

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-(4-Bromophenyl)-2-oxoethyl 4-chlorobenzoate

Hoong-Kun Fun,^{a*} Chin Sing Yeap,^{a§} B. Garudachari,^b
Arun M. Isloor^b and M. N. Satyanarayan^c^aX-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, ^bOrganic Chemistry Division, Department of Chemistry, National Institute of Technology-Karnataka, Surathkal, Mangalore 575 025, India, and ^cDepartment of Physics, National Institute of Technology-Karnataka, Surathkal, Mangalore 575 025, India

Correspondence e-mail: hkfun@usm.my

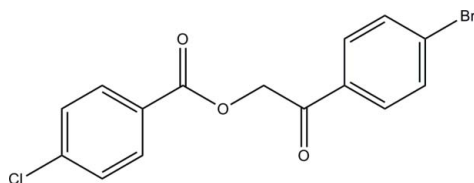
Received 26 May 2011; accepted 14 June 2011

Key indicators: single-crystal X-ray study; $T = 297$ K; mean $\sigma(\text{C}-\text{C}) = 0.008$ Å; R factor = 0.045; wR factor = 0.142; data-to-parameter ratio = 16.4.

The asymmetric unit of the title compound, $\text{C}_{15}\text{H}_{10}\text{BrClO}_3$, consists of three crystallographically independent molecules. The dihedral angles between the benzene rings in the three molecules are 68.8 (2), 0.7 (3) and 66.1 (2)°. In the crystal, the three independent molecules are interconnected by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, leading to isolated trimers.

Related literature

For background to phenacyl benzoate derivatives, see: Huang *et al.* (1996); Gandhi *et al.* (1995); Sheehan & Umezaw (1973); Ruzicka *et al.* (2002); Litera *et al.* (2006); Rather & Reid (1919). For related structures, see: Fun *et al.* (2011a,b,c). For the preparation, see: Kelly & Howard (1932).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{10}\text{BrClO}_3$
 $M_r = 353.59$
 Monoclinic, $P2_1$
 $a = 17.1061$ (11) Å
 $b = 5.3062$ (4) Å
 $c = 24.0376$ (16) Å
 $\beta = 101.502$ (1)°

$V = 2138.0$ (3) Å³
 $Z = 6$
 Mo $K\alpha$ radiation
 $\mu = 3.07$ mm⁻¹
 $T = 297$ K
 $0.41 \times 0.19 \times 0.15$ mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2009)
 $T_{\min} = 0.364$, $T_{\max} = 0.657$

18463 measured reflections
 8890 independent reflections
 5931 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.142$
 $S = 1.04$
 8890 reflections
 541 parameters
 1 restraint

H-atom parameters constrained
 $\Delta\rho_{\max} = 0.67$ e Å⁻³
 $\Delta\rho_{\min} = -0.54$ e Å⁻³
 Absolute structure: Flack (1983),
 3590 Friedel pairs
 Flack parameter: 0.065 (10)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C5A}-\text{H5AA}\cdots\text{O3B}$	0.93	2.59	3.227 (7)	126
$\text{C5B}-\text{H5BA}\cdots\text{O1A}$	0.93	2.48	3.217 (7)	136
$\text{C15B}-\text{H15B}\cdots\text{O1C}$	0.93	2.59	3.254 (7)	129

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; 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).

HKF and CSY thank Universiti Sains Malaysia for the Research University Grant 1001/PFIZIK/811160. AMI is thankful to the Department of Atomic Energy, Board for Research in Nuclear Sciences, Government of India, for the 'Young scientist' award. GB thanks the Department of Information Technology, New Delhi, India, for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2606).

References

- Bruker (2009). APEX2, SAINT and SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Fun, H.-K., Arshad, S., Garudachari, B., Isloor, A. M. & Satyanarayan, M. N. (2011a). *Acta Cryst.* **E67**, o1528.
- Fun, H.-K., Asik, S. I. J., Kumar, R., Isloor, A. M. & Shivananda, K. N. (2011b). *Acta Cryst.* **E67**, o1600.
- Fun, H.-K., Loh, W.-S., Garudachari, B., Isloor, A. M. & Satyanarayan, M. N. (2011c). *Acta Cryst.* **E67**, o1529.
- Gandhi, S. S., Bell, K. L. & Gibson, M. S. (1995). *Tetrahedron*, **51**, 13301–13308.
- Huang, W., Pian, J., Chen, B., Pei, W. & Ye, X. (1996). *Tetrahedron*, **52**, 10131–10136.
- Kelly, L. T. & Howard, H. W. (1932). *J. Am. Chem. Soc.* **54**, 4383–4385.
- Litera, J. K., Loya, A. D. & Klan, P. (2006). *J. Org. Chem.* **71**, 713–723.
- Rather, J. B. & Reid, E. (1919). *J. Am. Chem. Soc.* **41**, 75–83.
- Ruzicka, R., Zabada, M. & Klan, P. (2002). *Synth. Commun.* **32**, 2581–2590.
- Sheehan, J. C. & Umezaw, K. (1973). *J. Org. Chem.* **58**, 3771–3773.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

* Thomson Reuters ResearcherID: A-3561-2009.

§ Thomson Reuters ResearcherID: A-5523-2009.

supplementary materials

Acta Cryst. (2011). E67, o1723 [doi:10.1107/S1600536811022963]

2-(4-Bromophenyl)-2-oxoethyl 4-chlorobenzoate

H.-K. Fun, C. S. Yeap, B. Garudachari, A. M. Isloor and M. N. Satyanarayan

Comment

Phenacyl benzoates derivatives are very important in identification of organic acids (Rather & Reid, 1919). These compounds undergo photolysis in neutral and mild conditions (Sheehan & Umezaw, 1973; Ruzicka *et al.*, 2002; Litera *et al.*, 2006) and find applications in the field of synthetic chemistry for the synthesis of oxazoles, imidazoles (Huang *et al.*, 1996), benzoxazepine (Gandhi *et al.*, 1995). We hereby report the crystal structure of 2-(4-bromophenyl)-2-oxoethyl 4-chlorobenzoate of potential commercial importance.

