organic compounds

(3S*,4S*,E)-tert-Butyl 3,4-dibromo-5-oxocyclooct-1-enecarboxylate

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The title compound, $C_{13}H_{18}Br_2O_3$, was prepared by a bromination reaction of (1E,3Z)-methyl 5-oxocycloocta-1,3-dienecarboxylate, which was obtained by an epoxydation reaction of tert-butyl cycloocta-1,3-dienecarboxylate. The crystal structure confirms unequivocally the absolute configuration of both chiral centres to be S. In the crystal, C—H···O interactions link the molecules into chains running along the c axis.

Related literature

For the Michael addition of enantiomerically pure lithium amides, see: Davies et al. (2005). For their importance in pharmacology, see: Fülöp et al. (2001). For the reactivity of the cycloocta-1,5-diene in basic medium, see: Huber et al. (1969, 1970). For the preparation of analogous unsaturated cyclooctane esters, see: Garrido et al. (2008).
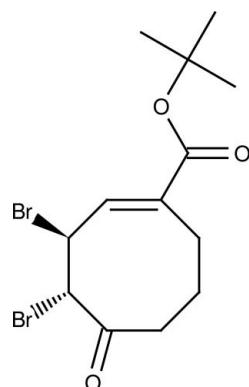
Experimental

Crystal data

$C_{13}H_{18}Br_2O_3$	$V = 1521.07$ (8) Å^3
$M_r = 382.09$	$Z = 4$
Orthorhombic, $Pca2_1$	Cu $K\alpha$ radiation
$a = 14.0658$ (4) Å	$\mu = 6.76$ mm^{-1}
$b = 9.5990$ (3) Å	$T = 298$ K
$c = 11.2657$ (3) Å	$0.24 \times 0.14 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	10215 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2006)	2170 independent reflections
$T_{min} = 0.370$, $T_{max} = 0.509$	2153 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.048$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$	H-atom parameters constrained
$wR(F^2) = 0.075$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
$S = 1.09$	$\Delta\rho_{min} = -0.46$ e Å^{-3}
2170 reflections	Absolute structure: Flack (1983), 803 Friedel pairs
166 parameters	Flack parameter: 0.06 (3)
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O3^i	0.98	2.57	3.525 (5)	165
C8—H8A···O3^i	0.97	2.63	3.590 (6)	172

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

Data collection: APEX2 (Bruker 2006); cell refinement: SAINT (Bruker 2006); 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., 2006); software used to prepare material for publication: SHELXTL (Sheldrick, 2008).

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supporting information

(3*S**,4*S**,*E*)-*tert*-Butyl 3,4-dibromo-5-oxocyclooct-1-enecarboxylate

Magda Blanco, Narciso M. Garrido, Francisca Sanz and David Diez

S1. Comment

In our research group there has been an enormous interest in the synthesis of conjugated unsaturated esters used as starting material in the Michael addition of enantiomerically pure lithium amides (Davies *et al.*, 2005) as a base tool in the asymmetric synthesis of *β*-amino acids and alkaloids because of their interest and value in the development of biologically active compounds for the pharmacology industry (Fülöp *et al.*, 2001). Considering that the reactivity of the cycloocta-1,5-diene is very peculiar, highlighting its trend in basic medium to conjugate its double bonds (Huber *et al.*, 1969) because of the greater thermodynamic stability (Huber *et al.*, 1970), is necessary and important to establish the exact structure in this class of unsaturated rings. This conjugation was determined previously for an isomer of compound **1** (*tert*-butyl cyclooct-1,7-dienecarboxylate) (Garrido *et al.*, 2008) and herein for compound **1** (*tert*-butylcyclooct-1,3-dienecarboxylate) by R—X spectroscopy of compound **6** which confirms unequivocally its configuration and structure. The crystal was afforded by epoxydation reaction of compound **1** with MCPBA and bromination reaction of compound **4** (Fig. 1).

The crystal contains an unique molecule in the asymmetric unit. The title molecule consists of a ring cyclooctene with two bromine atoms, a carbonyl group and a *tert*-butoxycarbonyl group as susbtituents. All the bond lengths and angles are within the normal ranges. The Br1—C3 and Br2—C4 bond lengths are 1.956 (4) Å and 1.946 (4) Å, respectively. The bromine atoms at C3 and C4 are nearly coplanar with the cyclooctene ring being the Br1—C3—C4—C5 and Br2—C4—C3—C2 torsion angles of 173.6 (3)° and -173.0 (1)°, respectively. In the case of the *tert*-butoxycarbonyl group at C1 is also coplanar with the cyclooctene ring being the O2—C9—O1—C1 torsion angle of 178.2 (7)°. The carbonyl group at atom C5 is twisted with the cyclooctene ring being the O3—C5—C4—C3 torsion angle of 123.3 (8)°.

