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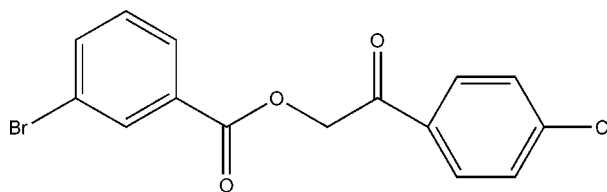
Crystal structure of 2-(4-chlorophenyl)-2-oxoethyl 3-bromobenzoate

Imtiaz Khan,^{a,b} Aliya Ibrar,^a Shahid Hameed,^a Jonathan M. White^c and Jim
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2-(4-Chlorophenyl)-2-oxoethyl 3-bromobenzoate, C₁₅H₁₀BrClO₃, was synthesized in a single-step reaction by condensation of 3-bromobenzoic acid with 2-bromo-1-(4-chlorophenyl)ethanone in dimethylformamide in the presence of triethylamine as a catalyst. The structure consists of an aryl ketone moiety linked to an aryl ester unit by a methylene group. Both units are reasonably planar (r.m.s. deviations of 0.119 and 0.010 Å for the aryl ketone and aryl ester units, respectively) and are almost orthogonal, with an angle of 88.60 (3)° between them. In the crystal, molecules form five separate sets of inversion dimers. Three of these are generated by two C—H...O interactions and a C—H...Br contact, and form chains along *c* and along the *ab* cell diagonal. In addition, two inversion-related π - π stacking interactions between like aryl rings again form chains of molecules but in this instance along the *bc* diagonal. These contacts generate infinite layers of molecules parallel to (011) and stack the molecules along the *a*-axis direction.

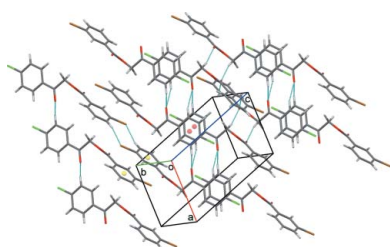
1. Chemical context

Keto esters, an important class of versatile intermediates, have been reported to show antitumor activity against Ehrlich cells and HeLa cells (Kinoshita & Umezawa, 1960). They also regulate the flowering times of some plants (Kai *et al.*, 2007). Recent studies have revealed that they also exhibit inhibitory activity against two isozymes of 11 β -hydroxysteroid dehydrogenases (11 β -HSD1 and 11 β -HSD2), which catalyse the interconversion of active cortisol and inactive cortisone (Zhang *et al.*, 2009). Dicarbonyl compounds and their derivatives are also among the most versatile and frequently employed synthons in organic synthesis, especially in heterocyclic chemistry (Stanovnik & Svete, 2004; Sheibani *et al.*, 2006*a,b*, 2007; Pal *et al.*, 2008). In this work, we report the synthesis of 2-(4-chlorophenyl)-2-oxoethyl 3-bromobenzoate, (1), which may be used as an effective synthon in organic chemistry.



2. Structural commentary

The structure of (1) consists of an aryl ketone moiety linked to an aryl ester unit by the C8 methylene group and both



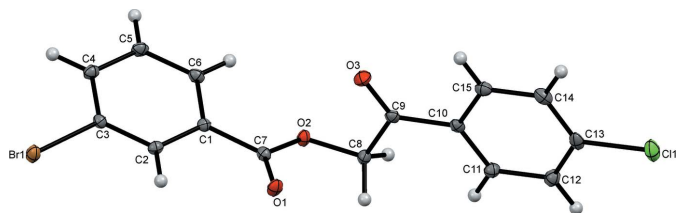


Figure 1
Fig. 1. The structure of (1) with displacement ellipsoids drawn at the 50% probability level.

