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2,3-Dibromo-1,3-bis(4-chlorophenyl)-propan-1-one

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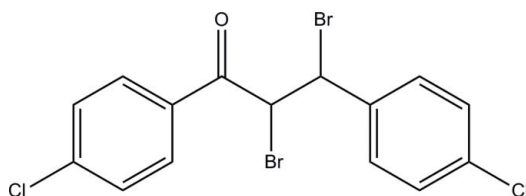
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.053; wR factor = 0.130; data-to-parameter ratio = 22.7.

In the title compound, $\text{C}_{15}\text{H}_{10}\text{Br}_2\text{Cl}_2\text{O}$, the terminal benzene rings make a dihedral angle of $31.1(2)^\circ$ with each other. In the crystal, molecules are stacked along the a axis and consolidated by $\text{C}-\text{H}\cdots\pi$ interactions. Short $\text{Cl}\cdots\text{Cl}$ [3.1140 (17) Å] and $\text{Br}\cdots\text{Cl}$ [3.4565 (13) Å] contacts are observed.

Related literature

For general background to and the biological activity of chalcones, see: Dimmock *et al.* (1999); Opletalova & Sedivy (1999); Nowakowska (2007). For the preparation of the title compound, see: Rai *et al.* (2008). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986). For bond-length data, see: Allen *et al.* (1987). For a related structure, see: Fun *et al.* (2010).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{10}\text{Br}_2\text{Cl}_2\text{O}$
 $M_r = 436.95$
Orthorhombic, $Pbca$
 $a = 5.7599(3)$ Å

$b = 17.1233(8)$ Å
 $c = 30.1983(13)$ Å
 $V = 2978.4(2)$ Å³
 $Z = 8$

Mo $K\alpha$ radiation
 $\mu = 5.79$ mm⁻¹

$T = 100$ K
 $0.52 \times 0.48 \times 0.34$ mm

Data collection

Bruker SMART APEXII CCD
area-detector diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.154$, $T_{\max} = 0.241$

16656 measured reflections
4100 independent reflections
3459 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.039$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.130$
 $S = 1.12$
4100 reflections

181 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.58$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.62$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of C1–C6 ring.

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C11}-\text{H11A}\cdots\text{Cg1}^{\text{I}}$	0.93	2.96	3.638 (5)	131

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2626).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2009). *APEX2*, *SAINTE* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.
Dimmock, J. R., Elias, D. W., Beazely, M. A. & Kandepu, N. M. (1999). *Curr. Med. Chem.* **6**, 1125–1149.
Fun, H.-K., Quah, C. K., Shetty, S. & Kalluraya, B. (2010). *Acta Cryst.* **E66**, o3128.
Nowakowska, Z. (2007). *Eur. J. Med. Chem.* **42**, 125–137.
Opletalova, V. & Sedivy, D. (1999). *Ceska Slov. Farm.* **48**, 252–255.
Rai, N. S., Kalluraya, B., Lingappa, B., Shenoy, S. & Puranic, V. G. (2008). *Eur. J. Med. Chem.* **43**, 1715–1720.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

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2,3-Dibromo-1,3-bis(4-chlorophenyl)propan-1-one**Hoong-Kun Fun, Ching Kheng Quah, Shobhitha Shetty and Balakrishna Kalluraya****S1. Comment**

Chalcones are well known for their biological activities (Dimmock *et al.*, 1999). These have been reported as potential antifungal, chemotherapeutic (Opletalova & Sedivy, 1999), anti-infective & anti-inflammatory agents (Nowakowska, 2007). Chalcones are prepared by the condensation of acetophenone with appropriately substituted aromatic aldehydes in ethanol medium employing sodium hydroxide as catalyst. Bromination of these propenones were carried out using bromine in glacial acetic acid medium to give dibromopropanones (Rai *et al.*, 2008). In view of the importance of chalcones, the synthesis and crystal structure of the title compound has been carried out.

In the title molecule, Fig. 1, the benzene rings make a dihedral angle of 31.1 (2) ° with each other. There are short Cl1...Cl1 [symmetry code: $-x, -y, -z$; distance = 3.1140 (17) Å] and Br1...Cl1 [symmetry code: $-1/2-x, 1/2+y, z$; distance = 3.4565 (13) Å] contacts which are shorter than the sum of van der Waals radii of chlorine/chlorine and chlorine/bromine atoms. Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and comparable to a related structure (Fun *et al.*, 2010).