In the crystal structure, molecules are connected by intermolecular C—H···O interactions to form infinite chains running along [001] direction, which seems to be effective in the stabilization of the structure (Table 1).

S2. Experimental

Epoxydation reaction, synthesis of (1E,3Z)-tert-butyl 5-oxocycloocta-1,3-dienecarboxylate **4**. Compound **1** (623.8 mg, 3.0 mmol, 1 equiv) was dissolved in DCM (30 ml), and stirred at 0°C, MCPBA (568.5 mg, 3.3 mmol, 1.1 equiv) was added slowly and the solution was stirred for 5 h at room temperature. The reaction mixture was quenched with saturated $Na_2S_2O_3$ (10 ml), extracted with DCM (3 x 80 ml), washed with H_2O, saturated $NaHCO_3$ and $Na_2S_2O_3$. The combined organic extracts were dried over Na_2SO_4, filtered and concentrated *in vacuo*. Purification by silica gel for flash column chromatography (Hex/EtOAc (99:1 *v/v*) gave recovery of starting material (16%), (1*Z*,3*R**,4*S**) *tert*-butyl cycloocta-1,3-diene carboxylate 3,4 oxide **2** (450 mg, 67%), (1*R**,2*S**,3*E*) *tert*-butyl cycloocta-3,4-diene carboxylate 1,2 oxide **3** (47 mg, 7%), (1E,3*Z*)-*tert*-butyl 5-hydroxycycloocta-1,3-dienecarboxylate **5** (47 mg, 7%) and (1*E*,3*Z*)-*tert*-butyl 5-oxocyclo-octa-1,3-dienecarboxylate **4** as a pale yellow oil (27 mg, 4%), IR *v*max (neat): 2976 and 2868 (C—H), 1707 (C=OOtBu), 1663 (C=O), 1456, 1370, 1292 (C—O), 1252, 1157 cm^{-1}. ^{1}H NMR (400 MHz; CDCl$_3$): δ 1.52 (9*H*, s, COOC(CH$_3$)$_3$); 2.10

(2*H*, quint, J 6.6, H-7); 2.50 (2*H*, t, J 6.6, H-8); 2.57 (2*H*, t, J 6.6, H-6); 6.03 (1*H*, d, J 12.6, H-4); 6.57 (1*H*, dd, J 5.5 and 12.6, H-3); 7.26 (1*H*, d, J 5.5, H-2). ^{13}C RMN (50 MHz; CDCl$_3$): δ 26.3 (CH$_2$, C-7); 28.0 (CH$_3$ *x* 3, COOC(CH$_3$)$_3$); 31.8 (CH$_2$, C-8); 38.5 (CH$_2$, C-6); 81.4 (C, COOC(CH$_3$)$_3$); 133.6 (CH, C-4); 134.7 (CH, C-2); 135.8 (CH, C-3); 140.1 (C, C-1); 165.5 (C, COOC(CH$_3$)$_3$); 205.2 (C, C-5). m/z (Cl$^+$) (*rel*. intensity): 222 (*M*$^+$, 5) 205 (3), 186 (5), 166 (19), 149 (19), 121 (22), 94 (13), 77 (26), 57 (100).