groupings are reasonably planar. There is an r.m.s. deviation of 0.119 Å from the best-fit plane through atoms Br1, C1–C8, O1, O2 [maximum deviation 0.2477 (11) Å for O1] while the plane of the carboxylate unit subtends an angle of 15.5 (2)° to that of the bromobenzene ring. In addition, the plane of the aryl ketone unit C8–C15, O3, C11 has an r.m.s. deviation of 0.010 Å [maximum deviation 0.0171 (15) Å for C15]. The aryl ketone and aryl ester planes are almost orthogonal with an angle of 88.61 (3)° between them. Bond lengths and angles in the molecule are normal and are generally similar to those found

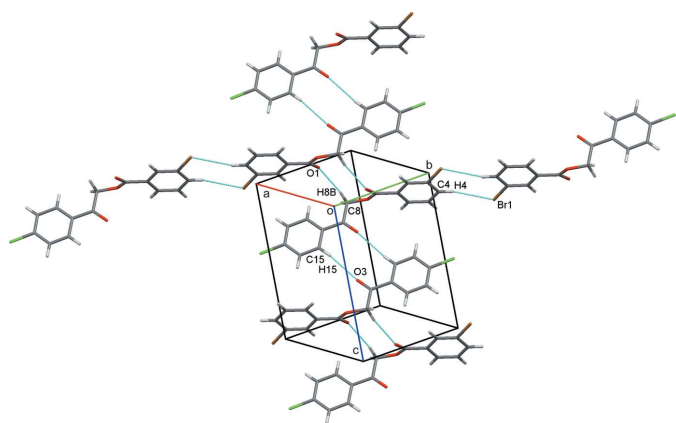


Figure 2
Chains of linked inversion dimers generated by C–H...O and C–H...Br hydrogen bonds, drawn as dashed lines.

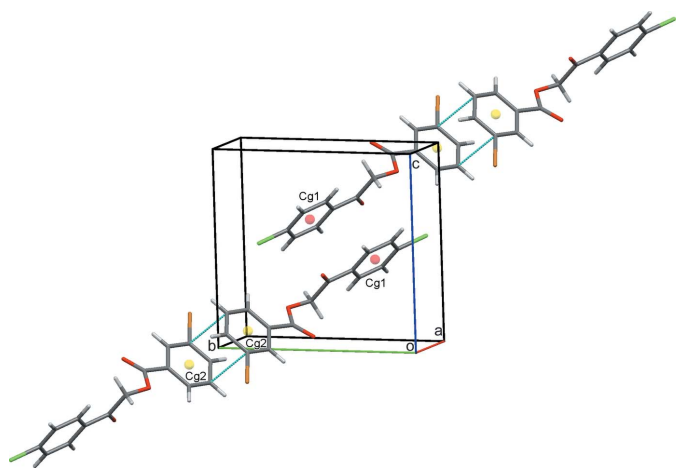


Figure 3
A chain of inversion dimers generated by π – π contacts, dotted green lines, between 3-bromophenyl and 4-chlorophenyl rings. Ring centroids are displayed as coloured spheres.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4–H4...Br1 ⁱ	0.95	2.97	3.8762 (18)	160
C8–H8B...O1 ⁱⁱ	0.99	2.42	3.396 (2)	168
C15–H15...O3 ⁱⁱⁱ	0.95	2.60	3.418 (2)	144

Symmetry codes: (i) $-x-1, -y+2, -z$; (ii) $-x+1, -y+1, -z$; (iii) $-x+1, -y+1, -z+1$.

in closely related molecules (see for example Fun *et al.*, 2011a; Chidan Kumar *et al.*, 2014c).