The asymmetric unit of title compound consists of three crystallographically independent molecules, *A*, *B* and *C* (Fig. 1). The dihedral angles between the two terminal phenyl rings for each molecules being 68.8 (2), 0.7 (3) and 66.1 (2)°, respectively, show that molecule *A* and *C* are in similar conformation. The geometric parameters are comparable to those previously reported structures (Fun *et al.*, 2011*a,b,c*). The three independent molecules are interconnected by C5A—H5AA···O3B, C5B—H5BA···O1A and C15B—H15B···O1C hydrogen bonds (Fig. 1, Table 1). In the crystal structure, the molecules are arranged into columns (Fig. 2) parallel to the *b* axis.

Experimental

A mixture of 4-chlorobenzoic acid (1.0 g, 0.0063 mol) sodium carbonate (0.744 g, 0.0070 mol) and 2-bromo-1-phenylethanone (1.94 g, 0.0070 mol) in dimethylformamide (10 ml) was stirred at room temperature for 2 h. On cooling, colourless needle-shaped crystals of 2-(4-bromophenyl)-2-oxoethyl 4-chlorobenzoate separated. The compound was collected by filtration and re-crystallized from ethanol. Yield: 2.18 g, 96.88%, M. p.: 400–401 K (Kelly & Howard, 1983).

Refinement

All hydrogen atoms were positioned geometrically [$C-H = 0.93$ or 0.97 Å] and refined using a riding model, with $U_{iso}(H) = 1.2 U_{eq}(C)$. The presence of pseudosymmetry in the structure suggests the higher symmetry space group $P2_1/c$, but attempts to refine the structure in this space group resulted in much higher *R* and *wR* values and chemically incorrect structure. A total of 3590 Friedel pairs were used to determine the absolute structure.

Figures

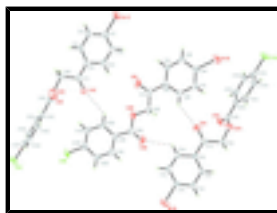


Fig. 1. The molecular structure of the title compound, with 30% probability ellipsoids for non-H atoms. Hydrogen bonds (dashed lines) are shown.

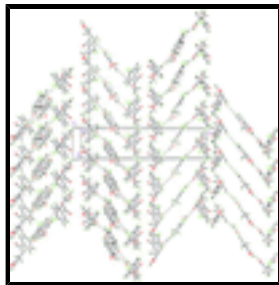


Fig. 2. Crystal structure of title compound, showing molecules arranged into columns parallel to the *b* axis. Hydrogen bonds (dashed lines) are shown.

2-(4-Bromophenyl)-2-oxoethyl 4-chlorobenzoate

Crystal data

$C_{15}H_{10}BrClO_3$

$M_r = 353.59$

Monoclinic, $P2_1$

Hall symbol: P 2yb

$a = 17.1061$ (11) Å

$b = 5.3062$ (4) Å

$c = 24.0376$ (16) Å

$\beta = 101.502$ (1)°

$V = 2138.0$ (3) Å³

$Z = 6$

$F(000) = 1056$

$D_x = 1.648$ Mg m⁻³

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

Cell parameters from 4899 reflections

$\theta = 2.3$ – 26.1 °

$\mu = 3.07$ mm⁻¹

$T = 297$ K

Block, colourless

$0.41 \times 0.19 \times 0.15$ mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2009)

$T_{\min} = 0.364$, $T_{\max} = 0.657$

18463 measured reflections

8890 independent reflections

5931 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.021$

$\theta_{\max} = 27.3$ °, $\theta_{\min} = 1.9$ °

$h = -22 \rightarrow 21$

$k = -6 \rightarrow 6$

$l = -31 \rightarrow 31$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.045$

$wR(F^2) = 0.142$

$S = 1.04$

8890 reflections

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.4681P]$

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

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

$\Delta\rho_{\max} = 0.67$ e Å⁻³

541 parameters

$$\Delta\rho_{\min} = -0.54 \text{ e } \text{\AA}^{-3}$$

1 restraint

Absolute structure: Flack (1983), 3590 Friedel pairs

Primary atom site location: structure-invariant direct methods

Flack parameter: 0.065 (10)

Special details

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.