In the crystal packing, Fig. 2, the molecules are stacked down the *a* axis and consolidated by C11—H11A...Cg1 (Table 1) interactions, where Cg1 is the centroid of the C1–C6 benzene ring. There is no significant hydrogen bond observed in this compound.

S2. Experimental

1,3-Bis(*p*-chlorophenyl)prop-2-en-1-one (0.01 mol) was dissolved in glacial acetic acid (25 ml) by gentle warming. A solution of bromine in glacial acetic acid [30% (w/v)] was added to it with constant stirring till the yellow color of the bromine persisted. The reaction mixture was kept aside at room temperature for overnight. Crystals of dibromopropanones separated out were collected by filtration and washed with ethanol and dried. It was then recrystallized from ethanol. Crystals suitable for X-ray analysis were obtained from ethanol by slow evaporation.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 or 0.98 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The highest residual electron density peak is located at 1.07 Å from Br2 and the deepest hole is located at 1.79 Å from H11A.

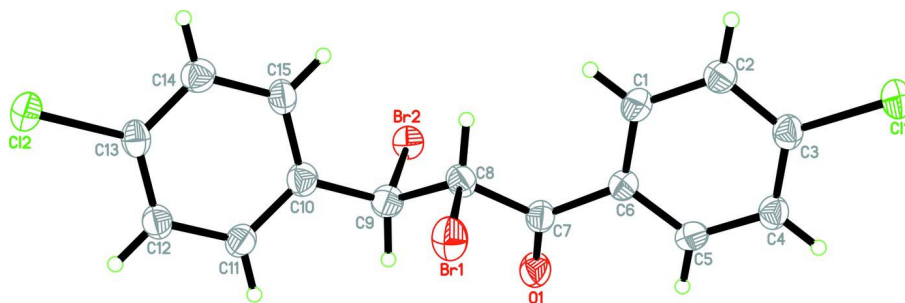


Figure 1

The molecular structure of the title compound showing 50% probability displacement ellipsoids for non-H atoms and the atom-numbering scheme.