3. Supramolecular features

In the crystal structure, each molecule forms five separate inversion dimers. C8–H8B...O1 and C15–H15 O3 hydrogen bonds each generate $R_2^2(10)$ rings, forming zigzag chains along c . Additional C4–H4...Br1 contacts also form inversion dimers with $R_2^2(8)$ rings and these combine with the C8–H8B...O1 contacts to link alternating pairs of dimers into infinite chains approximately along the ab cell diagonal, Table 1, Fig. 2. Interestingly, infinite chains of alternating inversion dimers also result from a pair of π – π stacking interactions between adjacent 3-bromophenyl rings, $Cg1\cdots Cg1^{iv} = 3.6987$ (10) Å, and neighbouring 4-chlorophenyl rings $Cg2\cdots Cg2^v = 3.8585$ (11) Å, in this case along the bc diagonal, Fig. 3 [Cg1 and Cg2 are the centroids of the C1–C6 and C10–C15 rings, respectively; symmetry codes (iv) $-x, 2-y, -z$; (v) $2-x, 1-y, 1-z$]. These contacts combine to generate extended layers of molecules parallel to (011), Fig. 4, and to stack molecules along the a -axis direction, Fig. 5.

4. Database survey

A search of the Cambridge Crystallographic Database (Groom & Allen, 2014) reveals only eight structures with the 2-oxo-2-phenylethyl benzoate skeleton. These include the archetypal 2-oxo-2-phenylethyl benzoate (Fun *et al.*, 2011a), three additional 2-(4-chlorophenyl)-2-oxoethyl derivatives (Fun *et al.*, 2011b; Chidan Kumar *et al.*, 2014a,b) and the

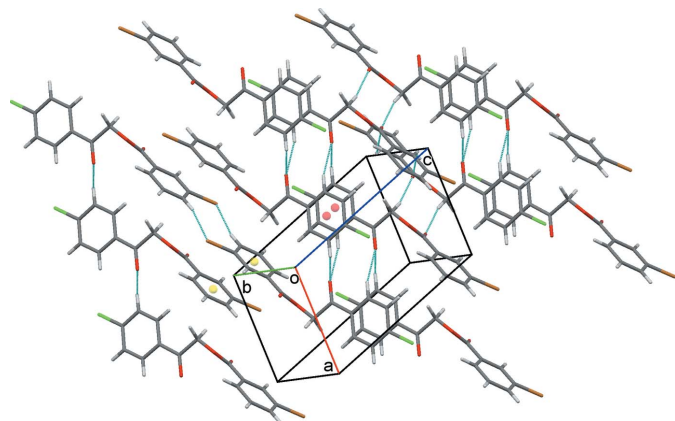


Figure 4
Overall packing of (1) viewed at right angles to (011).

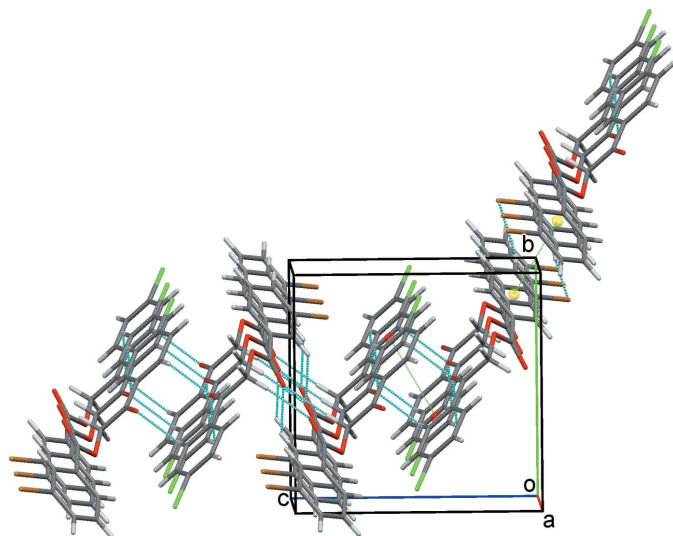


Figure 5
Overall packing of (1) viewed along the *a*-axis direction.

corresponding compound 2-(4-bromophenyl)-2-oxoethyl 3-chlorobenzoate with the chloro- and bromo-substituents reversed (Chidan Kumar *et al.*, 2014c). Interestingly, inversion-dimer formation is a feature of the packing in several of these structures.