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1A	0.67824 (3)	1.30681 (17)	0.56131 (3)	0.0834 (2)
Cl1A	-0.15600 (8)	0.7707 (5)	0.61298 (7)	0.0905 (6)
O1A	0.3290 (2)	0.9241 (10)	0.63191 (17)	0.0841 (16)
O2A	0.1988 (2)	1.1811 (8)	0.58442 (16)	0.0632 (10)
O3A	0.1936 (2)	0.8415 (10)	0.52826 (15)	0.0736 (12)
C1A	0.4376 (3)	1.3623 (11)	0.56106 (19)	0.0501 (12)
H1AA	0.3959	1.4676	0.5447	0.060*
C2A	0.5157 (3)	1.4119 (11)	0.55351 (19)	0.0526 (13)
H2AA	0.5265	1.5531	0.5334	0.063*
C3A	0.5759 (3)	1.2477 (12)	0.57638 (18)	0.0502 (13)
C4A	0.5622 (3)	1.0413 (15)	0.60918 (18)	0.0556 (12)
H4AA	0.6038	0.9348	0.6252	0.067*
C5A	0.4865 (3)	0.9992 (11)	0.61718 (18)	0.0503 (12)
H5AA	0.4771	0.8628	0.6392	0.060*
C6A	0.4229 (3)	1.1541 (10)	0.59333 (17)	0.0462 (12)
C7A	0.3413 (3)	1.0949 (12)	0.60142 (19)	0.0533 (15)
C8A	0.2734 (3)	1.2495 (13)	0.5706 (2)	0.0622 (15)
H8AA	0.2839	1.4259	0.5798	0.075*
H8AB	0.2698	1.2297	0.5300	0.075*
C9A	0.1655 (3)	0.9666 (12)	0.5602 (2)	0.0533 (15)
C10A	0.0870 (3)	0.9183 (11)	0.57661 (18)	0.0471 (12)
C11A	0.0551 (3)	1.0787 (13)	0.61134 (18)	0.0536 (13)
H11A	0.0844	1.2180	0.6271	0.064*
C12A	-0.0192 (3)	1.0367 (17)	0.6231 (2)	0.0652 (14)
H12A	-0.0405	1.1465	0.6464	0.078*
C13A	-0.0615 (3)	0.8289 (14)	0.5998 (2)	0.0604 (15)
C14A	-0.0303 (3)	0.6605 (12)	0.56479 (19)	0.0569 (14)
H14A	-0.0592	0.5200	0.5493	0.068*
C15A	0.0445 (3)	0.7093 (13)	0.55400 (19)	0.0548 (14)

supplementary materials

H15A	0.0665	0.5996	0.5312	0.066*
Br1B	0.06535 (3)	0.5564 (2)	0.73157 (2)	0.0802 (2)
Cl1B	0.92955 (6)	-0.0329 (3)	0.77621 (5)	0.0628 (4)
O1B	0.4359 (2)	0.0185 (11)	0.78999 (16)	0.0786 (11)
O2B	0.54903 (19)	0.2829 (8)	0.75497 (14)	0.0627 (10)
O3B	0.5851 (2)	0.5857 (10)	0.70158 (16)	0.0768 (12)
C1B	0.2738 (3)	0.1656 (11)	0.7806 (2)	0.0593 (14)
H1BA	0.2901	0.0219	0.8020	0.071*
C2B	0.1972 (3)	0.2425 (14)	0.7741 (2)	0.0666 (17)
H2BA	0.1620	0.1554	0.7920	0.080*
C3B	0.1714 (3)	0.4476 (12)	0.74136 (18)	0.0525 (14)
C4B	0.2241 (3)	0.5826 (13)	0.71591 (19)	0.0553 (14)
H4BA	0.2066	0.7235	0.6939	0.066*
C5B	0.3026 (3)	0.5066 (13)	0.72344 (19)	0.0552 (14)
H5BA	0.3382	0.5967	0.7065	0.066*
C6B	0.3285 (3)	0.2963 (12)	0.75623 (17)	0.0495 (12)
C7B	0.4133 (3)	0.2139 (12)	0.76452 (19)	0.0579 (14)
C8B	0.4699 (3)	0.3840 (13)	0.74273 (18)	0.0605 (16)
H8BA	0.4527	0.4044	0.7020	0.073*
H8BB	0.4697	0.5487	0.7602	0.073*
C9B	0.6007 (3)	0.4055 (12)	0.7307 (2)	0.0558 (14)
C10B	0.6816 (3)	0.2864 (12)	0.74456 (18)	0.0488 (11)
C11B	0.7416 (3)	0.3983 (13)	0.7210 (2)	0.0677 (16)
H11B	0.7300	0.5398	0.6980	0.081*
C12B	0.8182 (3)	0.2997 (15)	0.7317 (2)	0.0657 (15)
H12B	0.8586	0.3755	0.7168	0.079*
C13B	0.8332 (3)	0.0859 (12)	0.76505 (18)	0.0497 (12)
C14B	0.7754 (3)	-0.0248 (12)	0.78855 (18)	0.0545 (15)
H14B	0.7873	-0.1653	0.8118	0.065*
C15B	0.6984 (3)	0.0746 (13)	0.77740 (18)	0.0525 (13)
H15B	0.6584	-0.0031	0.7923	0.063*
Br1C	0.32100 (3)	-0.72787 (16)	0.94037 (3)	0.0849 (3)
Cl1C	1.15846 (7)	-0.2313 (4)	0.89129 (6)	0.0805 (5)
O1C	0.6700 (2)	-0.3571 (9)	0.86746 (17)	0.0800 (14)
O2C	0.7989 (2)	-0.6225 (9)	0.91351 (17)	0.0693 (12)
O3C	0.8057 (2)	-0.2890 (10)	0.97126 (16)	0.0758 (12)
C1C	0.5592 (3)	-0.7933 (11)	0.93756 (19)	0.0515 (13)
H1CA	0.5997	-0.9063	0.9520	0.062*
C2C	0.4832 (3)	-0.8341 (12)	0.9467 (2)	0.0552 (14)
H2CA	0.4729	-0.9727	0.9678	0.066*
C3C	0.4234 (3)	-0.6727 (13)	0.92500 (19)	0.0533 (13)
C4C	0.4372 (3)	-0.4662 (16)	0.89371 (19)	0.0611 (14)
H4CA	0.3958	-0.3569	0.8788	0.073*
C5C	0.5134 (3)	-0.4232 (13)	0.88469 (18)	0.0549 (13)
H5CA	0.5231	-0.2843	0.8635	0.066*
C6C	0.5757 (3)	-0.5854 (10)	0.90700 (18)	0.0450 (12)
C7C	0.6575 (3)	-0.5282 (11)	0.8977 (2)	0.0519 (14)
C8C	0.7228 (3)	-0.6899 (14)	0.9273 (2)	0.0642 (15)
H8CA	0.7264	-0.6749	0.9679	0.077*

