Acta Crystallographica Section E
Structure Reports Online

ISSN 1600-5368

4-Chloro-2-(2-chlorobenzoyl)phenol

A. Bushra Begum,[a] S. Madan Kumar,[b] B. C. Manjunath,[b] Shaukath Ara Khanum[a]* and N. K. Lokanath[b]

[a]Department of Chemistry, Yuvaraja's College (Autonomous), University of Mysore, Mysore, 570 005, India, and [b]Department of Studies in Physics, Manasagangotri, University of Mysore, Mysore, 570 006, India
Correspondence e-mail: shaukathara@yahoo.co.in

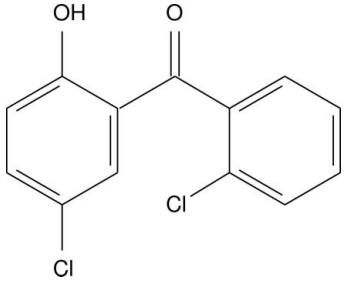
Received 4 September 2013; accepted 16 September 2013

Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.105; data-to-parameter ratio = 12.8.

In the title molecule, $C_{13}H_8Cl_2O_2$, the dihedral angle between the benzene rings is 74.53 (9)°. An intramolecular O—H···O hydrogen bond leading to a S(6) ring is observed. In the crystal, the molecules are connected into a three-dimensional network by C—H···O and π–π [inter-centroid distance = 3.6254 (10) Å] interactions.

Related literature

For the biological activity of benzophenone derivatives, see: Khanum *et al.* (2005, 2010). For a related structure, see: Devaiah *et al.* (2006).



Experimental

Crystal data

$C_{13}H_8Cl_2O_2$
M_r = 267.09
Orthorhombic, *Pbca*
a = 16.0231 (4) Å
b = 7.4216 (2) Å
c = 19.6843 (5) Å
V = 2340.80 (10) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 4.87 mm^{-1}
T = 295 K
0.20 × 0.19 × 0.18 mm

Data collection

Bruker X8 Proteum diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.442, T_{max} = 0.474
15868 measured reflections
1972 independent reflections
1712 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.105
S = 1.06
1972 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O16—H16···O9	0.82	1.88	2.598 (2)	146
C13—H13···O9^{i}	0.93	2.50	3.413 (3)	168

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

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *Mercury*.

The authors thank the IOE and the University of Mysore for providing the single crystal X-ray diffractometer facility.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK5253).

References

Bruker (2013). *APEX2, SAINT and SADABS*. Bruker AXS Inc., Madaison, Wisconsin, USA.
Devaiah, V. T., Naveen, S., Shashikanth, S., Anandalwar, S. M. & Prasad, J. S. (2006). *Anal. Sci. X-ray Struct. Anal. Online*, **22**, x157–x158.
Khanum, S. A., Begum, B. A., Girish, V. & Khanum, N. F. (2010). *Int. J. Biomed. Sci.* **6**, 60–65.
Khanum, S. A., Shashikanth, S., Umesha, S. & Kavitha, R. (2005). *Eur. J. Med. Chem.* **40**, 1156–1162.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2013). E**69**, o1568 [doi:10.1107/S1600536813025609]

4-Chloro-2-(2-chlorobenzoyl)phenol

A. Bushra Begum, S. Madan Kumar, B. C. Manjunath, Shaukath Ara Khanum and N. K. Lokanath

1. Comment

The on-going research in synthesizing benzophenone derivatives in our laboratory resulted in the title molecule. These derivatives used in the preparation of anti-inflammatory (Khanum *et al.*, 2010) and anti-fungal (Khanum *et al.*, 2005) compounds.

In the title molecule (Fig. 1), the dihedral angle between chlorobenzene (C1–C6) and chlorohydroxybenzene (C10–C15) rings is 74.53 (9)°. The molecule features an intramolecular O—H..O hydrogen bond forming a *S*(6) ring (Table 1). The bond lengths and bond angles are similar to those in the 5-chloro-2-hydroxyphenyl-4-chlorophenyl-methanone structure (Devaiah *et al.*, 2006)

The molecules are connected by C13–H13···O9 hydrogen bonds forming chains along the *a* axis (Fig. 2 and Table 1). Additional C6—Cl7···π(Cg1), Table 1, and π(Cg2···π(Cg2) interactions, with inter-centroid distance 3.6254 (10) Å [*x*-1, -*y*, *z*-1], lead to a three-dimensional architecture, Fig. 2; where Cg1: C1–C6 and Cg2: C10–C15.

