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3-(2-Bromoethoxy)-4-(4-bromophenyl)-furan-5(2H)-one

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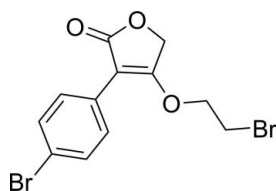
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.010$ Å; disorder in main residue; R factor = 0.061; wR factor = 0.187; data-to-parameter ratio = 16.7.

In the title compound, $\text{C}_{12}\text{H}_{10}\text{Br}_2\text{O}_3$, the dihedral angle between the furan-5(2H)-one ring and the benzene ring is $1.2(3)^\circ$. Two intramolecular $\text{C}-\text{H}\cdots\text{O}$ interactions occur in the molecule, both of which generate $S(6)$ rings. The bromoethyl fragment is disordered over two sets of sites in a 0.773(8):0.227(8) ratio. In the crystal, inversion dimers linked by pairs of $\text{C}-\text{H}\cdots\pi$ interactions occur.

Related literature

For background to furanones, see: Bailly *et al.* (2008); Weber *et al.* (2005).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{10}\text{Br}_2\text{O}_3$ $M_r = 362.02$ Monoclinic, $P2_1/c$ $a = 8.6171(13)$ Å $b = 10.4434(16)$ Å $c = 13.958(2)$ Å $\beta = 95.831(3)^\circ$ $V = 1249.6(3)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 6.48$ mm⁻¹
 $T = 298$ K

0.20 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

 $T_{\min} = 0.357$, $T_{\max} = 0.564$ 7195 measured reflections
2582 independent reflections
1765 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.061$ $wR(F^2) = 0.187$ $S = 1.05$

2582 reflections

155 parameters

29 restraints

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 1.37$ e Å⁻³ $\Delta\rho_{\text{min}} = -1.15$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

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

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2\cdots\text{O}1$	0.93	2.35	3.018 (10)	129
$\text{C}6-\text{H}6\cdots\text{O}3$	0.93	2.25	2.916 (8)	128
$\text{C}9-\text{H}9\text{B}\cdots\text{C}g1^i$	0.97	2.80	3.632 (9)	144

Symmetry code: (i) $-x, -y + 1, -z + 1$.

Data collection: SMART (Bruker, 2007); cell refinement: SAINT (Bruker, 2007); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5720).

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supporting information

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3-(2-Bromoethoxy)-4-(4-bromophenyl)furan-5(2*H*)-one

Zhu-Ping Xiao, Wen-Bin Yan, Zhu-Xiang Liu, Li-hua Chen and Xiao-Chun Peng

S1. Comment

Many compounds with γ -butyrolactone-core (furanone) show diverse biological activities such as antitumor and anti-inflammatory activity (Bailly *et al.*, 2008; Weber *et al.*, 2005). Recently, we focused our efforts to synthesize enamines with γ -butyrolactone-core for antibacterial activity screening. Herein, we report the crystal structure of the title compound (I).

Bond distance C7—C10 (1.334 (10) Å) is followed in the range of a typical double bond (1.32–1.38 Å), and the title compound was therefore identified as a furan-5(2*H*)-one not a furan-2(3*H*)-one. C10—O3 (1.348 (8) Å) bond has shorter bond distance than the standard C—O single bond (1.41–1.44 Å), but longer than C—O double bond (1.19–1.23 Å). This clearly indicated that an sp^3 orbital of O3 is conjugated with the π molecular orbital of C7—C10 double bond, which was supported by the small torsion angle (0.4 (12) °) of C1—C7—C10—O3. The stereochemistry of the double bond in lactone ring was assigned as (*E*)-configuration based on X-ray crystallography of the title compound (Fig. 1). The butyrolactone moiety makes a dihedral angle of 1.2 (3) ° with the 4-fluorophenyl group. The side chain bromoethyl group is disorder (Fig. 1). C—H \cdots O contacts link molecules into dimers (Fig. 2), and the result dimers are packed by van der Waals.