5. Synthesis and crystallization

The preparation followed a procedure developed for the preparation of a related compound (Khan *et al.*, 2012). Triethylamine (4–5 drops) was added at room temperature to a stirred solution of 3-bromobenzoic acid (1.0 mmol) in *N,N*-dimethylformamide (DMF), followed by a solution of 2-bromo-1-(4-chlorophenyl)ethanone (1.0 mmol). The reaction mixture was stirred for 2 h. Progress of the reaction was monitored by TLC. After completion, the mixture was poured into water and the precipitated solid was filtered, dried and recrystallized (EtOAc/hexane) to afford 2-(4-chlorophenyl)-2-oxoethyl 3-bromobenzoate (1). The formation of keto ester (3) was indicated by the appearance of two typical stretching vibrations $\nu(\text{C}=\text{O})$ ester (1724) and $\nu(\text{C}=\text{O})$ keto (1698) cm^{-1} , respectively and the disappearance of characteristic IR stretching absorptions ascribable to the carboxylic acid group in the region of 3400–2400 cm^{-1} . In the ^1H NMR spectrum, the signals for the aromatic protons appeared in their respective regions and the disappearance of a characteristic signal for the COOH proton confirmed the formation of the title compound (1). The ^{13}C NMR spectrum displayed two characteristic signals for the keto and ester carbonyl carbon atoms at 190.7 and 165.3 p.p.m., respectively. Yield: 88%; m.p. 372–373 K; R_f : 0.72 (10% EtOAc/hexane); IR (ATR, cm^{-1}): 3089 ($\text{Csp}^2\text{-H}$), 2933, 2856 ($\text{Csp}^3\text{-H}$), 1724 ($\text{C}=\text{O}$ ester), 1698 ($\text{C}=\text{O}$ ketone), 1585, 1479 ($\text{C}=\text{C}$ Ar), 1225 ($\text{C}-\text{O}$); ^1H NMR (300 MHz, CDCl_3): δ 8.06–8.03 (*m*, 1H, Ar-H), 7.94–7.90 (*m*, 2H, Ar-H), 7.73–7.70 (*m*, 1H, Ar-H), 7.52–7.48 (*m*, 2H, Ar-H), 7.46–7.36 (*m*, 2H, Ar-H), 5.57 (*s*, 2H, CH_2); ^{13}C NMR

Table 2
Experimental details.

Crystal data	
Chemical formula	$\text{C}_{15}\text{H}_{10}\text{BrClO}_3$
M_r	353.59
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	130
a, b, c (\AA)	6.6797 (3), 10.0238 (4), 10.7851 (5)
α, β, γ ($^\circ$)	90.980 (4), 107.573 (4), 92.138 (3)
V (\AA^3)	687.64 (5)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.19
Crystal size (mm)	0.50 \times 0.40 \times 0.20
Data collection	
Diffractometer	Agilent SuperNova (Dual, Cu at zero, Atlas CCD)
Absorption correction	Multi-scan (<i>CrysAlis PRO</i> ; Agilent, 2011)
$T_{\text{min}}, T_{\text{max}}$	0.505, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10822, 3327, 3062
R_{int}	0.033
$(\sin \theta/\lambda)_{\text{max}}$ (\AA^{-1})	0.687
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.027, 0.064, 1.05
No. of reflections	3327
No. of parameters	181
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e \AA^{-3})	0.35, -0.60

Computer programs: *CrysAlis PRO*, (Agilent, 2011), *SHELXS97* and *SHELXL2014* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008), *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009) and *pubCIF* (Westrip, 2010).

(75 MHz, CDCl_3): δ 190.7, 165.3, 140.6, 134.5, 133.1, 132.4, 132.0, 131.0, 129.3, 129.3, 127.3, 122.1, 66.5.

6. Refinement

All H atoms were refined using a riding model, with $\text{C}-\text{H} = 0.95 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic, and $\text{C}-\text{H} = 0.99 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for the methylene H atoms.