2. Experimental

A mixture of anhydrous aluminium chloride (1.74 g, 12.94 mmol) and **include the name of the compound here** (2.0 g, 8.62 mmol), was protected from moisture by a calcium chloride guard tube and heated over an oil bath at 80–90 °C for 45 min. At the end of this period the contents were cooled and decomposed by acidulated ice-cold water. The residual solid was crushed into a powder, dissolved in ether (40 ml) and extracted with 10% sodium hydroxide (3 x 30 ml). The basic aqueous solution was neutralized with 10% hydrochloric acid. The filtered solid was washed with distilled water (3 x 30 ml) and recrystallized from ethanol to afford yellow needles of the title compound. Yield 1.6 g (80%). M.Pt: 357–359 K. IR (Nujol): 1615 *v*(C═O), 3525–3655 cm^{-1} *v*(OH). ^{1}H NMR ($CDCl_3$): δ 6.9–7.5 (m, 7H, Ar—H), 9.2 (bs, 1H, OH). EI–MS: m/*z* 267 (*M*+, 81), 266 (100), 154.5 (57), 111.5 (50). Anal. Calcd. for $C_{13}H_8Cl_2O_2$ (267): C, 58.46; H, 3.02; Cl, 26.55. Found: C, 58.54; H, 3.25; Cl, 26.32%.

3. Refinement

All the hydrogen atoms of the compound are fixed geometrically (C—H = 0.93–0.97 Å, O—H= 0.82 Å) and refined as riding with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C, O).

Computing details

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT* (Bruker, 2013); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *Mercury* (Macrae *et al.*, 2008).

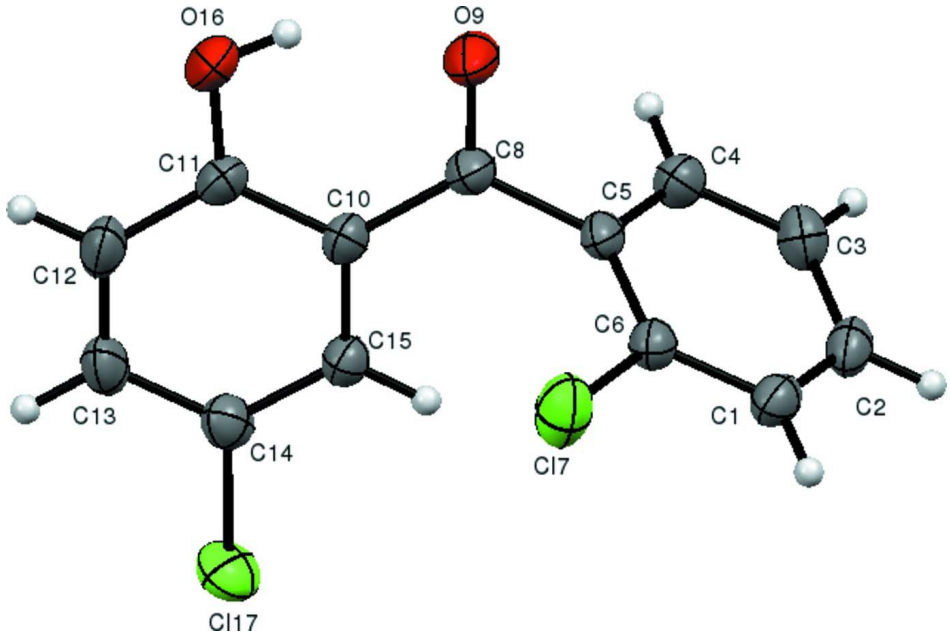


Figure 1

Molecular structure of the title compound along *b*-axis with 50% probability ellipsoids.