S2. Experimental

3-(4-Bromophenyl)-4-hydroxyfuran-5(2*H*)-one (0.77 g, 3 mmol) was added to a solution of 1,2-dibromoethane (2.8 g, 15 mmol) and triethylamine (0.7 g, 7 mmol) in dry acetone. The stirring was maintained at reflux temperature for 5 h. After the solvent was removed, the residue was partitioned between EtOAc and water. The organic layer was then dried over MgSO₄ and concentrated under reduced pressure. Flash chromatography (EtOAc/petroleum ether, 1/1, *v/v*) gave a fraction, which was partially evaporated to give the colorless blocks of (I).

S3. Refinement

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H = 0.93 and 0.96 Å for the aromatic and CH₂ type H atoms, respectively. $U_{\text{iso}} = 1.2U_{\text{eq}}$ (parent atoms) were assigned for all H atoms.

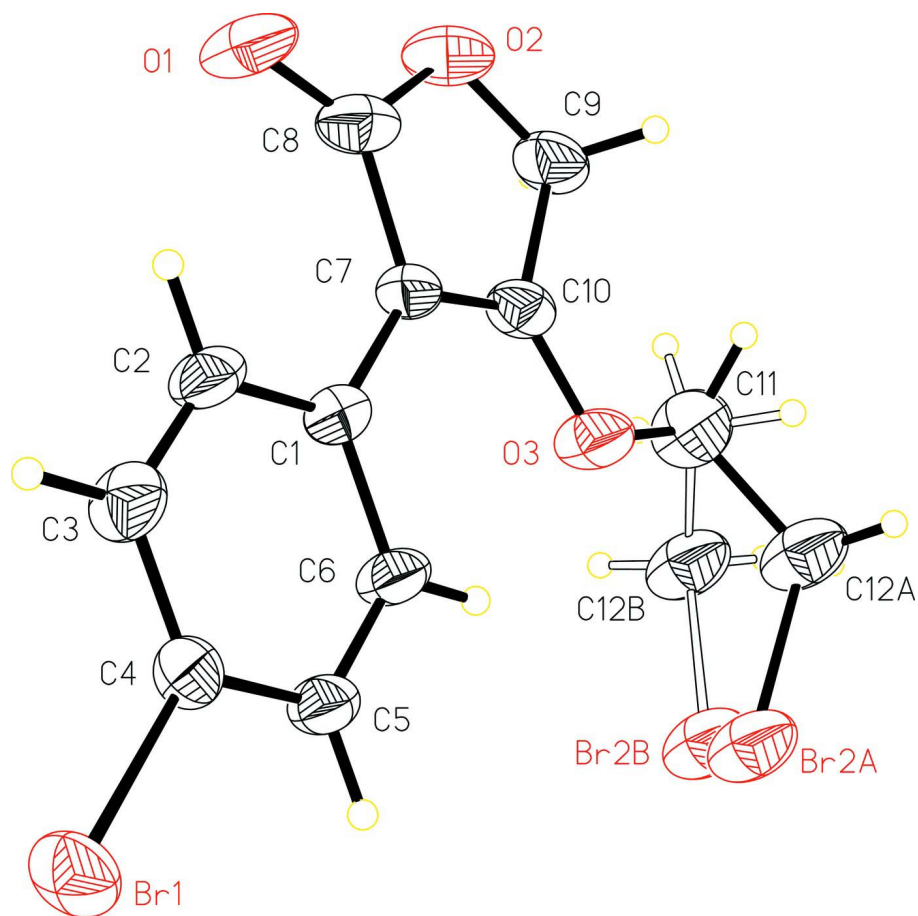


Figure 1

Molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

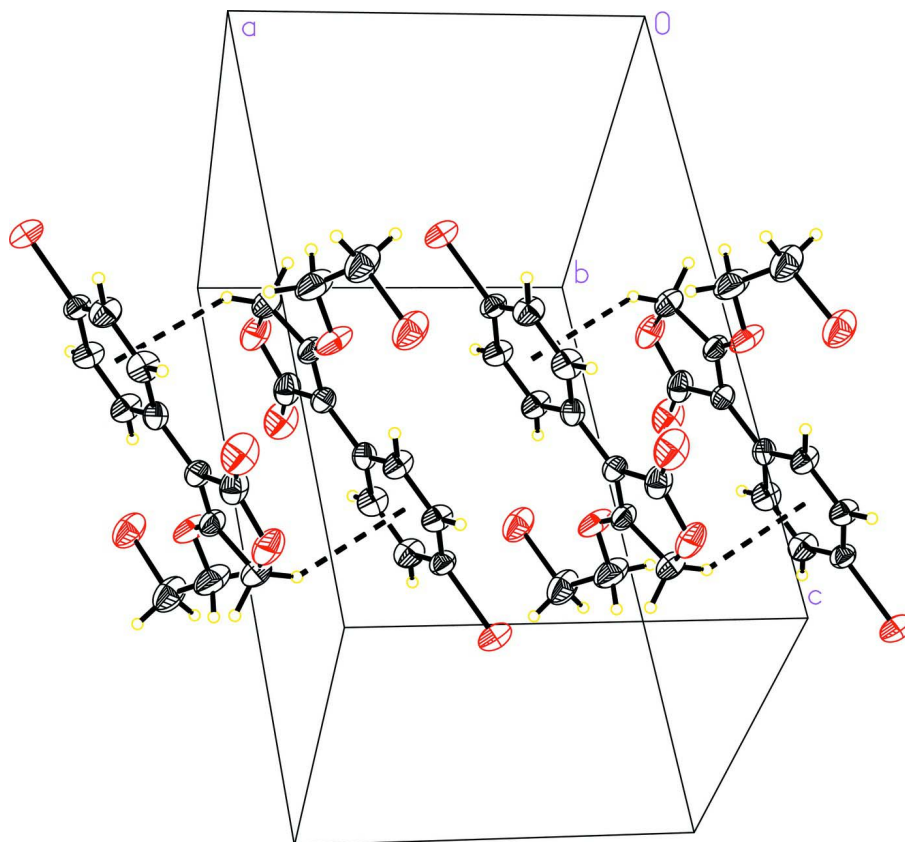


Figure 2

Dimers are formed through intermolecular C—H... π hydrogen bond pairs. Dashed lines indicate C—H... π contacts.

3-(2-Bromoethoxy)-4-(4-bromophenyl)furan-5(2H)-one

Crystal data

$C_{12}H_{10}Br_2O_3$

$M_r = 362.02$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 8.6171(13)\ \text{\AA}$

$b = 10.4434(16)\ \text{\AA}$

$c = 13.958(2)\ \text{\AA}$

$\beta = 95.831(3)^\circ$

$V = 1249.6(3)\ \text{\AA}^3$

$Z = 4$

$F(000) = 704$

$D_x = 1.924\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 1832 reflections

$\theta = 2.6\text{--}26.1^\circ$

$\mu = 6.48\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, colorless

$0.20 \times 0.10 \times 0.10\ \text{mm}$

Data collection

Bruker SMART APEX CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.357$, $T_{\max} = 0.564$

7195 measured reflections

2582 independent reflections

1765 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\max} = 26.5^\circ$, $\theta_{\min} = 2.4^\circ$

$h = -10 \rightarrow 10$

$k = -6 \rightarrow 13$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.187$
 $S = 1.05$
 2582 reflections
 155 parameters
 29 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
 $w = 1/[\sigma^2(F_o^2) + (0.1008P)^2 + 2.0749P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 1.37 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -1.15 \text{ e } \text{Å}^{-3}$