Acknowledgements

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References

- Agilent (2011). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
- Allen, F. H., Johnson, O., Shields, G. P., Smith, B. R. & Towler, M. (2004). *J. Appl. Cryst.* **37**, 335–338.
- Chidan Kumar, C. S., Chia, T. S., Chandraju, S., Ooi, C. W., Quah, C. H. & Fun, H.-K. (2014c). *Z. Kristallogr.* **229**, 328–342.
- Chidan Kumar, C. S., Yohannan Panicker, C., Fun, H.-K., Sheena Mary, Y., Harikumar, B., Chandraju, S., Quah, C. H. & Ooi, C. W. (2014a). *Spectrochim. Acta Part A*, **126**, 208–219.
- Chidan Kumar, C. S., Yohannan Panicker, C., Fun, H.-K., Sheena Mary, Y., Harikumar, B., Chandraju, S., Quah, C. H. & Ooi, C. W. (2014b). *Spectrochim. Acta Part A*, **128**, 327–336.
- Fun, H.-K., Arshad, S., Garudachari, B., Isloor, A. M. & Satyanarayan, M. N. (2011a). *Acta Cryst.* **E67**, o1528.

- Fun, H.-K., Loh, W.-S., Garudachari, B., Isloor, A. M. & Satyanarayan, M. N. (2011*b*). *Acta Cryst.* **E67**, o1597.
- Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed. Engl.* **53**, 662–671.
- Kai, K., Yano, F., Suzuki, F., Kitagawa, H., Suzuki, M., Yokoyama, M. & Watanabe, N. (2007). *Tetrahedron*, **63**, 10630–10636.
- Khan, I., Ibrar, A., Korzański, A. & Kubicki, M. (2012). *Acta Cryst.* **E68**, o3465.
- Kinoshita, M. & Umezawa, S. (1960). *Bull. Chem. Soc. Jpn*, **33**, 1075–1080.
- 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.
- Pal, S., Mareddy, J. & Devi, N. S. (2008). *J. Braz. Chem. Soc.* **19**, 1207–1214.
- Sheibani, H., Islami, M. R., Hassanpour, A. & Hosseininasab, F. A. (2006*b*). *ARKIVOC*, **15**, 68–75.
- Sheibani, H., Islami, M. R., Hassanpour, A. & Saidi, K. (2007). *Phosphorus Sulfur Silicon Relat. Elem.* **183**, 13–20.
- Sheibani, H., Mosslemin, M. H., Behzadi, S., Islami, M. R. & Saidi, K. (2006*a*). *Synthesis*, **3**, 435–439.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Stanovnik, B. & Svete, J. (2004). *Chem. Rev.* **104**, 2433–2480.
- Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
- Zhang, L., Shen, Y., Zhu, H. J., Wang, F., Leng, Y. & Liu, J. K. (2009). *J. Antibiot.* **62**, 239–242.

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Crystal structure of 2-(4-chlorophenyl)-2-oxoethyl 3-bromobenzoate

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Computing details

Data collection: *CrysAlis PRO*, (Agilent, 2011); cell refinement: *CrysAlis PRO*, (Agilent, 2011); data reduction: *CrysAlis PRO*, (Agilent, 2011); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2008); molecular graphics: *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXL2014* (Sheldrick, 2008), *enCIFer* (Allen *et al.*, 2004), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

2-(4-Chlorophenyl)-2-oxoethyl 3-bromobenzoate

Crystal data

$C_{15}H_{10}BrClO_3$

$M_r = 353.59$

Triclinic, $P\bar{1}$

$a = 6.6797$ (3) Å

$b = 10.0238$ (4) Å

$c = 10.7851$ (5) Å

$\alpha = 90.980$ (4)°

$\beta = 107.573$ (4)°

$\gamma = 92.138$ (3)°

$V = 687.64$ (5) Å³

$Z = 2$

$F(000) = 352$

$D_x = 1.708$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 6648 reflections

