120.2	C7	C3	H3	120.2
C5	C6	H6	119.7	C7	C6	H6	119.7
C3	C7	H7	119.9	C6	C7	H7	119.9
C4	C8	H8	107.7	C9	C8	H8	107.7
C12	C8	H8	107.7	O11	C9	H9	107.4
C8	C9	H9	107.4	C18	C9	H9	107.4
O11	C10	H10A	108.9	O11	C10	H10B	108.9
C5	C10	H10A	108.9	C5	C10	H10B	108.9

H10A	C10	H10B	107.7	C12	C13	H13	119.7
C14	C13	H13	119.7	C13	C14	H14	119.7
C15	C14	H14	119.7	C14	C15	H15	120.3
C16	C15	H15	120.3	C15	C16	H16	119.8
C17	C16	H16	119.8	C12	C17	H17	119.7
C16	C17	H17	119.6	C18	C19	H19	119.2
C20	C19	H19	119.2	C19	C20	H20	120.5
C21	C20	H20	120.5	C21	C22	H22	120.2
C23	C22	H22	120.2	C18	C23	H23	119.7
C22	C23	H23	119.7				

Table 8. Torsion Angles(°)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C9	O11	C10	C5	52.3(2)	C10	O11	C9	C8	-71.7(2)
C10	O11	C9	C18	161.20(18)	C3	C2	C4	C5	-0.5(4)
C3	C2	C4	C8	177.3(2)	C4	C2	C3	C7	-0.4(4)
C2	C3	C7	C6	0.7(4)	C2	C4	C5	C6	1.2(3)
C2	C4	C5	C10	-177.6(2)	C2	C4	C8	C9	161.2(2)
C2	C4	C8	C12	-72.8(3)	C5	C4	C8	C9	-21.1(3)
C5	C4	C8	C12	105.0(2)	C8	C4	C5		
C6	-176.57(19)								
C8	C4	C5	C10	4.6(3)	C4	C5	C6	C7	-0.9(4)
C4	C5	C10	O11	-19.4(3)	C6	C5	C10	O11	161.8(2)
C10	C5	C6	C7	177.9(2)	C5	C6	C7	C3	-0.0(4)
C4	C8	C9	O11	53.7(2)	C4	C8	C9	C18	
177.95(17)									
C4	C8	C12	C13	139.0(2)	C4	C8	C12	C17	-39.7(3)
C9	C8	C12	C13	-97.9(2)	C9	C8	C12	C17	83.3(3)
C12	C8	C9	O11	-70.6(3)	C12	C8	C9	C18	53.6(3)
O11	C9	C18	C19	-151.61(19)	O11	C9	C18	C23	27.5(3)
C8	C9	C18	C19	84.3(3)	C8	C9	C18	C23	-96.6(3)
C8	C12	C13	C14	-178.8(2)	C8	C12	C17	C16	179.2(2)
C13	C12	C17	C16	0.5(4)	C17	C12	C13	C14	-0.1(4)
C12	C13	C14	C15	0.2(4)	C13	C14	C15	C16	-0.6(4)
C14	C15	C16	C17	1.0(4)	C15	C16	C17	C12	-1.0(4)
C9	C18	C19	C20	177.1(2)	C9	C18	C23	C22	-177.1(2)
C19	C18	C23	C22	2.1(4)	C23	C18	C19	C20	-2.1(4)
C18	C19	C20	C21	0.6(4)	C19	C20	C21	Cl24	-179.1(2)
C19	C20	C21	C22	0.9(4)	Cl24	C21	C22	C23	
179.09(19)									
C20	C21	C22	C23	-1.0(4)	C21	C22	C23	C18	-0.6(4)

