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2,4-Dichlorobenzyl 2-methoxybenzoate

Arun M. Isloor,[a] B. Garudachari,[a] Thomas Gerber,[b] Eric Hosten[b] and Richard Betz[b]*

[a]National Institute of Technology-Karnataka, Department of Chemistry, Surathkal, Mangalore 575 025, India, and [b]Nelson Mandela Metropolitan University, Summerstrand Campus, Department of Chemistry, University Way, Summerstrand, PO Box 77000, Port Elizabeth, 6031, South Africa
Correspondence e-mail: richard.betz@webmail.co.za

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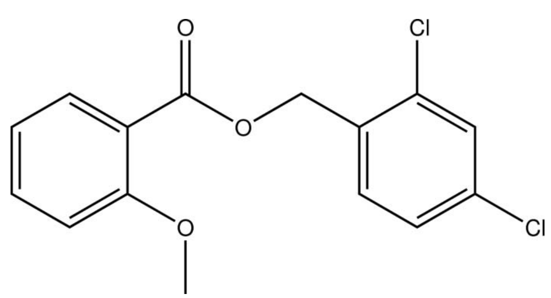
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.080; data-to-parameter ratio = 18.6.

In the title compound, $C_{15}H_{12}Cl_2O_3$, the aromatic rings make a dihedral angle of 10.78 (4)°. In the molecule, there is a short C—H···O contact. In the crystal, C—H···O contacts connect the molecules into $C(7)C(8)$ chains along the b axis. The shortest intercentroid distance between two benzoic acid aromatic systems is 3.7416 (8) Å.

Related literature

For pharmacological properties of phenyl benzoates, see: Oxford *et al.* (2005); Ostergaard (1994). For graph-set analysis of hydrogen bonds, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

$C_{15}H_{12}Cl_2O_3$
M_r = 311.15
Monoclinic, $P2_1/c$
a = 12.1816 (5) Å
b = 15.2481 (6) Å
c = 7.4207 (4) Å
β = 99.299 (2)°
V = 1360.25 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.48 mm^{-1}
T = 200 K
0.38 × 0.37 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.847, T_{max} = 0.982
12933 measured reflections
3379 independent reflections
2868 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.080
S = 1.03
3379 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3A···O2^i	0.98	2.59	3.5433 (18)	165
C15—H15···O2^i	0.95	2.51	3.3945 (15)	154
C16—H16···O3	0.95	2.48	3.3989 (15)	163

Symmetry code: (i) $-x+1, y+\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5318).

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supplementary materials

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2,4-Dichlorobenzyl 2-methoxybenzoate

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Comment

For decades, phenyl benzoate derivatives have found ample application in the identification of organic acids. 2,4-Dichlorobenzyl derivatives show pharmacological activity such as acting as mild antiseptics and showing the ability to kill bacteria and viruses associated with mouth and throat infections (Oxford *et al.*, 2005; Ostergaard 1994). In continuation of our research focused on the crystal structures of medical compounds, the title compound was synthesized.

The molecule is essentially flat. The atom deviating most from the least-squares plane defined by all non-hydrogen atoms is the chlorine atom in *para* position to the benzyloxy moiety with a deviation of 0.237 (1) Å. The least-squares planes defined by the respective carbon atoms of the aromatic systems intersect at an angle of 10.78 (4) ° (Fig. 1).

In the crystal, intermolecular C–H···O contacts whose range falls by more than 0.1 Å below the sum of van-der-Waals radii are observed. These are established between one of the hydrogen atoms of the methoxy group as well as one of the two hydrogen atoms in *ortho* position to the chlorine atom in *para* position to the benzyloxy group and chelate the ketonic oxygen atom of the ester moiety. In total, the molecules are connected to chains along the crystallographic *b* axis. In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995), the descriptor for these contacts is $C^1_1(7)C^1_1(8)$ on the unary level. Information about metrical parameters as well as the symmetry of those contacts has been summarized in Table 1. The shortest intercentroid distance between two aromatic systems was measured at 3.7416 (8) Å and is apparent between the benzoic acid moiety and its symmetry-generated equivalent (Fig. 2).

The packing of the title compound in the crystal structure is shown in Figure 3.