$\theta = 3.2$ – 29.2 °

$\mu = 3.19$ mm⁻¹

$T = 130$ K

Block, colourless

$0.50 \times 0.40 \times 0.20$ mm

Data collection

Agilent SuperNova (Dual, Cu at zero, Atlas CCD)

diffractometer

Radiation source: SuperNova (Mo) X-ray Source

Mirror monochromator

ω scans

Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)

$T_{\min} = 0.505$, $T_{\max} = 1.000$

10822 measured reflections

3327 independent reflections

3062 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 29.2$ °, $\theta_{\min} = 3.2$ °

$h = -8 \rightarrow 8$

$k = -13 \rightarrow 13$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.064$

$S = 1.05$

3327 reflections

181 parameters

0 restraints

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0307P)^2 + 0.1071P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.35$ e Å⁻³

$\Delta\rho_{\min} = -0.60$ e Å⁻³

Special details

Experimental. Absorption correction: CrysAlisPro, Agilent (2011), Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1452 (3)	0.76183 (16)	0.09834 (16)	0.0153 (3)
C2	-0.0463 (3)	0.73719 (17)	0.00240 (16)	0.0165 (3)
H2	-0.0671	0.6625	-0.0562	0.020*
C3	-0.2052 (3)	0.82374 (17)	-0.00564 (16)	0.0164 (3)
C4	-0.1816 (3)	0.93075 (18)	0.08011 (17)	0.0200 (4)
H4	-0.2944	0.9879	0.0739	0.024*
C5	0.0097 (3)	0.95371 (18)	0.17577 (17)	0.0213 (4)
H5	0.0280	1.0271	0.2356	0.026*
C6	0.1742 (3)	0.87044 (17)	0.18485 (17)	0.0189 (3)
H6	0.3057	0.8874	0.2496	0.023*
C7	0.3120 (3)	0.66531 (17)	0.10555 (16)	0.0169 (3)
C8	0.6639 (3)	0.62000 (17)	0.20718 (17)	0.0182 (3)
H8A	0.8031	0.6678	0.2388	0.022*
H8B	0.6573	0.5687	0.1266	0.022*
C9	0.6368 (3)	0.52539 (17)	0.30950 (16)	0.0176 (3)
C10	0.8001 (3)	0.42573 (17)	0.35889 (16)	0.0166 (3)
C11	0.9739 (3)	0.41710 (18)	0.31370 (17)	0.0201 (4)
H11	0.9919	0.4770	0.2502	0.024*
C12	1.1213 (3)	0.32135 (19)	0.36109 (18)	0.0231 (4)
H12	1.2400	0.3152	0.3305	0.028*
C13	1.0919 (3)	0.23586 (18)	0.45295 (18)	0.0229 (4)
C14	0.9204 (3)	0.24247 (19)	0.49972 (18)	0.0242 (4)
H14	0.9029	0.1821	0.5630	0.029*
C15	0.7757 (3)	0.33824 (19)	0.45280 (17)	0.0215 (4)
H15	0.6584	0.3446	0.4848	0.026*
O1	0.28187 (18)	0.55756 (12)	0.05094 (12)	0.0222 (3)
O2	0.50195 (18)	0.71409 (12)	0.17991 (12)	0.0186 (3)
O3	0.48450 (19)	0.53074 (14)	0.34811 (13)	0.0257 (3)
Cl1	1.27582 (8)	0.11559 (5)	0.51284 (5)	0.03372 (13)
Br1	-0.46435 (2)	0.79269 (2)	-0.13927 (2)	0.02456 (7)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0181 (8)	0.0132 (8)	0.0156 (8)	0.0008 (6)	0.0067 (7)	0.0013 (6)
C2	0.0189 (8)	0.0136 (8)	0.0178 (8)	-0.0008 (6)	0.0069 (7)	-0.0010 (6)
C3	0.0165 (8)	0.0160 (9)	0.0160 (8)	-0.0002 (6)	0.0039 (7)	0.0007 (6)