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O11	C4	2.805(3)	O11	C12	3.075(3)
O11	C17	3.273(3)	O11	C23	2.819(3)
C2	C6	2.755(4)	C2	C12	3.186(4)
C2	C17	3.574(4)	C3	C5	2.782(4)
C4	C7	2.781(4)	C4	C17	2.957(4)
C5	C9	2.775(4)	C5	C12	3.472(4)
C5	C17	3.544(4)	C8	C10	2.921(4)
C8	C19	3.321(4)	C8	C23	3.480(4)
C9	C13	3.451(4)	C9	C17	3.360(3)
C12	C15	2.794(3)	C12	C18	3.054(3)
C12	C23	3.430(4)	C13	C16	2.756(4)
C13	C18	3.414(4)	C14	C17	2.752(4)
C18	C21	2.773(4)	C19	C22	2.751(4)
C20	C23	2.769(4)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Cl24	H20	2.803	Cl24	H22	2.813
O11	H8	3.246	O11	H17	2.942
O11	H23	2.541	C2	H7	3.227
C2	H8	2.661	C2	H17	3.413
C3	H6	3.226	C4	H3	3.251
C4	H6	3.245	C4	H9	2.659

C4	H10A	3.014	C4	H10B	3.215
C4	H17	2.701	C5	H2	3.239
C5	H7	3.247	C5	H8	3.215
C5	H9	2.819	C5	H17	2.953
C6	H3	3.228	C6	H10A	2.862
C6	H10B	2.648	C7	H2	3.225
C8	H2	2.666	C8	H10A	3.398
C8	H13	2.653	C8	H17	2.705
C8	H19	3.268	C8	H23	3.544
C9	H10A	2.587	C9	H10B	3.171
C9	H13	3.503	C9	H17	3.355
C9	H19	2.614	C9	H23	2.705
C10	H6	2.669	C10	H9	2.394
C10	H17	3.283	C12	H2	3.127
C12	H9	3.374	C12	H14	3.243
C12	H16	3.251	C12	H23	3.298
C13	H8	2.554	C13	H15	3.238
C13	H17	3.225	C14	H16	3.215
C15	H13	3.233	C15	H17	3.233
C16	H14	3.213	C17	H8	3.293
C17	H13	3.225	C17	H15	3.240
C17	H23	3.530	C18	H8	2.790
C18	H13	3.302	C18	H20	3.249
C18	H22	3.244	C19	H8	3.125
C19	H9	2.566	C19	H13	3.529

C19	H23	3.222	C20	H22	3.238
C21	H19	3.214	C21	H23	3.229
C22	H20	3.239	C23	H9	3.242
C23	H19	3.218	H2	H3	2.306
H2	H8	2.530	H3	H7	2.314
H6	H7	2.299	H6	H10A	2.894

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H6	H10B	2.515	H8	H9	2.285
H8	H13	2.356	H8	H17	3.579
H8	H19	2.865	H9	H10A	2.278
H9	H10B	3.323	H9	H19	2.400
H9	H23	3.522	H10B	H17	3.416
H13	H14	2.302	H14	H15	2.308
H15	H16	2.305	H16	H17	2.316
H19	H20	2.300	H22	H23	2.317

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
O11	C15[1]	3.455(3)	C3	C14[2]	3.600(4)
C9	C15[1]	3.581(4)	C14	C3[3]	3.600(4)
C15	O11[4]	3.455(3)	C15	C9[4]	3.581(4)
C15	C18[4]	3.516(4)	C18	C15[1]	3.516(4)

Symmetry Operators:

(1) X,Y,Z-1
(2) -X+1,Y+1/2,-Z+2
(3) -X+1,Y+1/2-1,-Z+2
(4) X,Y,Z+1

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Cl24	H2[1]	3.035	Cl24	H9[2]	3.323
Cl24	H10A[2]	3.268	Cl24	H10A[3]	3.555
Cl24	H10B[3]	3.355	Cl24	H16[4]	3.042
Cl24	H17[4]	3.561	O11	H8[5]	2.806
O11	H15[6]	2.877	O11	H19[5]	2.915
C2	H6[7]	3.423	C2	H10B[7]	3.158
C2	H22[8]	3.550	C3	H6[7]	3.512
C3	H13[9]	3.259	C3	H14[9]	3.091
C3	H22[8]	3.104	C3	H23[8]	3.435
C4	H10B[7]	3.112	C5	H2[5]	3.532
C6	H2[5]	3.425	C6	H20[10]	3.086
C7	H13[9]	3.355	C7	H14[9]	3.031
C7	H20[10]	3.433	C7	H22[8]	3.199
C7	H23[8]	3.314	C8	H10B[7]	3.042
C9	H15[6]	3.078	C10	H8[5]	2.960
C10	H15[6]	3.595	C10	H16[6]	3.584

C13	H3[1]	3.011	C13	H7[1]	3.425
C13	H16[7]	3.210	C13	H17[7]	3.529
C14	H3[1]	3.154	C14	H6[4]	3.429
C14	H7[1]	3.409	C14	H7[4]	3.219
C14	H9[11]	3.536	C14	H16[7]	3.200
C15	H6[4]	3.458	C15	H7[4]	2.852
C15	H9[11]	3.196	C15	H10A[11]	3.322
C15	H19[12]	3.281	C16	H7[4]	3.009
C16	H10A[11]	3.177	C16	H13[5]	3.216
C16	H14[5]	3.522	C16	H19[12]	3.436
C17	H7[4]	3.522	C17	H13[5]	3.215
C18	H14[6]	3.474	C18	H15[6]	2.847
C19	H3[1]	3.597	C19	H14[6]	2.979
C19	H15[13]	3.355	C19	H15[6]	3.358
C19	H23[7]	3.166	C20	H3[1]	3.310
C20	H14[6]	3.241	C20	H22[7]	3.411
C20	H23[7]	3.266	C21	H2[1]	3.336
C21	H3[1]	3.134	C22	H2[1]	3.556
C22	H3[1]	3.262	C22	H20[5]	3.140
C23	H3[1]	3.563	C23	H7[4]	3.415
C23	H15[6]	3.094	C23	H19[5]	3.396

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C23	H20^{5}	3.249	H2	Cl24^{9}	3.035
H2	C5^{7}	3.532	H2	C6^{7}	3.425
H2	C21^{9}	3.336	H2	C22^{9}	3.556
H2	H6^{7}	3.315	H2	H10B^{7}	3.020
H2	H17^{7}	2.993	H2	H22^{9}	3.438
H3	C13^{9}	3.011	H3	C14^{9}	3.154
H3	C19^{9}	3.597	H3	C20^{9}	3.310
H3	C21^{9}	3.134	H3	C22^{9}	3.262
H3	C23^{9}	3.563	H3	H6^{7}	3.466
H3	H13^{9}	2.608	H3	H14^{9}	2.874
H3	H22^{8}	3.265	H3	H23^{8}	3.142
H6	C2^{5}	3.423	H6	C3^{5}	3.512
H6	C14^{8}	3.429	H6	C15^{8}	3.458
H6	H2^{5}	3.315	H6	H3^{5}	3.466
H6	H14^{8}	3.201	H6	H15^{8}	3.266
H6	H20^{10}	2.946	H7	C13^{9}	3.425
H7	C14^{9}	3.409	H7	C14^{8}	3.219
H7	C15^{8}	2.852	H7	C16^{8}	3.009