H8CB	0.7108	-0.8643	0.9168	0.077*
C9C	0.8341 (3)	-0.4191 (13)	0.93900 (19)	0.0511 (13)
C10C	0.9124 (3)	-0.3701 (10)	0.92392 (18)	0.0482 (13)
C11C	0.9457 (3)	-0.5376 (11)	0.89043 (19)	0.0517 (13)
H11C	0.9170	-0.6783	0.8748	0.062*
C12C	1.0218 (3)	-0.4933 (14)	0.8805 (2)	0.0573 (14)
H12C	1.0445	-0.6050	0.8585	0.069*
C13C	1.0634 (3)	-0.2850 (12)	0.9034 (2)	0.0514 (13)
C14C	1.0313 (3)	-0.1212 (12)	0.9361 (2)	0.0566 (13)
H14C	1.0605	0.0188	0.9516	0.068*
C15C	0.9556 (3)	-0.1613 (13)	0.9465 (2)	0.0542 (13)
H15C	0.9339	-0.0477	0.9687	0.065*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1A	0.0459 (3)	0.1128 (7)	0.0917 (4)	-0.0160 (4)	0.0140 (3)	0.0005 (4)
Cl1A	0.0560 (7)	0.1171 (17)	0.1044 (11)	-0.0214 (11)	0.0302 (8)	0.0042 (12)
O1A	0.065 (2)	0.102 (4)	0.087 (3)	-0.003 (2)	0.019 (2)	0.047 (3)
O2A	0.0488 (19)	0.057 (3)	0.090 (2)	-0.005 (2)	0.0276 (18)	-0.009 (2)
O3A	0.065 (2)	0.082 (3)	0.082 (2)	0.003 (3)	0.0341 (19)	-0.015 (3)
C1A	0.050 (2)	0.043 (3)	0.057 (3)	0.000 (3)	0.008 (2)	0.003 (3)
C2A	0.058 (3)	0.045 (3)	0.057 (3)	-0.004 (3)	0.017 (2)	0.009 (2)
C3A	0.043 (2)	0.056 (4)	0.048 (2)	-0.009 (3)	0.003 (2)	-0.003 (3)
C4A	0.050 (2)	0.053 (3)	0.060 (3)	0.004 (4)	0.000 (2)	0.004 (3)
C5A	0.053 (3)	0.041 (3)	0.054 (2)	0.003 (3)	0.004 (2)	0.013 (3)
C6A	0.050 (3)	0.044 (3)	0.042 (2)	-0.004 (2)	0.004 (2)	0.004 (2)
C7A	0.052 (3)	0.058 (4)	0.049 (2)	-0.008 (3)	0.010 (2)	0.001 (3)
C8A	0.050 (3)	0.054 (4)	0.087 (3)	0.004 (3)	0.024 (3)	0.012 (3)
C9A	0.048 (3)	0.055 (4)	0.057 (3)	0.008 (3)	0.011 (2)	0.003 (3)
C10A	0.044 (2)	0.049 (3)	0.048 (2)	-0.003 (2)	0.007 (2)	0.002 (2)
C11A	0.053 (3)	0.048 (3)	0.062 (3)	-0.007 (3)	0.018 (2)	-0.009 (3)
C12A	0.062 (3)	0.069 (4)	0.071 (3)	0.000 (4)	0.029 (2)	-0.011 (4)
C13A	0.046 (3)	0.071 (4)	0.065 (3)	0.001 (3)	0.011 (2)	0.015 (3)
C14A	0.052 (3)	0.057 (4)	0.058 (3)	-0.014 (3)	0.001 (2)	0.000 (3)
C15A	0.060 (3)	0.055 (4)	0.050 (3)	0.010 (3)	0.012 (2)	-0.002 (3)
Br1B	0.0535 (3)	0.1015 (6)	0.0861 (4)	0.0141 (4)	0.0153 (3)	-0.0053 (4)
Cl1B	0.0359 (5)	0.0806 (12)	0.0704 (7)	0.0202 (6)	0.0067 (5)	-0.0083 (7)
O1B	0.072 (2)	0.070 (3)	0.091 (2)	0.030 (3)	0.0089 (19)	0.019 (3)
O2B	0.0516 (18)	0.073 (3)	0.0619 (19)	0.024 (2)	0.0083 (15)	0.008 (2)
O3B	0.073 (2)	0.072 (3)	0.085 (2)	0.028 (3)	0.0124 (19)	0.023 (3)
C1B	0.073 (3)	0.045 (3)	0.062 (3)	0.013 (3)	0.018 (3)	0.016 (3)
C2B	0.076 (4)	0.064 (4)	0.064 (3)	-0.008 (3)	0.026 (3)	0.016 (3)
C3B	0.044 (2)	0.068 (4)	0.046 (2)	0.003 (2)	0.0098 (19)	-0.006 (3)
C4B	0.053 (3)	0.049 (4)	0.062 (3)	0.012 (3)	0.009 (2)	0.015 (3)
C5B	0.044 (2)	0.058 (4)	0.062 (3)	0.007 (3)	0.008 (2)	0.015 (3)
C6B	0.055 (3)	0.050 (3)	0.040 (2)	0.012 (3)	0.0021 (19)	-0.002 (2)
C7B	0.068 (3)	0.051 (4)	0.048 (3)	0.029 (3)	-0.005 (2)	-0.001 (2)