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 (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Br1	0.50480 (10)	0.34315 (8)	0.21873 (6)	0.0737 (4)	
Br2A	0.2920 (5)	0.92938 (17)	0.48473 (18)	0.0900 (8)	0.773 (8)
Br2B	0.2207 (13)	0.9329 (7)	0.4681 (7)	0.0900 (8)	0.227 (8)
C1	0.2553 (7)	0.4284 (6)	0.4943 (5)	0.0478 (14)	
C2	0.2843 (9)	0.3041 (7)	0.4667 (5)	0.0600 (18)	
H2	0.2534	0.2368	0.5041	0.072*	
C3	0.3561 (9)	0.2770 (7)	0.3870 (6)	0.0627 (18)	
H3	0.3711	0.1923	0.3696	0.075*	
C4	0.4069 (8)	0.3750 (7)	0.3319 (5)	0.0516 (15)	
C5	0.3815 (9)	0.5002 (6)	0.3576 (5)	0.0562 (17)	
H5	0.4147	0.5668	0.3205	0.067*	
C6	0.3076 (8)	0.5270 (6)	0.4379 (5)	0.0566 (17)	
H6	0.2920	0.6117	0.4549	0.068*	
C7	0.1745 (8)	0.4574 (7)	0.5800 (5)	0.0491 (15)	
C8	0.1126 (9)	0.3606 (9)	0.6426 (6)	0.067 (2)	
C9	0.0595 (10)	0.5553 (9)	0.7061 (5)	0.069 (2)	
H9A	0.1208	0.5923	0.7614	0.083*	
H9B	-0.0425	0.5954	0.6988	0.083*	
C10	0.1405 (8)	0.5708 (7)	0.6165 (5)	0.0548 (16)	
C11	0.1273 (12)	0.7904 (7)	0.6271 (7)	0.092 (3)	
H11A	0.1406	0.7743	0.6959	0.110*	0.773 (8)
H11B	0.0172	0.8050	0.6085	0.110*	0.773 (8)
H11C	0.2125	0.8202	0.6724	0.110*	0.227 (8)
H11D	0.0412	0.7671	0.6633	0.110*	0.227 (8)
C12A	0.2190 (15)	0.9106 (9)	0.6052 (6)	0.0900 (8)	0.773 (8)

H12A	0.3085	0.9160	0.6532	0.108*	0.773 (8)
H12B	0.1534	0.9840	0.6150	0.108*	0.773 (8)
C12B	0.076 (3)	0.899 (2)	0.5565 (19)	0.0900 (8)	0.227 (8)
H12C	0.0599	0.9763	0.5929	0.108*	0.227 (8)
H12D	-0.0235	0.8763	0.5214	0.108*	0.227 (8)
O1	0.1076 (8)	0.2448 (6)	0.6373 (5)	0.0886 (18)	
O2	0.0467 (7)	0.4217 (6)	0.7160 (4)	0.0808 (17)	
O3	0.1739 (7)	0.6861 (5)	0.5802 (4)	0.0731 (16)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0856 (6)	0.0761 (6)	0.0639 (5)	0.0142 (4)	0.0292 (4)	-0.0115 (4)
Br2A	0.102 (2)	0.0635 (7)	0.1132 (11)	-0.0048 (10)	0.0551 (13)	-0.0016 (6)
Br2B	0.102 (2)	0.0635 (7)	0.1132 (11)	-0.0048 (10)	0.0551 (13)	-0.0016 (6)
C1	0.046 (3)	0.045 (4)	0.052 (3)	-0.003 (3)	0.008 (3)	0.007 (3)
C2	0.080 (5)	0.038 (4)	0.064 (4)	-0.003 (3)	0.016 (4)	0.012 (3)
C3	0.076 (5)	0.038 (4)	0.076 (5)	0.008 (3)	0.017 (4)	-0.002 (3)
C4	0.055 (4)	0.049 (4)	0.053 (3)	0.004 (3)	0.015 (3)	-0.008 (3)
C5	0.079 (5)	0.040 (4)	0.054 (4)	-0.005 (3)	0.024 (3)	0.002 (3)
C6	0.076 (5)	0.038 (3)	0.060 (4)	-0.004 (3)	0.028 (3)	0.006 (3)
C7	0.052 (4)	0.054 (4)	0.043 (3)	-0.005 (3)	0.012 (3)	0.005 (3)
C8	0.060 (4)	0.081 (6)	0.061 (4)	-0.002 (4)	0.015 (3)	0.022 (4)
C9	0.074 (5)	0.089 (6)	0.049 (4)	-0.004 (4)	0.026 (3)	0.000 (4)
C10	0.053 (4)	0.068 (5)	0.046 (3)	0.000 (3)	0.018 (3)	0.001 (3)
C11	0.116 (7)	0.071 (5)	0.096 (6)	-0.002 (5)	0.054 (5)	-0.024 (4)
C12A	0.102 (2)	0.0635 (7)	0.1132 (11)	-0.0048 (10)	0.0551 (13)	-0.0016 (6)
C12B	0.102 (2)	0.0635 (7)	0.1132 (11)	-0.0048 (10)	0.0551 (13)	-0.0016 (6)
O1	0.106 (5)	0.067 (4)	0.099 (4)	-0.010 (3)	0.037 (3)	0.031 (3)
O2	0.084 (4)	0.098 (5)	0.067 (3)	-0.003 (3)	0.039 (3)	0.021 (3)
O3	0.111 (4)	0.048 (3)	0.069 (3)	0.000 (3)	0.053 (3)	-0.006 (2)