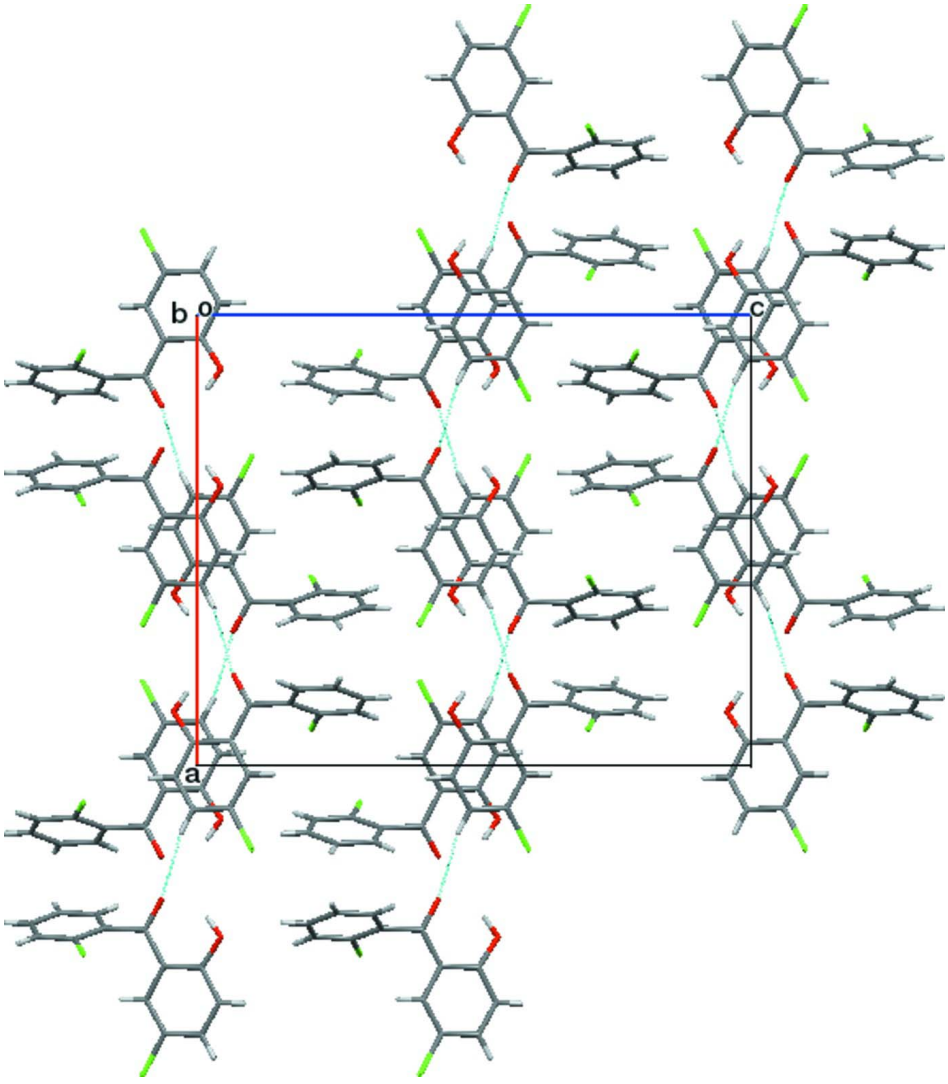


Figure 2

Packing diagram, viewed along the crystallographic *b* axis. Dotted lines represents C—H···O interactions.

4-Chloro-2-(2-chlorobenzoyl)phenol

Crystal data

$C_{13}H_8Cl_2O_2$
$M_r = 267.09$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 16.0231\ (4)$ Å
$b = 7.4216\ (2)$ Å
$c = 19.6843\ (5)$ Å
$V = 2340.80\ (10)$ Å^3
$Z = 8$

$F(000) = 1088$
$D_x = 1.516$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Cell parameters from 1972 reflections
$\theta = 4.5$–$64.9°$
$\mu = 4.87$ mm^{-1}
$T = 295$ K
Needle, yellow
0.20 × 0.19 × 0.18 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 10.7 pixels mm^{-1}
\φ and \ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)