supplementary materials

C8B	0.057 (3)	0.079 (4)	0.045 (2)	0.030 (3)	0.007 (2)	0.006 (2)
C9B	0.062 (3)	0.061 (4)	0.046 (2)	0.014 (3)	0.015 (2)	0.000 (3)
C10B	0.055 (2)	0.047 (3)	0.044 (2)	0.011 (3)	0.009 (2)	0.007 (2)
C11B	0.077 (3)	0.065 (4)	0.065 (3)	0.014 (3)	0.021 (3)	0.016 (3)
C12B	0.057 (3)	0.074 (4)	0.073 (3)	0.005 (3)	0.030 (2)	0.006 (3)
C13B	0.050 (2)	0.046 (3)	0.053 (2)	0.012 (3)	0.010 (2)	0.001 (3)
C14B	0.050 (3)	0.059 (4)	0.054 (2)	0.012 (3)	0.008 (2)	0.014 (3)
C15B	0.053 (3)	0.054 (4)	0.053 (2)	0.003 (3)	0.017 (2)	0.005 (3)
Br1C	0.0474 (3)	0.1115 (7)	0.0968 (4)	-0.0085 (4)	0.0169 (3)	-0.0075 (4)
Cl1C	0.0425 (6)	0.1100 (15)	0.0921 (9)	-0.0088 (9)	0.0205 (6)	0.0061 (10)
O1C	0.066 (2)	0.087 (4)	0.090 (3)	-0.011 (2)	0.022 (2)	0.036 (3)
O2C	0.055 (2)	0.064 (3)	0.096 (3)	-0.001 (2)	0.034 (2)	-0.004 (3)
O3C	0.065 (2)	0.086 (3)	0.082 (2)	-0.006 (2)	0.0283 (19)	-0.024 (3)
C1C	0.054 (3)	0.044 (3)	0.059 (3)	0.006 (3)	0.017 (2)	0.010 (3)
C2C	0.052 (3)	0.053 (4)	0.060 (3)	-0.007 (3)	0.012 (2)	0.007 (3)
C3C	0.047 (2)	0.057 (4)	0.057 (3)	-0.005 (3)	0.012 (2)	-0.010 (3)
C4C	0.052 (3)	0.064 (4)	0.062 (3)	0.009 (4)	-0.002 (2)	-0.001 (4)
C5C	0.064 (3)	0.048 (4)	0.051 (2)	-0.008 (3)	0.006 (2)	-0.002 (3)
C6C	0.052 (3)	0.038 (3)	0.046 (2)	-0.002 (2)	0.013 (2)	-0.005 (2)
C7C	0.057 (3)	0.046 (4)	0.056 (3)	-0.008 (3)	0.018 (2)	0.004 (3)
C8C	0.052 (3)	0.056 (4)	0.089 (4)	-0.012 (3)	0.024 (3)	-0.003 (3)
C9C	0.051 (3)	0.050 (4)	0.052 (2)	0.002 (3)	0.011 (2)	0.001 (3)
C10C	0.049 (3)	0.048 (4)	0.047 (2)	0.012 (2)	0.007 (2)	0.005 (2)
C11C	0.052 (3)	0.048 (4)	0.057 (2)	0.004 (3)	0.016 (2)	0.004 (2)
C12C	0.055 (3)	0.059 (4)	0.063 (3)	0.006 (3)	0.022 (2)	0.005 (3)
C13C	0.041 (2)	0.058 (4)	0.056 (3)	0.002 (3)	0.012 (2)	0.009 (3)
C14C	0.057 (3)	0.049 (3)	0.061 (3)	-0.004 (3)	0.004 (2)	0.001 (3)
C15C	0.057 (3)	0.049 (4)	0.057 (3)	0.000 (3)	0.012 (2)	-0.001 (3)

Geometric parameters (Å, °)

Br1A—C3A	1.884 (4)	C6B—C7B	1.488 (7)
Cl1A—C13A	1.735 (5)	C7B—C8B	1.493 (9)
O1A—C7A	1.210 (7)	C8B—H8BA	0.9700
O2A—C9A	1.351 (7)	C8B—H8BB	0.9700
O2A—C8A	1.428 (6)	C9B—C10B	1.497 (7)
O3A—C9A	1.187 (6)	C10B—C15B	1.370 (8)
C1A—C6A	1.401 (7)	C10B—C11B	1.401 (7)
C1A—C2A	1.409 (6)	C11B—C12B	1.386 (8)
C1A—H1AA	0.9300	C11B—H11B	0.9300
C2A—C3A	1.376 (7)	C12B—C13B	1.384 (9)
C2A—H2AA	0.9300	C12B—H12B	0.9300
C3A—C4A	1.396 (9)	C13B—C14B	1.365 (6)
C4A—C5A	1.366 (6)	C14B—C15B	1.395 (6)
C4A—H4AA	0.9300	C14B—H14B	0.9300
C5A—C6A	1.392 (7)	C15B—H15B	0.9300
C5A—H5AA	0.9300	Br1C—C3C	1.884 (4)
C6A—C7A	1.481 (6)	Cl1C—C13C	1.732 (5)
C7A—C8A	1.493 (7)	O1C—C7C	1.208 (6)