Synthesis of (3R*,4R*,E)-tert-butyl 3,4-dibromo-5-oxocycloocta-1-enecarboxylate **6**. Compound **4** (27.00 mg, 0.12 mmol) was dissolved in CCl$_4$ (10 ml) and the reaction system was stirred and cooled down at 0°C. After, Br$_2$ (0.01 ml, 31 mmol) was added and stirred for 30 min, the ice bath was removed and stirred for 4 h at r.t. The reaction mixture was dissolved in DCM (20 ml), washed with HCl 2 N., NaHCO$_3$(sat.), H$_2$O and NaCl (sat.); dried over Na$_2$SO$_4$, filtered and concentrated *in vacuo*. It afforded *tert*-butyl 3,4-dibromo-5-oxocyclooct-1-enecarboxylate **6** (43.00 mg, 91%) which crystallizes in Hex/EtOAc (1:1 *v/v*), mp 161–162 °C, IR *v*max (neat): 2976 and 2930 (C—H), 1712 (C=O), 1449 (C=C), 1369, 1292 (C—O), 1253, 1159, 1127, 1110 (C—Br) cm$^{-1.}$ ^{1}H NMR (400 MHz; CDCl$_3$): δ 1.47 (9*H*, s, COOC(CH$_3$)$_3$); 2.00–3.02 (6*H*, m, H-6, H-7, H-8); 4.23 (1*H*, d, J 11.2, H-4); 5.01 (1*H*, dd, J 11.2 and 9.6, H-3); 6.80 (1*H*, d, J 9.6, H-2). ^{13}C RMN (50 MHz; CDCl$_3$): δ 27.1 (CH$_2$, C-7); 27.8 (CH$_2$, C-8); 28.2 (CH$_3$ *x* 3, COOC(CH$_3$)$_3$); 37.8 (CH$_2$, C-6); 46.8 (CH); 60.4 (CH); 82.2 (C,COOC(CH$_3$)$_3$); 137.5 (CH, C-2); 138.1 (C, C-1); 164.7 (C, COOC(CH$_3$)$_3$); 202.0 (C, C-5). HRMS (ESI) m/z calcd.for C$_{13}$H$_{18}$Br$_2$O$_3$ [*M*+Na]:402.9515; found 402.9543; R—X.

S3. Refinement

The hydrogen atoms were positioned geometrically, with C—H distances constrained to 0.93 Å (aromatic CH), 0.96 Å (methyl CH3), 0.97 Å methylene CH2), 098 Å (methine CH) and refined in riding mode with U_{iso}(H) = xUeq(C), where x = 1.5 for methyl H atoms and x = 1.2 for all other atoms.
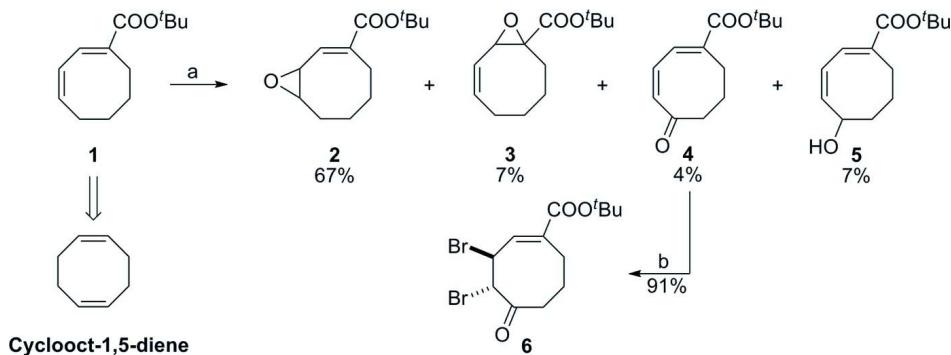
Figure 1

Reaction scheme for the synthesis of the title compound.
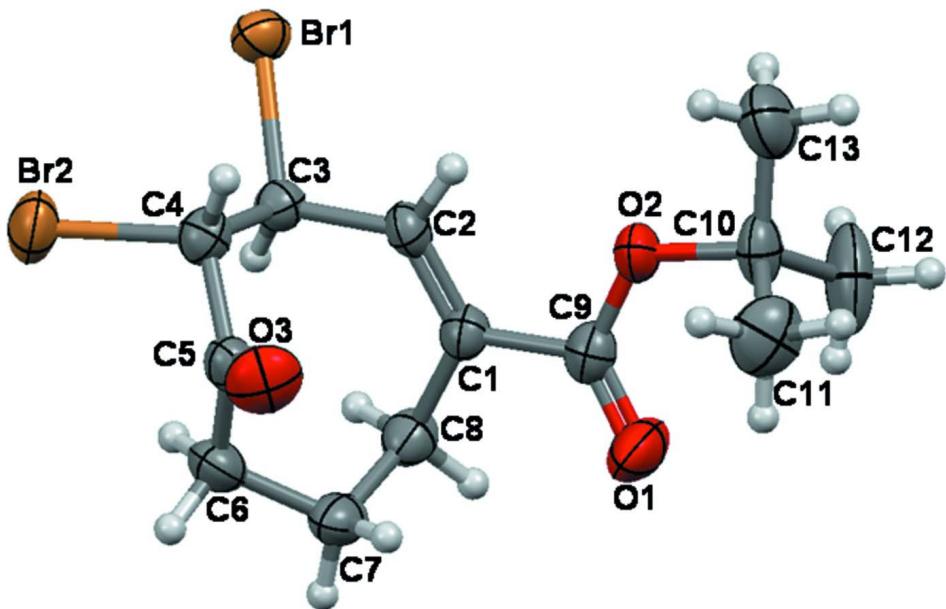
Figure 2