T_{min} = 0.442, T_{max} = 0.474
15868 measured reflections
1972 independent reflections
1712 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 64.9°, θ_{min} = 4.5°
h = −18→18
k = −8→4
l = −23→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.105
S = 1.06
1972 reflections
154 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
$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2 + 0.4839P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl7	0.41862 (4)	0.46830 (6)	0.29403 (2)	0.0481 (2)
Cl17	0.68664 (3)	0.11456 (7)	0.40130 (3)	0.0439 (2)
O9	0.29828 (9)	0.2266 (2)	0.43513 (7)	0.0486 (5)
O16	0.38062 (10)	0.3221 (2)	0.54298 (7)	0.0428 (5)
C1	0.37955 (13)	0.1912 (2)	0.21207 (9)	0.0330 (5)
C2	0.35120 (13)	0.0191 (2)	0.19888 (9)	0.0341 (5)
C3	0.32654 (14)	−0.0927 (2)	0.25143 (10)	0.0376 (6)
C4	0.33080 (13)	−0.0318 (2)	0.31807 (9)	0.0356 (6)
C5	0.36117 (12)	0.1391 (2)	0.33254 (9)	0.0289 (5)
C6	0.38485 (12)	0.2493 (2)	0.27869 (9)	0.0296 (5)
C8	0.36471 (13)	0.1988 (2)	0.40532 (9)	0.0317 (5)
C10	0.44516 (12)	0.2149 (2)	0.43988 (9)	0.0288 (5)
C11	0.44873 (12)	0.2764 (2)	0.50758 (9)	0.0310 (5)
C12	0.52597 (14)	0.2929 (2)	0.53977 (9)	0.0390 (6)
C13	0.59771 (13)	0.2463 (3)	0.50739 (10)	0.0373 (5)
C14	0.59454 (12)	0.1799 (2)	0.44109 (10)	0.0324 (5)

C15	0.52003 (12)	0.1657 (2)	0.40762 (9)	0.0289 (5)
H1	0.39490	0.26710	0.17660	0.0400*
H2	0.34870	−0.02210	0.15430	0.0410*
H3	0.30720	−0.20820	0.24220	0.0450*
H4	0.31320	−0.10620	0.35330	0.0430*
H12	0.52840	0.33640	0.58400	0.0470*
H13	0.64880	0.25860	0.52940	0.0450*
H15	0.51890	0.12320	0.36320	0.0350*
H16	0.33910	0.30760	0.51930	0.0640*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl7	0.0670 (4)	0.0413 (3)	0.0359 (3)	−0.0138 (2)	−0.0087 (2)	0.0009 (2)
Cl17	0.0269 (3)	0.0563 (3)	0.0484 (3)	0.0061 (2)	−0.0024 (2)	0.0039 (2)
O9	0.0283 (8)	0.0882 (10)	0.0294 (7)	0.0028 (7)	0.0032 (7)	−0.0086 (7)
O16	0.0401 (9)	0.0647 (8)	0.0237 (7)	−0.0061 (7)	0.0043 (6)	−0.0069 (6)
C1	0.0312 (10)	0.0440 (9)	0.0239 (9)	0.0027 (8)	−0.0001 (8)	0.0026 (7)
C2	0.0311 (10)	0.0465 (9)	0.0247 (9)	0.0068 (8)	−0.0053 (8)	−0.0055 (7)
C3	0.0368 (11)	0.0413 (9)	0.0347 (10)	−0.0027 (8)	−0.0084 (10)	−0.0037 (7)
C4	0.0332 (10)	0.0454 (10)	0.0281 (9)	−0.0050 (8)	−0.0019 (9)	0.0052 (7)
C5	0.0208 (9)	0.0437 (8)	0.0222 (8)	0.0011 (7)	−0.0026 (8)	0.0000 (6)
C6	0.0264 (9)	0.0381 (8)	0.0244 (8)	0.0004 (7)	−0.0020 (8)	−0.0006 (7)
C8	0.0262 (10)	0.0449 (9)	0.0241 (9)	0.0002 (7)	0.0020 (8)	0.0012 (7)
C10	0.0295 (10)	0.0349 (8)	0.0220 (8)	−0.0020 (7)	−0.0023 (8)	0.0030 (6)
C11	0.0331 (11)	0.0382 (8)	0.0216 (8)	−0.0044 (7)	0.0024 (8)	0.0022 (6)
C12	0.0465 (13)	0.0476 (9)	0.0228 (9)	−0.0109 (9)	−0.0073 (9)	0.0013 (7)
C13	0.0336 (10)	0.0475 (9)	0.0307 (9)	−0.0070 (8)	−0.0104 (9)	0.0074 (7)
C14	0.0291 (10)	0.0359 (8)	0.0322 (9)	−0.0006 (7)	−0.0036 (9)	0.0066 (7)
C15	0.0291 (10)	0.0353 (8)	0.0224 (8)	0.0005 (7)	−0.0009 (8)	0.0013 (6)

Geometric parameters (Å, º)