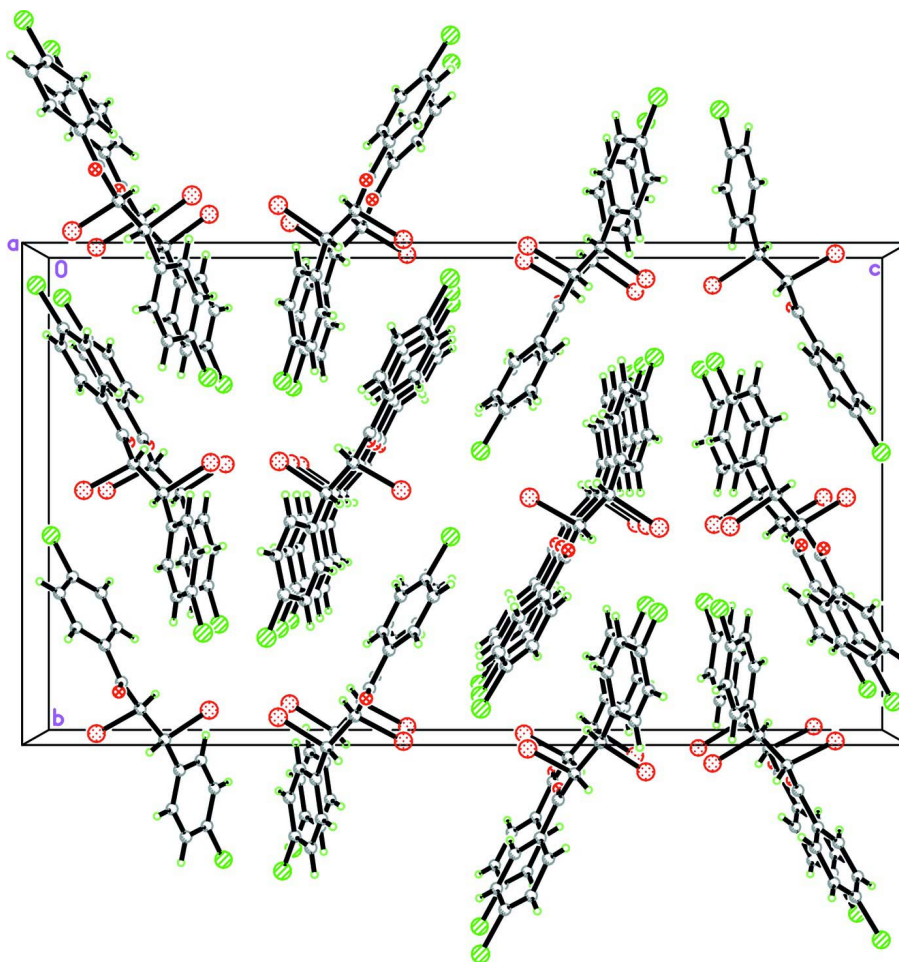


Figure 2

The crystal structure of the title compound, viewed along the *a* axis.

2,3-Dibromo-1,3-bis(4-chlorophenyl)propan-1-one

Crystal data

C₁₅H₁₀Br₂Cl₂O $M_r = 436.95$ Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

 $a = 5.7599$ (3) Å $b = 17.1233$ (8) Å $c = 30.1983$ (13) Å $V = 2978.4$ (2) Å³ $Z = 8$ $F(000) = 1696$ $D_x = 1.949$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 7210 reflections

 $\theta = 2.5$ – 29.4° $\mu = 5.79$ mm⁻¹ $T = 100$ K

Block, light yellow

 $0.52 \times 0.48 \times 0.34$ mm

Data collection

Bruker SMART APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2009)

 $T_{\min} = 0.154$, $T_{\max} = 0.241$

16656 measured reflections

4100 independent reflections

3459 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.039$ $\theta_{\max} = 29.4^\circ$, $\theta_{\min} = 2.4^\circ$ $h = -7 \rightarrow 6$ $k = -23 \rightarrow 22$ $l = -41 \rightarrow 41$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.053$ $wR(F^2) = 0.130$ $S = 1.12$

4100 reflections

181 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0496P)^2 + 11.6475P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 1.58$ e Å⁻³ $\Delta\rho_{\min} = -0.62$ e Å⁻³

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cyrosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.**Geometry.** 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.**Refinement.** Refinement of F^2 against ALL reflections. The weighted R-factor wR 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.Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	-0.01792 (9)	0.49293 (3)	0.070047 (16)	0.03500 (14)
Br2	0.29597 (8)	0.43618 (3)	0.207223 (14)	0.02966 (13)
Cl1	-0.0272 (2)	0.08541 (7)	0.01691 (4)	0.0361 (3)