H7	C17[8]	3.522	H7	C23[8]	3.415
H7	H13[9]	2.804	H7	H14[9]	2.771
H7	H15[8]	3.071	H7	H16[8]	3.299
H7	H20[10]	3.532	H7	H22[8]	3.405
H7	H23[8]	2.903	H8	O11[7]	2.806
H8	C10[7]	2.960	H8	H10B[7]	2.323
H8	H17[7]	3.132	H9	Cl24[10]	3.323
H9	C14[6]	3.536	H9	C15[6]	3.196
H9	H10B[7]	3.124	H9	H14[6]	3.536
H9	H15[6]	2.941	H9	H16[13]	3.448
H10A	Cl24[10]	3.268	H10A	Cl24[14]	3.555
H10A	C15[6]	3.322	H10A	C16[6]	3.177
H10A	H15[6]	3.263	H10A	H16[6]	3.021
H10B	Cl24[14]	3.355	H10B	C2[5]	3.158
H10B	C4[5]	3.112	H10B	C8[5]	3.042
H10B	H2[5]	3.020	H10B	H8[5]	2.323
H10B	H9[5]	3.124	H10B	H16[6]	3.545
H10B	H19[5]	3.466	H13	C3[1]	3.259
H13	C7[1]	3.355	H13	C16[7]	3.216

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H13	C17^{7}	3.215	H13	H3^{1}	2.608
H13	H7^{1}	2.804	H13	H16^{7}	3.035
H13	H17^{7}	3.043	H13	H23^{7}	3.047
H14	C3^{1}	3.091	H14	C7^{1}	3.031
H14	C16^{7}	3.522	H14	C18^{11}	3.474
H14	C19^{11}	2.979	H14	C20^{11}	3.241
H14	H3^{1}	2.874	H14	H6^{4}	3.201
H14	H7^{1}	2.771	H14	H9^{11}	3.536
H14	H16^{7}	3.020	H14	H19^{11}	3.008
H14	H20^{11}	3.428	H15	O11^{11}	2.877
H15	C9^{11}	3.078	H15	C10^{11}	3.595
H15	C18^{11}	2.847	H15	C19^{11}	3.358
H15	C19^{12}	3.355	H15	C23^{11}	3.094
H15	H6^{4}	3.266	H15	H7^{4}	3.071
H15	H9^{11}	2.941	H15	H10A^{11}	3.263
H15	H19^{12}	2.594	H15	H20^{12}	3.129
H15	H23^{11}	3.323	H16	Cl24^{8}	3.042
H16	C10^{11}	3.584	H16	C13^{5}	3.210
H16	C14^{5}	3.200	H16	H7^{4}	3.299
H16	H9^{12}	3.448	H16	H10A^{11}	3.021

H16	H10B^{11}	3.545	H16	H13^{5}	3.035
H16	H14^{5}	3.020	H16	H19^{12}	2.903
H17	Cl24^{8}	3.561	H17	C13^{5}	3.529
H17	H2^{5}	2.993	H17	H8^{5}	3.132
H17	H13^{5}	3.043	H17	H22^{8}	3.522
H19	O11^{7}	2.915	H19	C15^{13}	3.281
H19	C16^{13}	3.436	H19	C23^{7}	3.396
H19	H10B^{7}	3.466	H19	H14^{6}	3.008
H19	H15^{13}	2.594	H19	H16^{13}	2.903
H19	H23^{7}	2.910	H20	C6^{2}	3.086
H20	C7^{2}	3.433	H20	C22^{7}	3.140
H20	C23^{7}	3.249	H20	H6^{2}	2.946
H20	H7^{2}	3.532	H20	H14^{6}	3.428
H20	H15^{13}	3.129	H20	H22^{7}	2.910
H20	H23^{7}	3.105	H22	C2^{4}	3.550
H22	C3^{4}	3.104	H22	C7^{4}	3.199
H22	C20^{5}	3.411	H22	H2^{1}	3.438

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H22	H3^{4}	3.265	H22	H7^{4}	3.405

H22	H17[4]	3.522	H22	H20[5]	2.910
H23	C3[4]	3.435	H23	C7[4]	3.314
H23	C19[5]	3.166	H23	C20[5]	3.266
H23	H3[4]	3.142	H23	H7[4]	2.903
H23	H13[5]	3.047	H23	H15[6]	3.323
H23	H19[5]	2.910	H23	H20[5]	3.105

Symmetry Operators:

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