Molecular structure of $C_{13}H_{18}Br_2O_3$. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms are shown as spheres of arbitrary radius.
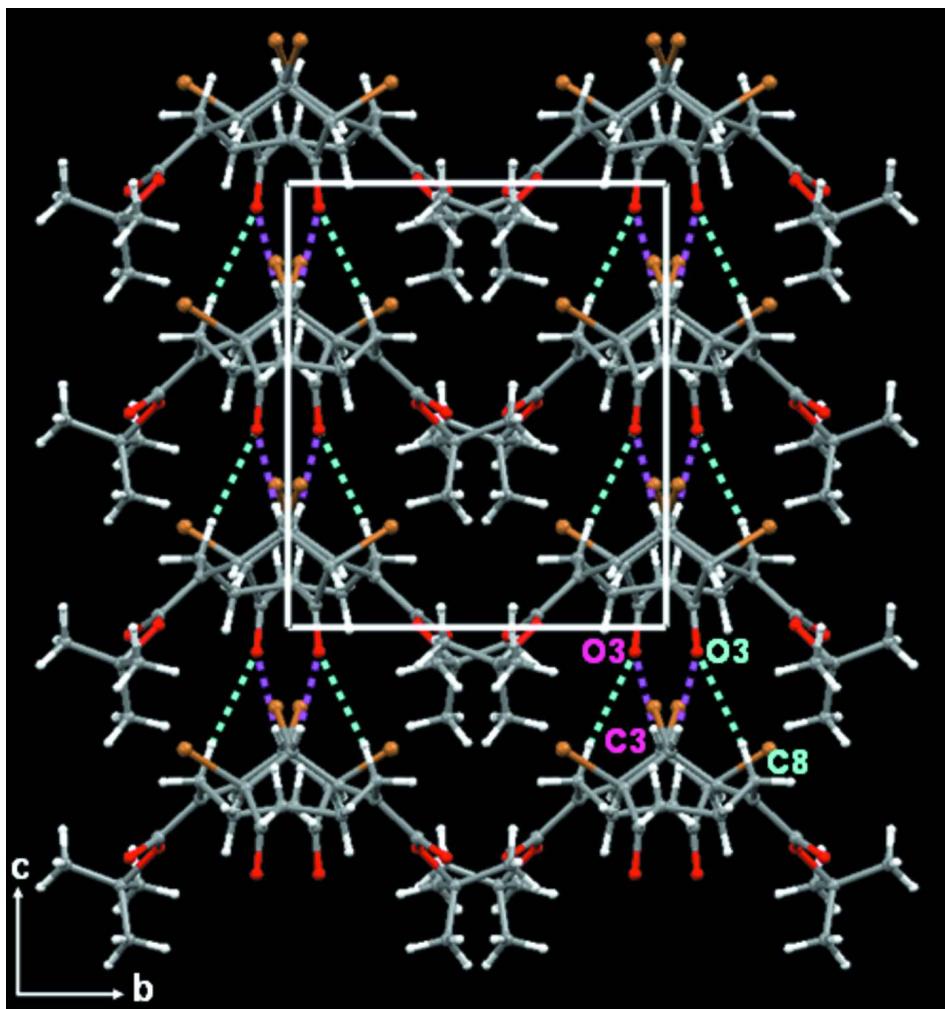
Figure 3
Crystal packing of $C_{13}H_{18}Br_2O_3$ view along *a* axis, showing intermolecular C—H···O interactions.

(3S*,4S*,E)-tert-Butyl 3,4-dibromo-5-oxocyclooct-1-enecarboxylate

Crystal data

$C_{13}H_{18}Br_2O_3$	$F(000) = 760$
$M_r = 382.09$	$D_x = 1.669$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Cu $K\alpha$ radiation, $\lambda = 1.54178$ Å
Hall symbol: P 2c -2ac	Cell parameters from 9578 reflections
$a = 14.0658$ (4) Å	$\theta = 4.6–66.5°$
$b = 9.5990$ (3) Å	$\mu = 6.76$ mm^{-1}
$c = 11.2657$ (3) Å	$T = 298$ K
$V = 1521.07$ (8) Å^3	Prismatic, colourless
$Z = 4$	$0.24 \times 0.14 \times 0.10$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	phi and ω scans
Radiation source: fine-focus sealed tube	Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
Graphite monochromator	$T_{min} = 0.370$, $T_{max} = 0.509$