Experimental

A mixture of 1-(bromomethyl)-2,4-dichlorobenzene (0.1 g, 0.0004 mol), potassium carbonate (0.062 g, 0.00045 mol) and 2-methoxybenzoic acid (0.068 g, 0.00045 mol) in dimethylformamide (5 ml) was stirred at 60 °C for 2 h. After completion of the reaction, the reaction mixture was poured into ice-cold water. The solid product obtained was filtered, washed with water and recrystallized from ethanol (yield: 0.120 g, 93.0%).

Refinement

Carbon-bound H atoms were placed in calculated positions (C–H 0.95 Å for aromatic carbon atoms and C–H 0.99 Å for methylene groups) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C). The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C–C bond to best fit the experimental electron density with U(H) set to 1.5U_{eq}(C).

Computing details

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT* (Bruker, 2010); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2008);

software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

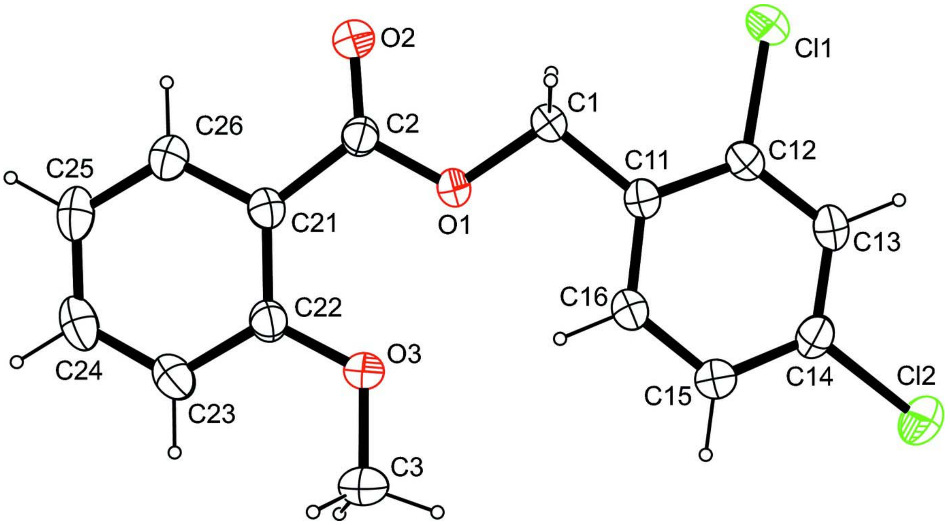

Figure 1

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level).