Geometric parameters (Å, °)

Br1—C4	1.895 (6)	C8—O2	1.377 (10)
Br2A—C12A	1.864 (5)	C9—O2	1.407 (11)
Br2B—C12B	1.878 (6)	C9—C10	1.501 (9)
C1—C2	1.384 (10)	C9—H9A	0.9700
C1—C6	1.397 (9)	C9—H9B	0.9700
C1—C7	1.476 (9)	C10—O3	1.348 (8)
C2—C3	1.357 (11)	C11—O3	1.352 (9)
C2—H2	0.9300	C11—C12A	1.531 (5)
C3—C4	1.378 (10)	C11—C12B	1.539 (6)
C3—H3	0.9300	C11—H11A	0.9700
C4—C5	1.379 (10)	C11—H11B	0.9700
C5—C6	1.372 (9)	C11—H11C	0.9700
C5—H5	0.9300	C11—H11D	0.9700
C6—H6	0.9300	C12A—H12A	0.9700

C7—C10	1.334 (10)	C12A—H12B	0.9700
C7—C8	1.471 (10)	C12B—H12C	0.9700
C8—O1	1.212 (10)	C12B—H12D	0.9700
C2—C1—C6	117.2 (6)	O3—C11—C12B	111.5 (12)
C2—C1—C7	122.0 (6)	C12A—C11—C12B	52.3 (13)
C6—C1—C7	120.8 (6)	O3—C11—H11A	109.1
C3—C2—C1	122.2 (6)	C12A—C11—H11A	109.1
C3—C2—H2	118.9	C12B—C11—H11A	139.4
C1—C2—H2	118.9	O3—C11—H11B	109.1
C2—C3—C4	120.0 (6)	C12A—C11—H11B	109.1
C2—C3—H3	120.0	C12B—C11—H11B	60.0
C4—C3—H3	120.0	H11A—C11—H11B	107.9
C3—C4—C5	119.4 (6)	O3—C11—H11C	109.3
C3—C4—Br1	121.9 (5)	C12A—C11—H11C	59.7
C5—C4—Br1	118.6 (5)	C12B—C11—H11C	109.3
C6—C5—C4	120.3 (6)	H11A—C11—H11C	53.5
C6—C5—H5	119.9	H11B—C11—H11C	141.2
C4—C5—H5	119.9	O3—C11—H11D	109.3
C5—C6—C1	120.8 (7)	C12A—C11—H11D	138.3
C5—C6—H6	119.6	C12B—C11—H11D	109.3
C1—C6—H6	119.6	H11A—C11—H11D	57.4
C10—C7—C8	106.0 (6)	H11B—C11—H11D	53.1
C10—C7—C1	129.2 (6)	H11C—C11—H11D	108.0
C8—C7—C1	124.8 (7)	C11—C12A—Br2A	119.6 (6)
O1—C8—O2	119.5 (7)	C11—C12A—H12A	107.4
O1—C8—C7	131.4 (8)	Br2A—C12A—H12A	107.4
O2—C8—C7	109.0 (7)	C11—C12A—H12B	107.4