C4	0.0244 (9)	0.0160 (9)	0.0213 (9)	0.0033 (7)	0.0091 (7)	-0.0003 (7)
C5	0.0288 (9)	0.0157 (9)	0.0196 (9)	0.0016 (7)	0.0078 (8)	-0.0043 (7)
C6	0.0219 (8)	0.0167 (9)	0.0164 (8)	-0.0014 (7)	0.0035 (7)	-0.0012 (6)
C7	0.0166 (8)	0.0178 (9)	0.0159 (8)	-0.0010 (6)	0.0043 (7)	0.0009 (6)
C8	0.0159 (8)	0.0179 (9)	0.0206 (9)	0.0023 (6)	0.0049 (7)	-0.0008 (7)
C9	0.0181 (8)	0.0192 (9)	0.0142 (8)	-0.0007 (6)	0.0035 (7)	-0.0043 (6)
C10	0.0179 (8)	0.0154 (9)	0.0144 (8)	-0.0023 (6)	0.0021 (7)	-0.0035 (6)
C11	0.0217 (8)	0.0187 (9)	0.0204 (9)	0.0004 (7)	0.0071 (7)	-0.0001 (7)
C12	0.0187 (8)	0.0241 (10)	0.0256 (10)	0.0009 (7)	0.0055 (8)	-0.0029 (7)
C13	0.0258 (9)	0.0169 (9)	0.0188 (9)	0.0036 (7)	-0.0041 (7)	-0.0050 (7)
C14	0.0314 (10)	0.0206 (10)	0.0181 (9)	-0.0022 (7)	0.0037 (8)	0.0015 (7)
C15	0.0227 (9)	0.0238 (10)	0.0174 (9)	-0.0021 (7)	0.0058 (7)	-0.0024 (7)
O1	0.0204 (6)	0.0167 (7)	0.0272 (7)	0.0018 (5)	0.0043 (5)	-0.0065 (5)
O2	0.0154 (6)	0.0162 (6)	0.0218 (6)	0.0014 (5)	0.0019 (5)	-0.0017 (5)
O3	0.0224 (6)	0.0325 (8)	0.0262 (7)	0.0046 (5)	0.0127 (6)	0.0037 (6)
Cl1	0.0352 (3)	0.0251 (3)	0.0316 (3)	0.0110 (2)	-0.0049 (2)	-0.0012 (2)
Br1	0.01750 (10)	0.02591 (12)	0.02626 (12)	0.00457 (7)	0.00057 (8)	-0.00652 (8)

Geometric parameters (Å, °)

C1—C6	1.391 (2)	C8—H8A	0.9900
C1—C2	1.391 (2)	C8—H8B	0.9900
C1—C7	1.488 (2)	C9—O3	1.212 (2)
C2—C3	1.380 (2)	C9—C10	1.490 (2)
C2—H2	0.9500	C10—C11	1.393 (2)
C3—C4	1.377 (2)	C10—C15	1.393 (2)
C3—Br1	1.8995 (16)	C11—C12	1.391 (2)
C4—C5	1.387 (3)	C11—H11	0.9500
C4—H4	0.9500	C12—C13	1.374 (3)
C5—C6	1.386 (2)	C12—H12	0.9500
C5—H5	0.9500	C13—C14	1.387 (3)
C6—H6	0.9500	C13—C11	1.7429 (18)
C7—O1	1.202 (2)	C14—C15	1.379 (3)
C7—O2	1.348 (2)	C14—H14	0.9500
C8—O2	1.4283 (19)	C15—H15	0.9500
C8—C9	1.515 (2)		
C6—C1—C2	120.59 (15)	C9—C8—H8B	109.7
C6—C1—C7	122.35 (15)	H8A—C8—H8B	108.2
C2—C1—C7	117.04 (15)	O3—C9—C10	121.67 (15)
C3—C2—C1	118.46 (16)	O3—C9—C8	120.25 (15)
C3—C2—H2	120.8	C10—C9—C8	118.08 (14)
C1—C2—H2	120.8	C11—C10—C15	119.46 (16)
C4—C3—C2	122.08 (16)	C11—C10—C9	122.21 (15)
C4—C3—Br1	119.44 (13)	C15—C10—C9	118.33 (15)
C2—C3—Br1	118.49 (13)	C12—C11—C10	120.38 (16)
C3—C4—C5	118.85 (16)	C12—C11—H11	119.8
C3—C4—H4	120.6	C10—C11—H11	119.8