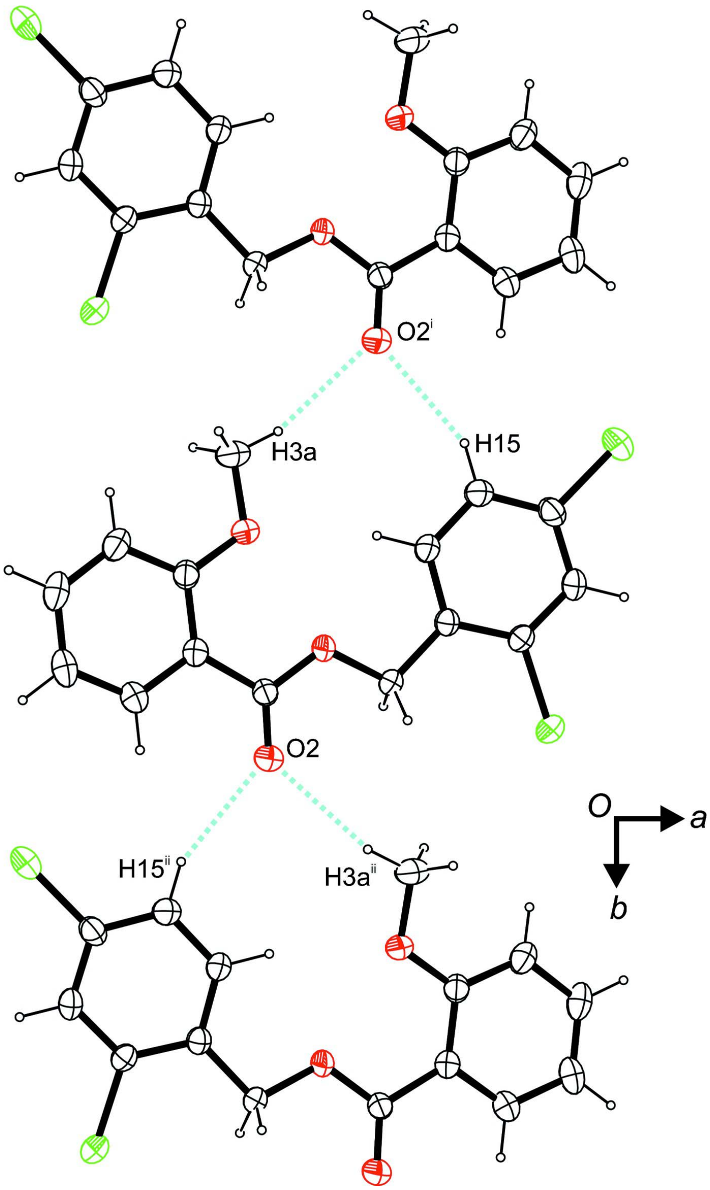

Figure 2

Intermolecular contacts, viewed along [0 0 1]. Symmetry operators: ^{i} $-x + 1$, $y + 1/2$, $-z + 1/2$; ^{ii} $-x + 1$, $y - 1/2$, $-z + 1/2$.