10215 measured reflections	$\theta_{max} = 66.5°$, $\theta_{min} = 4.6°$	
2170 independent reflections	$h = -16 \rightarrow 16$	
2153 reflections with $I > 2\sigma(I)$	$k = -11 \rightarrow 11$	
$R_{int} = 0.048$	$l = -10 \rightarrow 13$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.029$	H-atom parameters constrained
$wR(F^2) = 0.075$	$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.2923P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	
2170 reflections	$(\Delta/\sigma)_{max} = 0.001$
166 parameters	$\Delta\rho_{max} = 0.32$ e Å^{-3}
1 restraint	$\Delta\rho_{min} = -0.46$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 803 Friedel pairs
Secondary atom site location: difference Fourier map	Absolute structure parameter: 0.06 (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)

	x	y	z	U_{iso}*/U_{eq}
Br1	0.76354 (2)	0.96781 (4)	0.67951 (5)	0.04630 (14)
Br2	0.94176 (3)	0.72748 (4)	0.77399 (5)	0.05922 (16)
O1	0.9457 (2)	1.4186 (4)	1.0183 (4)	0.0671 (10)
O2	0.79492 (19)	1.3427 (3)	0.9957 (3)	0.0429 (6)
O3	0.9590 (2)	0.9175 (4)	1.0552 (3)	0.0605 (8)
C1	0.9148 (3)	1.2311 (3)	0.8856 (4)	0.0368 (7)
C2	0.8510 (2)	1.1346 (3)	0.8551 (3)	0.0359 (7)
H2	0.7890	1.1450	0.8824	0.043*
C3	0.8733 (2)	1.0108 (3)	0.7796 (4)	0.0350 (7)
H3	0.9285	1.0313	0.7294	0.042*
C4	0.8964 (3)	0.8890 (4)	0.8617 (3)	0.0403 (7)
H4	0.8391	0.8632	0.9060	0.048*
C5	0.9749 (3)	0.9286 (4)	0.9494 (4)	0.0403 (8)
C6	1.0669 (3)	0.9841 (5)	0.9021 (4)	0.0481 (10)
H6B	1.0700	0.9652	0.8176	0.058*
H6A	1.1189	0.9347	0.9398	0.058*
C7	1.0806 (3)	1.1419 (5)	0.9221 (5)	0.0503 (10)
H7B	1.0685	1.1627	1.0050	0.060*
H7A	1.1463	1.1655	0.9057	0.060*