C5—C4—H4	120.6	C13—C12—C11	118.76 (17)
C6—C5—C4	120.58 (17)	C13—C12—H12	120.6
C6—C5—H5	119.7	C11—C12—H12	120.6
C4—C5—H5	119.7	C12—C13—C14	122.01 (17)
C5—C6—C1	119.41 (16)	C12—C13—C11	119.05 (15)
C5—C6—H6	120.3	C14—C13—C11	118.94 (14)
C1—C6—H6	120.3	C15—C14—C13	118.85 (17)
O1—C7—O2	124.03 (15)	C15—C14—H14	120.6
O1—C7—C1	124.27 (15)	C13—C14—H14	120.6
O2—C7—C1	111.69 (15)	C14—C15—C10	120.54 (17)
O2—C8—C9	109.80 (13)	C14—C15—H15	119.7
O2—C8—H8A	109.7	C10—C15—H15	119.7
C9—C8—H8A	109.7	C7—O2—C8	114.81 (13)
O2—C8—H8B	109.7		
C6—C1—C2—C3	0.7 (2)	C8—C9—C10—C11	-0.3 (2)
C7—C1—C2—C3	179.07 (14)	O3—C9—C10—C15	-0.7 (3)
C1—C2—C3—C4	-1.8 (2)	C8—C9—C10—C15	-179.85 (16)
C1—C2—C3—Br1	178.47 (11)	C15—C10—C11—C12	0.5 (3)
C2—C3—C4—C5	1.4 (3)	C9—C10—C11—C12	-179.10 (16)
Br1—C3—C4—C5	-178.85 (12)	C10—C11—C12—C13	0.0 (3)
C3—C4—C5—C6	0.1 (3)	C11—C12—C13—C14	-0.1 (3)
C4—C5—C6—C1	-1.1 (3)	C11—C12—C13—C11	180.00 (14)
C2—C1—C6—C5	0.7 (2)	C12—C13—C14—C15	-0.3 (3)
C7—C1—C6—C5	-177.59 (15)	C11—C13—C14—C15	179.61 (14)
C6—C1—C7—O1	164.73 (17)	C13—C14—C15—C10	0.8 (3)
C2—C1—C7—O1	-13.6 (2)	C11—C10—C15—C14	-0.9 (3)
C6—C1—C7—O2	-15.9 (2)	C9—C10—C15—C14	178.71 (16)
C2—C1—C7—O2	165.76 (13)	O1—C7—O2—C8	-9.5 (2)
O2—C8—C9—O3	5.4 (2)	C1—C7—O2—C8	171.15 (13)
O2—C8—C9—C10	-175.45 (14)	C9—C8—O2—C7	-75.86 (17)
O3—C9—C10—C11	178.89 (17)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C4—H4...Br1 ⁱ	0.95	2.97	3.8762 (18)	160
C8—H8B...O1 ⁱⁱ	0.99	2.42	3.396 (2)	168
C15—H15...O3 ⁱⁱⁱ	0.95	2.60	3.418 (2)	144

Symmetry codes: (i) $-x-1, -y+2, -z$; (ii) $-x+1, -y+1, -z$; (iii) $-x+1, -y+1, -z+1$.