C12	-0.3932 (2)	0.76965 (7)	0.21154 (4)	0.0309 (2)
O1	0.4810 (6)	0.3947 (2)	0.10860 (12)	0.0343 (8)
C1	-0.0300 (8)	0.2749 (3)	0.09337 (15)	0.0274 (9)
H1A	-0.1334	0.3006	0.1120	0.033*
C2	-0.0933 (8)	0.2049 (3)	0.07360 (15)	0.0279 (9)
H2A	-0.2362	0.1821	0.0798	0.034*
C3	0.0597 (8)	0.1696 (3)	0.04456 (15)	0.0281 (9)
C4	0.2787 (8)	0.2004 (3)	0.03566 (15)	0.0277 (9)
H4A	0.3794	0.1756	0.0161	0.033*
C5	0.3415 (8)	0.2684 (3)	0.05661 (14)	0.0267 (9)
H5A	0.4885	0.2891	0.0517	0.032*
C6	0.1892 (8)	0.3070 (3)	0.08517 (14)	0.0248 (8)
C7	0.2737 (8)	0.3804 (3)	0.10644 (15)	0.0275 (9)
C8	0.0953 (8)	0.4392 (3)	0.12362 (15)	0.0283 (9)
H8A	-0.0327	0.4120	0.1384	0.034*
C9	0.2020 (8)	0.4987 (3)	0.15432 (15)	0.0298 (9)
H9A	0.3422	0.5197	0.1403	0.036*
C10	0.0499 (8)	0.5659 (3)	0.16830 (15)	0.0292 (9)
C11	0.1231 (8)	0.6422 (3)	0.16112 (15)	0.0292 (9)
H11A	0.2644	0.6513	0.1471	0.035*
C12	-0.0117 (8)	0.7051 (3)	0.17457 (15)	0.0287 (9)
H12A	0.0393	0.7559	0.1698	0.034*
C13	-0.2224 (8)	0.6915 (3)	0.19510 (14)	0.0270 (9)
C14	-0.2990 (8)	0.6163 (3)	0.20252 (15)	0.0297 (9)
H14A	-0.4406	0.6077	0.2165	0.036*
C15	-0.1632 (8)	0.5529 (3)	0.18901 (16)	0.0299 (9)
H15A	-0.2148	0.5021	0.1938	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0440 (3)	0.0307 (2)	0.0303 (2)	0.0071 (2)	-0.0066 (2)	0.00027 (18)
Br2	0.0316 (2)	0.0294 (2)	0.0280 (2)	0.00218 (17)	-0.00372 (17)	0.00071 (17)
C11	0.0440 (6)	0.0255 (5)	0.0389 (6)	-0.0021 (5)	0.0046 (5)	-0.0068 (4)
C12	0.0337 (6)	0.0292 (5)	0.0298 (5)	0.0063 (4)	0.0013 (4)	-0.0044 (4)
O1	0.0270 (16)	0.0321 (18)	0.0438 (19)	0.0008 (13)	0.0030 (14)	-0.0076 (14)
C1	0.026 (2)	0.029 (2)	0.028 (2)	0.0040 (17)	0.0015 (17)	-0.0029 (16)
C2	0.026 (2)	0.027 (2)	0.030 (2)	0.0007 (17)	0.0002 (18)	0.0005 (17)
C3	0.036 (2)	0.023 (2)	0.025 (2)	0.0025 (18)	-0.0001 (18)	-0.0002 (16)
C4	0.033 (2)	0.024 (2)	0.027 (2)	0.0034 (18)	0.0016 (18)	-0.0005 (16)
C5	0.026 (2)	0.029 (2)	0.0254 (19)	0.0017 (17)	0.0035 (17)	0.0029 (16)
C6	0.025 (2)	0.026 (2)	0.0229 (18)	0.0006 (16)	0.0006 (16)	-0.0011 (16)
C7	0.031 (2)	0.024 (2)	0.027 (2)	0.0023 (17)	0.0016 (18)	-0.0017 (16)
C8	0.030 (2)	0.022 (2)	0.032 (2)	-0.0006 (17)	0.0004 (18)	0.0007 (17)
C9	0.028 (2)	0.031 (2)	0.031 (2)	-0.0003 (18)	-0.0003 (18)	-0.0021 (18)
C10	0.029 (2)	0.028 (2)	0.030 (2)	-0.0005 (18)	-0.0007 (18)	-0.0037 (17)
C11	0.026 (2)	0.030 (2)	0.031 (2)	0.0004 (18)	0.0027 (18)	-0.0051 (18)
C12	0.033 (2)	0.024 (2)	0.029 (2)	-0.0001 (18)	0.0002 (19)	-0.0018 (17)

C13	0.032 (2)	0.023 (2)	0.0254 (19)	0.0044 (17)	-0.0053 (18)	-0.0014 (16)
C14	0.026 (2)	0.032 (2)	0.032 (2)	0.0006 (18)	0.0003 (18)	-0.0002 (18)
C15	0.031 (2)	0.022 (2)	0.037 (2)	0.0021 (17)	0.0011 (19)	-0.0038 (17)

Geometric parameters (Å, °)