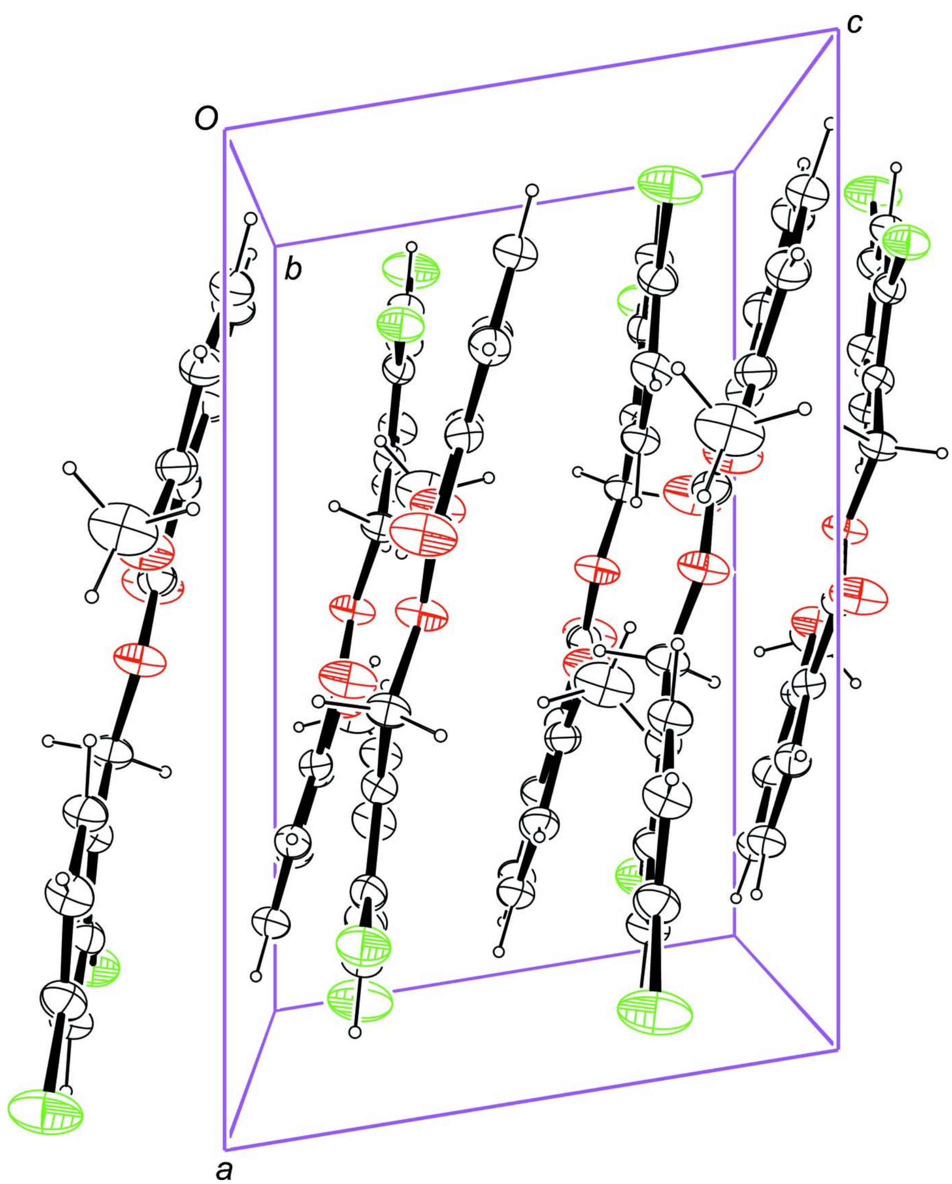

Figure 3

Molecular packing of the title compound, viewed along [0 1 0] (anisotropic displacement ellipsoids drawn at 50% probability level).

2,4-Dichlorobenzyl 2-methoxybenzoate

Crystal data

$C_{15}H_{12}Cl_2O_3$	V = 1360.25 (11) Å^3
M_r = 311.15	Z = 4
Monoclinic, $P2_1/c$	F(000) = 640
Hall symbol: -P 2ybc	D_x = 1.519 Mg m^{-3}
a = 12.1816 (5) Å	Melting point = 376–374 K
b = 15.2481 (6) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
c = 7.4207 (4) Å	Cell parameters from 6565 reflections
β = 99.299 (2)°	θ = 2.7–28.3°

μ = 0.48 mm^{-1}
T = 200 K

Block, colourless
0.38 × 0.37 × 0.15 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.847, T_{max} = 0.982

12933 measured reflections
3379 independent reflections
2868 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 28.4°, θ_{min} = 2.2°
$h = -16 \rightarrow 16$
$k = -20 \rightarrow 20$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.080
S = 1.03
3379 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0407P)^2 + 0.3593P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	0.83729 (2)	0.13621 (2)	0.22173 (5)	0.03453 (10)
Cl2	0.93726 (3)	0.47791 (2)	0.20393 (6)	0.05086 (12)
O1	0.50796 (7)	0.23368 (5)	0.32072 (13)	0.0302 (2)
O2	0.42680 (8)	0.10301 (6)	0.32410 (15)	0.0417 (2)
O3	0.39479 (7)	0.37220 (5)	0.34997 (14)	0.0354 (2)
C1	0.60253 (9)	0.19251 (7)	0.26253 (17)	0.0260 (2)
H1A	0.5802	0.1629	0.1435	0.031*
H1B	0.6354	0.1483	0.3532	0.031*
C2	0.42351 (9)	0.18141 (7)	0.34359 (16)	0.0250 (2)
C3	0.37406 (13)	0.46402 (9)	0.3289 (2)	0.0465 (4)
H3A	0.4363	0.4920	0.2823	0.070*
H3B	0.3664	0.4896	0.4473	0.070*
H3C	0.3053	0.4735	0.2425	0.070*
C11	0.68490 (9)	0.26426 (7)	0.24577 (15)	0.0234 (2)
C12	0.79427 (9)	0.24488 (7)	0.22730 (16)	0.0244 (2)
C13	0.87222 (10)	0.30935 (8)	0.21331 (17)	0.0291 (3)
H13	0.9464	0.2945	0.2012	0.035*
C14	0.83948 (10)	0.39622 (8)	0.21735 (17)	0.0303 (3)
C15	0.73151 (10)	0.41879 (8)	0.23178 (18)	0.0306 (3)
H15	0.7097	0.4786	0.2319	0.037*
C16	0.65539 (10)	0.35254 (8)	0.24611 (17)	0.0274 (2)
H16	0.5810	0.3678	0.2564	0.033*
C21	0.32638 (9)	0.22915 (7)	0.39497 (16)	0.0249 (2)
C22	0.31284 (9)	0.32091 (8)	0.39762 (16)	0.0268 (2)