O2—C9—C10	103.7 (6)	Br2A—C12A—H12B	107.4
O2—C9—H9A	111.0	H12A—C12A—H12B	106.9
C10—C9—H9A	111.0	C11—C12B—Br2B	113.0 (7)
O2—C9—H9B	111.0	C11—C12B—H12C	109.0
C10—C9—H9B	111.0	Br2B—C12B—H12C	109.0
H9A—C9—H9B	109.0	C11—C12B—H12D	109.0
C7—C10—O3	125.9 (6)	Br2B—C12B—H12D	109.0
C7—C10—C9	111.1 (6)	H12C—C12B—H12D	107.8
O3—C10—C9	123.0 (6)	C8—O2—C9	110.1 (6)
O3—C11—C12A	112.3 (6)	C10—O3—C11	116.9 (5)
C6—C1—C2—C3	-1.9 (11)	C8—C7—C10—O3	-178.9 (7)
C7—C1—C2—C3	178.8 (7)	C1—C7—C10—O3	0.4 (12)
C1—C2—C3—C4	1.8 (12)	C8—C7—C10—C9	1.8 (8)
C2—C3—C4—C5	-1.1 (11)	C1—C7—C10—C9	-178.9 (7)
C2—C3—C4—Br1	-178.9 (6)	O2—C9—C10—C7	-1.5 (9)
C3—C4—C5—C6	0.5 (11)	O2—C9—C10—O3	179.1 (7)
Br1—C4—C5—C6	178.4 (6)	O3—C11—C12A—Br2A	31.9 (14)
C4—C5—C6—C1	-0.6 (12)	C12B—C11—C12A—Br2A	-68.7 (13)
C2—C1—C6—C5	1.3 (11)	O3—C11—C12B—Br2B	-49 (2)

C7—C1—C6—C5	-179.4 (7)	C12A—C11—C12B—Br2B	53.0 (14)
C2—C1—C7—C10	178.8 (7)	O1—C8—O2—C9	-177.4 (8)
C6—C1—C7—C10	-0.5 (11)	C7—C8—O2—C9	0.4 (8)
C2—C1—C7—C8	-2.0 (11)	C10—C9—O2—C8	0.6 (8)
C6—C1—C7—C8	178.7 (7)	C7—C10—O3—C11	179.0 (8)
C10—C7—C8—O1	176.1 (9)	C9—C10—O3—C11	-1.7 (11)
C1—C7—C8—O1	-3.2 (13)	C12A—C11—O3—C10	158.9 (8)
C10—C7—C8—O2	-1.4 (8)	C12B—C11—O3—C10	-144.3 (13)
C1—C7—C8—O2	179.3 (6)		

Hydrogen-bond geometry (\AA , $^\circ$)

Cg1 is the centroid of the C1–C6 benzene 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>
C2—H2 \cdots O1	0.93	2.35	3.018 (10)	129
C6—H6 \cdots O3	0.93	2.25	2.916 (8)	128
C9—H9B \cdots Cg1 ⁱ	0.97	2.80	3.632 (9)	144

Symmetry code: (i) $-x, -y+1, -z+1$.