Br1—C8	1.972 (5)	C7—C8	1.529 (6)
Br2—C9	1.997 (5)	C8—C9	1.508 (6)
C11—C3	1.740 (5)	C8—H8A	0.9800
C12—C13	1.733 (5)	C9—C10	1.507 (6)
O1—C7	1.221 (6)	C9—H9A	0.9800
C1—C2	1.389 (6)	C10—C11	1.390 (7)
C1—C6	1.399 (6)	C10—C15	1.395 (7)
C1—H1A	0.9300	C11—C12	1.389 (6)
C2—C3	1.382 (6)	C11—H11A	0.9300
C2—H2A	0.9300	C12—C13	1.382 (7)
C3—C4	1.393 (7)	C12—H12A	0.9300
C4—C5	1.374 (6)	C13—C14	1.380 (6)
C4—H4A	0.9300	C14—C15	1.399 (6)
C5—C6	1.397 (6)	C14—H14A	0.9300
C5—H5A	0.9300	C15—H15A	0.9300
C6—C7	1.493 (6)		
C2—C1—C6	120.0 (4)	Br1—C8—H8A	110.3
C2—C1—H1A	120.0	C10—C9—C8	116.8 (4)
C6—C1—H1A	120.0	C10—C9—Br2	110.0 (3)
C3—C2—C1	118.9 (4)	C8—C9—Br2	103.9 (3)
C3—C2—H2A	120.6	C10—C9—H9A	108.6
C1—C2—H2A	120.6	C8—C9—H9A	108.6
C2—C3—C4	122.3 (4)	Br2—C9—H9A	108.6
C2—C3—C11	118.9 (4)	C11—C10—C15	119.1 (4)
C4—C3—C11	118.8 (3)	C11—C10—C9	119.9 (4)
C5—C4—C3	118.1 (4)	C15—C10—C9	121.0 (4)
C5—C4—H4A	121.0	C12—C11—C10	120.9 (4)
C3—C4—H4A	121.0	C12—C11—H11A	119.6
C4—C5—C6	121.3 (4)	C10—C11—H11A	119.6
C4—C5—H5A	119.3	C13—C12—C11	119.5 (4)
C6—C5—H5A	119.3	C13—C12—H12A	120.3
C5—C6—C1	119.4 (4)	C11—C12—H12A	120.3
C5—C6—C7	117.3 (4)	C14—C13—C12	120.7 (4)
C1—C6—C7	123.2 (4)	C14—C13—C12	119.5 (4)
O1—C7—C6	120.7 (4)	C12—C13—C12	119.8 (4)
O1—C7—C8	120.5 (4)	C13—C14—C15	119.9 (4)
C6—C7—C8	118.8 (4)	C13—C14—H14A	120.1
C9—C8—C7	112.3 (4)	C15—C14—H14A	120.1
C9—C8—Br1	108.9 (3)	C10—C15—C14	119.9 (4)
C7—C8—Br1	104.5 (3)	C10—C15—H15A	120.0
C9—C8—H8A	110.3	C14—C15—H15A	120.0

C7—C8—H8A	110.3		
C6—C1—C2—C3	-2.6 (7)	C7—C8—C9—C10	171.9 (4)
C1—C2—C3—C4	2.4 (7)	Br1—C8—C9—C10	56.6 (5)
C1—C2—C3—Cl1	-175.7 (3)	C7—C8—C9—Br2	-66.8 (4)
C2—C3—C4—C5	-0.3 (7)	Br1—C8—C9—Br2	178.0 (2)
Cl1—C3—C4—C5	177.8 (3)	C8—C9—C10—C11	-124.5 (5)
C3—C4—C5—C6	-1.6 (7)	Br2—C9—C10—C11	117.4 (4)
C4—C5—C6—C1	1.4 (7)	C8—C9—C10—C15	56.4 (6)
C4—C5—C6—C7	179.9 (4)	Br2—C9—C10—C15	-61.7 (5)
C2—C1—C6—C5	0.8 (7)	C15—C10—C11—C12	0.5 (7)
C2—C1—C6—C7	-177.6 (4)	C9—C10—C11—C12	-178.6 (4)
C5—C6—C7—O1	-19.2 (6)	C10—C11—C12—C13	-0.5 (7)
C1—C6—C7—O1	159.2 (5)	C11—C12—C13—C14	0.4 (7)
C5—C6—C7—C8	158.0 (4)	C11—C12—C13—Cl2	-179.4 (4)
C1—C6—C7—C8	-23.5 (6)	C12—C13—C14—C15	-0.3 (7)
O1—C7—C8—C9	-17.8 (6)	Cl2—C13—C14—C15	179.5 (4)
C6—C7—C8—C9	164.9 (4)	C11—C10—C15—C14	-0.5 (7)
O1—C7—C8—Br1	100.1 (5)	C9—C10—C15—C14	178.6 (4)
C6—C7—C8—Br1	-77.2 (4)	C13—C14—C15—C10	0.4 (7)

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of C1—C6 ring.

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C11—H11A \cdots Cg1 ⁱ	0.93	2.96	3.638 (5)	131

Symmetry code: (i) *x*, $-y-1/2$, $z-1/2$.