C8A—H8AA	0.9700	O2C—C9C	1.325 (8)
C8A—H8AB	0.9700	O2C—C8C	1.451 (5)
C9A—C10A	1.495 (6)	O3C—C9C	1.210 (6)
C10A—C15A	1.377 (8)	C1C—C2C	1.378 (6)
C10A—C11A	1.379 (7)	C1C—C6C	1.386 (7)
C11A—C12A	1.375 (6)	C1C—H1CA	0.9300
C11A—H11A	0.9300	C2C—C3C	1.356 (8)
C12A—C13A	1.375 (10)	C2C—H2CA	0.9300
C12A—H12A	0.9300	C3C—C4C	1.376 (10)
C13A—C14A	1.405 (8)	C4C—C5C	1.382 (6)
C14A—C15A	1.379 (7)	C4C—H4CA	0.9300
C14A—H14A	0.9300	C5C—C6C	1.392 (7)
C15A—H15A	0.9300	C5C—H5CA	0.9300
Br1B—C3B	1.873 (5)	C6C—C7C	1.491 (6)
Cl1B—C13B	1.735 (5)	C7C—C8C	1.474 (8)
O1B—C7B	1.227 (8)	C8C—H8CA	0.9700
O2B—C9B	1.324 (7)	C8C—H8CB	0.9700
O2B—C8B	1.431 (5)	C9C—C10C	1.477 (6)
O3B—C9B	1.183 (7)	C10C—C15C	1.382 (8)
C1B—C2B	1.351 (7)	C10C—C11C	1.395 (7)
C1B—C6B	1.387 (7)	C11C—C12C	1.390 (6)
C1B—H1BA	0.9300	C11C—H11C	0.9300
C2B—C3B	1.364 (9)	C12C—C13C	1.368 (9)
C2B—H2BA	0.9300	C12C—H12C	0.9300
C3B—C4B	1.385 (7)	C13C—C14C	1.358 (7)
C4B—C5B	1.379 (6)	C14C—C15C	1.383 (7)
C4B—H4BA	0.9300	C14C—H14C	0.9300
C5B—C6B	1.387 (8)	C15C—H15C	0.9300
C5B—H5BA	0.9300		
C9A—O2A—C8A	115.7 (5)	O2B—C8B—H8BB	109.5
C6A—C1A—C2A	119.9 (5)	C7B—C8B—H8BB	109.5
C6A—C1A—H1AA	120.1	H8BA—C8B—H8BB	108.1
C2A—C1A—H1AA	120.1	O3B—C9B—O2B	124.5 (5)
C3A—C2A—C1A	118.9 (5)	O3B—C9B—C10B	124.2 (5)
C3A—C2A—H2AA	120.6	O2B—C9B—C10B	111.2 (5)
C1A—C2A—H2AA	120.6	C15B—C10B—C11B	119.6 (5)
C2A—C3A—C4A	121.8 (4)	C15B—C10B—C9B	123.6 (5)
C2A—C3A—Br1A	117.9 (4)	C11B—C10B—C9B	116.8 (5)
C4A—C3A—Br1A	120.4 (4)	C12B—C11B—C10B	120.5 (6)
C5A—C4A—C3A	118.6 (5)	C12B—C11B—H11B	119.8
C5A—C4A—H4AA	120.7	C10B—C11B—H11B	119.8
C3A—C4A—H4AA	120.7	C13B—C12B—C11B	118.4 (5)
C4A—C5A—C6A	122.0 (5)	C13B—C12B—H12B	120.8
C4A—C5A—H5AA	119.0	C11B—C12B—H12B	120.8
C6A—C5A—H5AA	119.0	C14B—C13B—C12B	121.8 (5)
C5A—C6A—C1A	118.8 (4)	C14B—C13B—C11B	121.6 (4)
C5A—C6A—C7A	120.0 (5)	C12B—C13B—C11B	116.6 (4)
C1A—C6A—C7A	121.2 (5)	C13B—C14B—C15B	119.5 (5)
O1A—C7A—C6A	121.4 (5)	C13B—C14B—H14B	120.3