Cl7—C6	1.7394 (16)	C10—C15	1.406 (3)
Cl17—C14	1.740 (2)	C10—C11	1.410 (2)
O9—C8	1.233 (2)	C11—C12	1.396 (3)
O16—C11	1.339 (2)	C12—C13	1.359 (3)
O16—H16	0.8200	C13—C14	1.396 (3)
C1—C6	1.383 (2)	C14—C15	1.368 (3)
C1—C2	1.380 (2)	C1—H1	0.9300
C2—C3	1.384 (3)	C2—H2	0.9300
C3—C4	1.389 (3)	C3—H3	0.9300
C4—C5	1.388 (2)	C4—H4	0.9300
C5—C6	1.392 (2)	C12—H12	0.9300
C5—C8	1.501 (2)	C13—H13	0.9300
C8—C10	1.462 (3)	C15—H15	0.9300
C11—O16—H16	109.00	C11—C12—C13	120.98 (17)
C2—C1—C6	119.15 (16)	C12—C13—C14	119.83 (19)
C1—C2—C3	120.55 (16)	Cl17—C14—C13	119.24 (15)

C2—C3—C4	119.79 (15)	C13—C14—C15	120.62 (18)
C3—C4—C5	120.55 (16)	Cl17—C14—C15	120.14 (15)
C4—C5—C8	118.62 (15)	C10—C15—C14	120.49 (17)
C6—C5—C8	122.91 (14)	C2—C1—H1	120.00
C4—C5—C6	118.45 (16)	C6—C1—H1	120.00
Cl7—C6—C5	120.12 (13)	C1—C2—H2	120.00
C1—C6—C5	121.49 (15)	C3—C2—H2	120.00
Cl7—C6—C1	118.36 (13)	C2—C3—H3	120.00
O9—C8—C10	121.73 (16)	C4—C3—H3	120.00
C5—C8—C10	120.10 (17)	C3—C4—H4	120.00
O9—C8—C5	118.11 (18)	C5—C4—H4	120.00
C8—C10—C11	120.13 (17)	C11—C12—H12	120.00
C8—C10—C15	121.39 (16)	C13—C12—H12	119.00
C11—C10—C15	118.46 (17)	C12—C13—H13	120.00
O16—C11—C10	122.76 (17)	C14—C13—H13	120.00
O16—C11—C12	117.67 (16)	C10—C15—H15	120.00
C10—C11—C12	119.57 (17)	C14—C15—H15	120.00

C6—C1—C2—C3	1.5 (3)	O9—C8—C10—C15	173.55 (16)
C2—C1—C6—Cl7	−178.65 (16)	C5—C8—C10—C11	178.00 (14)
C2—C1—C6—C5	−0.9 (3)	C5—C8—C10—C15	−3.7 (2)
C1—C2—C3—C4	−0.4 (3)	C8—C10—C11—O16	0.0 (2)
C2—C3—C4—C5	−1.3 (3)	C8—C10—C11—C12	−179.36 (14)
C3—C4—C5—C6	1.8 (3)	C15—C10—C11—O16	−178.36 (15)
C3—C4—C5—C8	−179.90 (19)	C15—C10—C11—C12	2.3 (2)
C4—C5—C6—Cl7	176.96 (15)	C8—C10—C15—C14	−179.27 (15)
C4—C5—C6—C1	−0.7 (3)	C11—C10—C15—C14	−0.9 (2)
C8—C5—C6—Cl7	−1.2 (3)	O16—C11—C12—C13	178.94 (17)
C8—C5—C6—C1	−178.93 (18)	C10—C11—C12—C13	−1.7 (2)
C4—C5—C8—O9	−69.0 (2)	C11—C12—C13—C14	−0.4 (3)
C4—C5—C8—C10	108.3 (2)	C12—C13—C14—Cl17	−178.09 (14)
C6—C5—C8—O9	109.2 (2)	C12—C13—C14—C15	1.8 (3)
C6—C5—C8—C10	−73.5 (2)	Cl17—C14—C15—C10	178.75 (12)
O9—C8—C10—C11	−4.8 (2)	C13—C14—C15—C10	−1.1 (2)

Hydrogen-bond geometry (Å, º)

Cg1 is the centroid of the C1–C6 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O16—H16···O9	0.82	1.88	2.598 (2)	146
C13—H13···O9^{i}	0.93	2.50	3.413 (3)	168
C6—Cl7···*Cg*1^{ii}	1.74 (1)	3.89 (1)	4.901 (2)	116 (1)

Symmetry codes: (i) *x*+1/2, −*y*+1/2, −*z*+1; (ii) −*x*, *y*+1/2, −*z*+3/2.