C23	0.21596 (10)	0.35594 (9)	0.44706 (18)	0.0327 (3)
H23	0.2066	0.4177	0.4505	0.039*
C24	0.13359 (10)	0.30135 (10)	0.49094 (18)	0.0355 (3)
H24	0.0686	0.3260	0.5261	0.043*
C25	0.14469 (10)	0.21127 (10)	0.48428 (18)	0.0351 (3)
H25	0.0870	0.1740	0.5113	0.042*
C26	0.24091 (10)	0.17634 (8)	0.43777 (17)	0.0296 (3)
H26	0.2491	0.1144	0.4349	0.036*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.02915 (16)	0.02768 (16)	0.0483 (2)	0.00585 (11)	0.01086 (13)	−0.00228 (12)
Cl2	0.03554 (19)	0.03606 (19)	0.0846 (3)	−0.00991 (14)	0.02065 (18)	0.00427 (17)
O1	0.0219 (4)	0.0242 (4)	0.0472 (5)	−0.0005 (3)	0.0138 (4)	−0.0033 (3)
O2	0.0334 (5)	0.0234 (4)	0.0723 (7)	−0.0010 (4)	0.0204 (5)	−0.0008 (4)
O3	0.0301 (4)	0.0218 (4)	0.0571 (6)	0.0015 (3)	0.0155 (4)	0.0014 (4)
C1	0.0220 (5)	0.0230 (5)	0.0345 (6)	0.0021 (4)	0.0090 (4)	−0.0009 (5)
C2	0.0220 (5)	0.0251 (5)	0.0280 (6)	−0.0011 (4)	0.0040 (4)	0.0020 (4)
C3	0.0460 (8)	0.0234 (6)	0.0740 (11)	0.0030 (6)	0.0218 (8)	0.0014 (6)
C11	0.0219 (5)	0.0261 (5)	0.0227 (5)	0.0005 (4)	0.0048 (4)	0.0003 (4)
C12	0.0244 (5)	0.0259 (5)	0.0235 (5)	0.0033 (4)	0.0052 (4)	−0.0005 (4)
C13	0.0218 (5)	0.0350 (6)	0.0318 (6)	0.0008 (5)	0.0086 (4)	0.0001 (5)
C14	0.0282 (6)	0.0295 (6)	0.0347 (6)	−0.0058 (5)	0.0092 (5)	0.0012 (5)
C15	0.0311 (6)	0.0252 (6)	0.0372 (7)	0.0006 (5)	0.0102 (5)	0.0013 (5)
C16	0.0231 (5)	0.0276 (6)	0.0329 (6)	0.0023 (4)	0.0085 (5)	0.0009 (5)
C21	0.0220 (5)	0.0277 (6)	0.0250 (6)	0.0003 (4)	0.0036 (4)	−0.0007 (4)
C22	0.0224 (5)	0.0291 (6)	0.0290 (6)	0.0003 (4)	0.0044 (4)	−0.0006 (5)
C23	0.0274 (6)	0.0362 (7)	0.0346 (7)	0.0064 (5)	0.0053 (5)	−0.0045 (5)
C24	0.0238 (6)	0.0517 (8)	0.0318 (6)	0.0049 (5)	0.0068 (5)	−0.0059 (6)
C25	0.0245 (6)	0.0493 (8)	0.0328 (7)	−0.0062 (5)	0.0089 (5)	−0.0022 (6)
C26	0.0271 (6)	0.0330 (6)	0.0293 (6)	−0.0041 (5)	0.0063 (5)	−0.0003 (5)

Geometric parameters (Å, º)

Cl1—C12	1.7403 (12)	C13—C14	1.3849 (17)
Cl2—C14	1.7374 (12)	C13—H13	0.9500
O1—C2	1.3337 (13)	C14—C15	1.3804 (17)
O1—C1	1.4383 (13)	C15—C16	1.3869 (17)
O2—C2	1.2057 (15)	C15—H15	0.9500
O3—C22	1.3592 (14)	C16—H16	0.9500
O3—C3	1.4267 (15)	C21—C26	1.3929 (16)
C1—C11	1.5033 (15)	C21—C22	1.4093 (16)
C1—H1A	0.9900	C22—C23	1.3973 (16)
C1—H1B	0.9900	C23—C24	1.3826 (19)
C2—C21	1.4902 (16)	C23—H23	0.9500
C3—H3A	0.9800	C24—C25	1.382 (2)
C3—H3B	0.9800	C24—H24	0.9500
C3—H3C	0.9800	C25—C26	1.3811 (17)
C11—C12	1.3929 (16)	C25—H25	0.9500

C11—C16	1.3933 (16)	C26—H26	0.9500
C12—C13	1.3823 (17)		