supplementary materials

O1A—C7A—C8A	120.0 (4)	C15B—C14B—H14B	120.3
C6A—C7A—C8A	118.6 (5)	C10B—C15B—C14B	120.2 (5)
O2A—C8A—C7A	112.8 (5)	C10B—C15B—H15B	119.9
O2A—C8A—H8AA	109.0	C14B—C15B—H15B	119.9
C7A—C8A—H8AA	109.0	C9C—O2C—C8C	116.2 (4)
O2A—C8A—H8AB	109.0	C2C—C1C—C6C	120.6 (5)
C7A—C8A—H8AB	109.0	C2C—C1C—H1CA	119.7
H8AA—C8A—H8AB	107.8	C6C—C1C—H1CA	119.7
O3A—C9A—O2A	124.0 (5)	C3C—C2C—C1C	120.2 (5)
O3A—C9A—C10A	125.1 (5)	C3C—C2C—H2CA	119.9
O2A—C9A—C10A	110.8 (5)	C1C—C2C—H2CA	119.9
C15A—C10A—C11A	119.6 (4)	C2C—C3C—C4C	121.0 (4)
C15A—C10A—C9A	117.7 (5)	C2C—C3C—Br1C	119.1 (4)
C11A—C10A—C9A	122.6 (5)	C4C—C3C—Br1C	119.9 (4)
C12A—C11A—C10A	121.2 (6)	C3C—C4C—C5C	119.1 (6)
C12A—C11A—H11A	119.4	C3C—C4C—H4CA	120.4
C10A—C11A—H11A	119.4	C5C—C4C—H4CA	120.4
C11A—C12A—C13A	118.7 (6)	C4C—C5C—C6C	120.8 (6)
C11A—C12A—H12A	120.7	C4C—C5C—H5CA	119.6
C13A—C12A—H12A	120.7	C6C—C5C—H5CA	119.6
C12A—C13A—C14A	121.4 (4)	C1C—C6C—C5C	118.4 (4)
C12A—C13A—C11A	120.1 (5)	C1C—C6C—C7C	122.6 (5)
C14A—C13A—C11A	118.4 (5)	C5C—C6C—C7C	119.0 (5)
C15A—C14A—C13A	118.1 (5)	O1C—C7C—C8C	121.3 (5)
C15A—C14A—H14A	120.9	O1C—C7C—C6C	121.8 (5)
C13A—C14A—H14A	120.9	C8C—C7C—C6C	116.9 (4)
C10A—C15A—C14A	120.9 (5)	O2C—C8C—C7C	112.0 (5)
C10A—C15A—H15A	119.6	O2C—C8C—H8CA	109.2
C14A—C15A—H15A	119.6	C7C—C8C—H8CA	109.2
C9B—O2B—C8B	114.3 (5)	O2C—C8C—H8CB	109.2
C2B—C1B—C6B	121.4 (5)	C7C—C8C—H8CB	109.2
C2B—C1B—H1BA	119.3	H8CA—C8C—H8CB	107.9
C6B—C1B—H1BA	119.3	O3C—C9C—O2C	123.9 (5)
C1B—C2B—C3B	120.2 (5)	O3C—C9C—C10C	123.8 (6)
C1B—C2B—H2BA	119.9	O2C—C9C—C10C	112.3 (5)
C3B—C2B—H2BA	119.9	C15C—C10C—C11C	119.3 (4)
C2B—C3B—C4B	120.2 (5)	C15C—C10C—C9C	119.0 (5)
C2B—C3B—Br1B	120.8 (4)	C11C—C10C—C9C	121.6 (5)
C4B—C3B—Br1B	119.0 (4)	C12C—C11C—C10C	119.7 (5)
C5B—C4B—C3B	119.5 (6)	C12C—C11C—H11C	120.2
C5B—C4B—H4BA	120.2	C10C—C11C—H11C	120.2
C3B—C4B—H4BA	120.2	C13C—C12C—C11C	119.8 (5)
C4B—C5B—C6B	120.2 (5)	C13C—C12C—H12C	120.1
C4B—C5B—H5BA	119.9	C11C—C12C—H12C	120.1
C6B—C5B—H5BA	119.9	C14C—C13C—C12C	120.8 (5)
C1B—C6B—C5B	118.4 (4)	C14C—C13C—H11C	119.5 (5)
C1B—C6B—C7B	121.2 (5)	C12C—C13C—H11C	119.7 (4)
C5B—C6B—C7B	120.4 (5)	C13C—C14C—C15C	120.4 (5)
O1B—C7B—C6B	121.1 (6)	C13C—C14C—H14C	119.8

O1B—C7B—C8B	121.6 (5)	C15C—C14C—H14C	119.8
C6B—C7B—C8B	117.2 (5)	C10C—C15C—C14C	120.0 (5)
O2B—C8B—C7B	110.8 (5)	C10C—C15C—H15C	120.0
O2B—C8B—H8BA	109.5	C14C—C15C—H15C	120.0
C7B—C8B—H8BA	109.5		
C6A—C1A—C2A—C3A	2.5 (7)	C8B—O2B—C9B—O3B	0.8 (8)
C1A—C2A—C3A—C4A	-3.3 (8)	C8B—O2B—C9B—C10B	-179.0 (4)
C1A—C2A—C3A—Br1A	175.9 (4)	O3B—C9B—C10B—C15B	-178.7 (6)
C2A—C3A—C4A—C5A	1.8 (8)	O2B—C9B—C10B—C15B	1.2 (7)
Br1A—C3A—C4A—C5A	-177.3 (4)	O3B—C9B—C10B—C11B	-0.2 (8)
C3A—C4A—C5A—C6A	0.5 (8)	O2B—C9B—C10B—C11B	179.6 (5)
C4A—C5A—C6A—C1A	-1.3 (8)	C15B—C10B—C11B—C12B	-1.5 (9)
C4A—C5A—C6A—C7A	177.7 (5)	C9B—C10B—C11B—C12B	-180.0 (5)
C2A—C1A—C6A—C5A	-0.2 (7)	C10B—C11B—C12B—C13B	1.4 (9)
C2A—C1A—C6A—C7A	-179.2 (5)	C11B—C12B—C13B—C14B	-1.7 (9)
C5A—C6A—C7A—O1A	5.4 (8)	C11B—C12B—C13B—C11B	179.0 (5)
C1A—C6A—C7A—O1A	-175.7 (5)	C12B—C13B—C14B—C15B	2.0 (8)
C5A—C6A—C7A—C8A	-174.0 (5)	C11B—C13B—C14B—C15B	-178.7 (4)
C1A—C6A—C7A—C8A	4.9 (7)	C11B—C10B—C15B—C14B	1.8 (8)
C9A—O2A—C8A—C7A	-76.3 (6)	C9B—C10B—C15B—C14B	-179.8 (5)
O1A—C7A—C8A—O2A	3.6 (8)	C13B—C14B—C15B—C10B	-2.0 (8)
C6A—C7A—C8A—O2A	-177.0 (5)	C6C—C1C—C2C—C3C	1.0 (8)
C8A—O2A—C9A—O3A	-1.7 (7)	C1C—C2C—C3C—C4C	-0.1 (8)
C8A—O2A—C9A—C10A	-178.6 (4)	C1C—C2C—C3C—Br1C	-178.0 (4)
O3A—C9A—C10A—C15A	1.5 (8)	C2C—C3C—C4C—C5C	-0.3 (8)
O2A—C9A—C10A—C15A	178.3 (5)	Br1C—C3C—C4C—C5C	177.5 (4)
O3A—C9A—C10A—C11A	-176.0 (5)	C3C—C4C—C5C—C6C	-0.2 (8)
O2A—C9A—C10A—C11A	0.8 (7)	C2C—C1C—C6C—C5C	-1.6 (7)
C15A—C10A—C11A—C12A	-1.2 (8)	C2C—C1C—C6C—C7C	177.8 (5)
C9A—C10A—C11A—C12A	176.3 (5)	C4C—C5C—C6C—C1C	1.2 (7)
C10A—C11A—C12A—C13A	0.5 (9)	C4C—C5C—C6C—C7C	-178.3 (5)
C11A—C12A—C13A—C14A	0.2 (9)	C1C—C6C—C7C—O1C	174.4 (5)
C11A—C12A—C13A—C11A	-179.3 (5)	C5C—C6C—C7C—O1C	-6.2 (7)
C12A—C13A—C14A—C15A	-0.2 (8)	C1C—C6C—C7C—C8C	-5.8 (7)
C11A—C13A—C14A—C15A	179.3 (4)	C5C—C6C—C7C—C8C	173.6 (5)
C11A—C10A—C15A—C14A	1.2 (8)	C9C—O2C—C8C—C7C	77.1 (6)
C9A—C10A—C15A—C14A	-176.4 (5)	O1C—C7C—C8C—O2C	-2.3 (8)
C13A—C14A—C15A—C10A	-0.5 (8)	C6C—C7C—C8C—O2C	178.0 (4)
C6B—C1B—C2B—C3B	2.3 (9)	C8C—O2C—C9C—O3C	-1.4 (8)
C1B—C2B—C3B—C4B	-1.9 (9)	C8C—O2C—C9C—C10C	178.1 (4)
C1B—C2B—C3B—Br1B	179.8 (5)	O3C—C9C—C10C—C15C	-1.6 (8)
C2B—C3B—C4B—C5B	0.7 (8)	O2C—C9C—C10C—C15C	178.9 (5)
Br1B—C3B—C4B—C5B	179.1 (4)	O3C—C9C—C10C—C11C	174.3 (5)
C3B—C4B—C5B—C6B	0.1 (8)	O2C—C9C—C10C—C11C	-5.2 (7)
C2B—C1B—C6B—C5B	-1.5 (8)	C15C—C10C—C11C—C12C	0.4 (7)
C2B—C1B—C6B—C7B	178.7 (5)	C9C—C10C—C11C—C12C	-175.5 (5)
C4B—C5B—C6B—C1B	0.3 (8)	C10C—C11C—C12C—C13C	-0.5 (8)
C4B—C5B—C6B—C7B	-179.9 (5)	C11C—C12C—C13C—C14C	0.5 (8)
C1B—C6B—C7B—O1B	4.9 (8)	C11C—C12C—C13C—C11C	179.6 (4)