C2—O1—C1	116.68 (9)	C15—C14—Cl2	119.76 (10)
C22—O3—C3	117.96 (10)	C13—C14—Cl2	118.81 (9)
O1—C1—C11	106.55 (9)	C14—C15—C16	118.79 (11)
O1—C1—H1A	110.4	C14—C15—H15	120.6
C11—C1—H1A	110.4	C16—C15—H15	120.6
O1—C1—H1B	110.4	C15—C16—C11	121.85 (11)
C11—C1—H1B	110.4	C15—C16—H16	119.1
H1A—C1—H1B	108.6	C11—C16—H16	119.1
O2—C2—O1	122.41 (11)	C26—C21—C22	118.51 (11)
O2—C2—C21	123.93 (11)	C26—C21—C2	115.44 (10)
O1—C2—C21	113.66 (10)	C22—C21—C2	126.02 (10)
O3—C3—H3A	109.5	O3—C22—C23	122.36 (11)
O3—C3—H3B	109.5	O3—C22—C21	118.37 (10)
H3A—C3—H3B	109.5	C23—C22—C21	119.27 (11)
O3—C3—H3C	109.5	C24—C23—C22	120.50 (12)
H3A—C3—H3C	109.5	C24—C23—H23	119.8
H3B—C3—H3C	109.5	C22—C23—H23	119.8
C12—C11—C16	117.15 (11)	C25—C24—C23	120.74 (12)
C12—C11—C1	121.04 (10)	C25—C24—H24	119.6
C16—C11—C1	121.81 (10)	C23—C24—H24	119.6
C13—C12—C11	122.41 (11)	C26—C25—C24	118.96 (12)
C13—C12—Cl1	117.53 (9)	C26—C25—H25	120.5
C11—C12—Cl1	120.05 (9)	C24—C25—H25	120.5
C12—C13—C14	118.35 (11)	C25—C26—C21	121.99 (12)
C12—C13—H13	120.8	C25—C26—H26	119.0
C14—C13—H13	120.8	C21—C26—H26	119.0
C15—C14—C13	121.43 (11)		

C2—O1—C1—C11	179.67 (10)	O2—C2—C21—C26	6.06 (18)
C1—O1—C2—O2	2.90 (17)	O1—C2—C21—C26	−173.90 (10)
C1—O1—C2—C21	−177.14 (10)	O2—C2—C21—C22	−171.70 (12)
O1—C1—C11—C12	167.09 (10)	O1—C2—C21—C22	8.34 (16)
O1—C1—C11—C16	−13.58 (15)	C3—O3—C22—C23	−7.76 (18)
C16—C11—C12—C13	1.25 (18)	C3—O3—C22—C21	171.63 (12)
C1—C11—C12—C13	−179.38 (11)	C26—C21—C22—O3	−177.87 (11)
C16—C11—C12—Cl1	−178.92 (9)	C2—C21—C22—O3	−0.17 (18)
C1—C11—C12—Cl1	0.44 (16)	C26—C21—C22—C23	1.53 (17)
C11—C12—C13—C14	−0.22 (18)	C2—C21—C22—C23	179.23 (11)
Cl1—C12—C13—C14	179.96 (9)	O3—C22—C23—C24	178.65 (12)
C12—C13—C14—C15	−1.10 (19)	C21—C22—C23—C24	−0.73 (18)
C12—C13—C14—Cl2	178.88 (9)	C22—C23—C24—C25	−0.9 (2)
C13—C14—C15—C16	1.3 (2)	C23—C24—C25—C26	1.7 (2)
Cl2—C14—C15—C16	−178.68 (10)	C24—C25—C26—C21	−0.90 (19)
C14—C15—C16—C11	−0.19 (19)	C22—C21—C26—C25	−0.73 (18)
C12—C11—C16—C15	−1.04 (18)	C2—C21—C26—C25	−178.67 (11)
C1—C11—C16—C15	179.60 (11)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3*A*···O2[i]	0.98	2.59	3.5433 (18)	165
C15—H15···O2[i]	0.95	2.51	3.3945 (15)	154
C16—H16···O3	0.95	2.48	3.3989 (15)	163

Symmetry code: (i) −*x*+1, *y*+1/2, −*z*+1/2.