supplementary materials

C5B—C6B—C7B—O1B	-174.8 (5)	C12C—C13C—C14C—C15C	-0.6 (8)
C1B—C6B—C7B—C8B	-172.6 (5)	C11C—C13C—C14C—C15C	-179.7 (4)
C5B—C6B—C7B—C8B	7.7 (7)	C11C—C10C—C15C—C14C	-0.4 (7)
C9B—O2B—C8B—C7B	171.7 (4)	C9C—C10C—C15C—C14C	175.6 (5)
O1B—C7B—C8B—O2B	1.1 (7)	C13C—C14C—C15C—C10C	0.5 (8)
C6B—C7B—C8B—O2B	178.6 (4)		

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>
C5A—H5AA···O3B	0.93	2.59	3.227 (7)	126
C5B—H5BA···O1A	0.93	2.48	3.217 (7)	136
C15B—H15B···O1C	0.93	2.59	3.254 (7)	129

Fig. 1

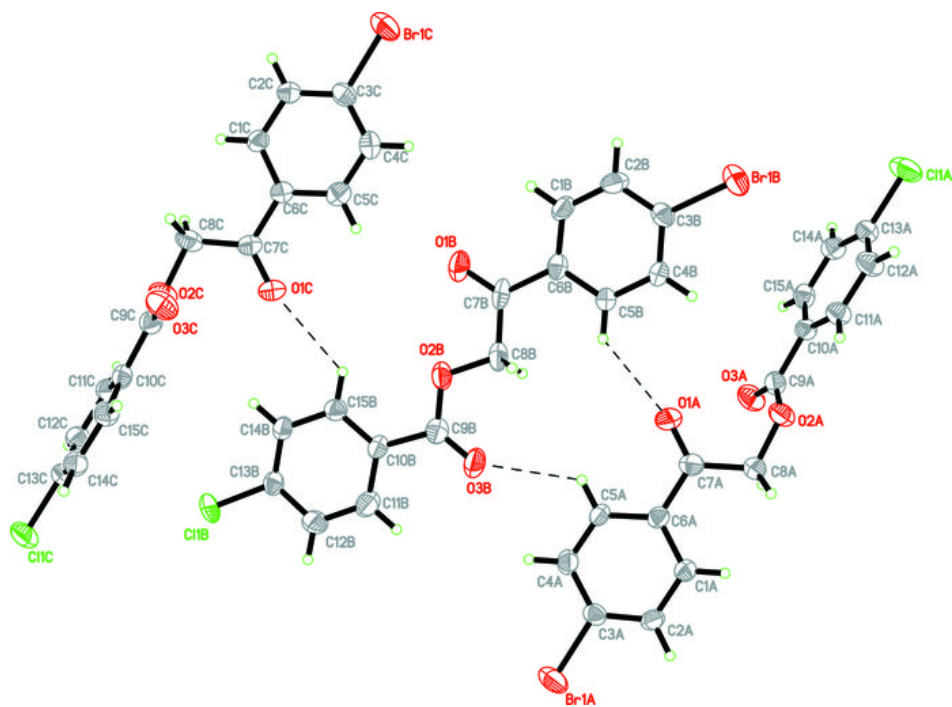


Fig. 2