C8	1.0164 (3)	1.2335 (4)	0.8455 (4)	0.0455 (9)
H8B	1.0396	1.3286	0.8479	0.055*
H8A	1.0197	1.2018	0.7638	0.055*
C9	0.8876 (3)	1.3426 (3)	0.9735 (3)	0.0407 (8)
C10	0.7509 (3)	1.4418 (4)	1.0807 (5)	0.0488 (10)
C11	0.7934 (4)	1.4216 (6)	1.2022 (4)	0.0701 (13)
H11A	0.7917	1.3246	1.2227	0.105*
H11B	0.7576	1.4740	1.2594	0.105*
H11C	0.8581	1.4534	1.2020	0.105*
C12	0.7647 (6)	1.5884 (5)	1.0332 (7)	0.086 (2)
H12A	0.8314	1.6092	1.0295	0.130*
H12B	0.7338	1.6537	1.0849	0.130*
H12C	0.7377	1.5950	0.9552	0.130*
C13	0.6482 (4)	1.3957 (6)	1.0791 (6)	0.0723 (14)
H13A	0.6223	1.4091	1.0011	0.108*
H13B	0.6125	1.4497	1.1353	0.108*
H13C	0.6444	1.2988	1.1000	0.108*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0408 (2)	0.0566 (2)	0.0415 (2)	−0.00053 (14)	−0.0054 (2)	−0.01174 (18)
Br2	0.0757 (3)	0.0393 (2)	0.0626 (3)	0.01023 (16)	−0.0059 (2)	−0.0133 (2)
O1	0.0599 (18)	0.0619 (19)	0.079 (3)	−0.0142 (15)	0.0064 (16)	−0.0333 (17)
O2	0.0436 (13)	0.0401 (12)	0.0451 (15)	0.0075 (11)	0.0017 (12)	−0.0121 (11)
O3	0.0719 (19)	0.0752 (19)	0.0343 (17)	−0.0037 (15)	−0.0071 (14)	0.0051 (16)
C1	0.0403 (17)	0.0328 (15)	0.0374 (19)	0.0002 (13)	0.0039 (17)	−0.0032 (14)
C2	0.0355 (16)	0.0375 (15)	0.0347 (18)	0.0076 (13)	0.0050 (14)	−0.0044 (14)
C3	0.0347 (17)	0.0374 (14)	0.0330 (17)	−0.0026 (13)	0.0021 (16)	−0.0025 (15)
C4	0.0439 (18)	0.0394 (15)	0.038 (2)	−0.0016 (14)	0.0021 (17)	−0.0007 (14)
C5	0.0402 (19)	0.0435 (16)	0.037 (2)	0.0070 (16)	−0.0026 (16)	−0.0026 (16)
C6	0.0339 (19)	0.057 (2)	0.054 (3)	0.0070 (16)	−0.0030 (17)	−0.0068 (19)
C7	0.0347 (17)	0.060 (2)	0.056 (3)	−0.0034 (17)	0.0025 (17)	−0.0164 (19)
C8	0.044 (2)	0.0453 (17)	0.047 (2)	−0.0076 (15)	0.0098 (17)	−0.0082 (15)
C9	0.050 (2)	0.0352 (15)	0.037 (2)	0.0011 (15)	0.0018 (16)	−0.0017 (14)
C10	0.066 (2)	0.0397 (17)	0.040 (2)	0.0135 (17)	0.009 (2)	−0.0088 (18)
C11	0.088 (3)	0.082 (3)	0.041 (3)	−0.001 (3)	0.006 (2)	−0.006 (2)
C12	0.138 (5)	0.038 (2)	0.083 (4)	0.026 (3)	0.038 (4)	0.002 (2)
C13	0.063 (3)	0.076 (3)	0.078 (4)	0.019 (2)	0.005 (3)	−0.023 (3)

Geometric parameters (Å, º)

Br1—C3	1.956 (4)	C6—H6A	0.9700
Br2—C4	1.946 (4)	C7—C8	1.528 (7)
O1—C9	1.206 (5)	C7—H7B	0.9700
O2—C9	1.328 (5)	C7—H7A	0.9700
O2—C10	1.486 (5)	C8—H8B	0.9700
O3—C5	1.217 (5)	C8—H8A	0.9700

C1—C2	1.334 (5)	C10—C11	1.507 (7)
C1—C8	1.499 (5)	C10—C13	1.511 (7)
C1—C9	1.508 (5)	C10—C12	1.518 (7)
C2—C3	1.495 (5)	C11—H11A	0.9600
C2—H2	0.9300	C11—H11B	0.9600
C3—C4	1.526 (5)	C11—H11C	0.9600
C3—H3	0.9800	C12—H12A	0.9600
C4—C5	1.529 (6)	C12—H12B	0.9600
C4—H4	0.9800	C12—H12C	0.9600
C5—C6	1.498 (6)	C13—H13A	0.9600
C6—C7	1.543 (6)	C13—H13B	0.9600
C6—H6B	0.9700	C13—H13C	0.9600
C9—O2—C10	122.1 (3)	C1—C8—C7	112.6 (4)
C2—C1—C8	125.0 (3)	C1—C8—H8B	109.1
C2—C1—C9	119.5 (3)	C7—C8—H8B	109.1
C8—C1—C9	115.4 (3)	C1—C8—H8A	109.1
C1—C2—C3	123.9 (3)	C7—C8—H8A	109.1
C1—C2—H2	118.1	H8B—C8—H8A	107.8
C3—C2—H2	118.1	O1—C9—O2	125.9 (3)
C2—C3—C4	108.0 (3)	O1—C9—C1	122.2 (3)
C2—C3—Br1	109.3 (2)	O2—C9—C1	111.9 (3)
C4—C3—Br1	110.8 (2)	O2—C10—C11	109.7 (4)
C2—C3—H3	109.6	O2—C10—C13	101.7 (4)
C4—C3—H3	109.6	C11—C10—C13	110.7 (5)
Br1—C3—H3	109.6	O2—C10—C12	108.3 (4)
C3—C4—C5	110.8 (3)	C11—C10—C12	112.9 (5)
C3—C4—Br2	111.9 (3)	C13—C10—C12	113.0 (5)
C5—C4—Br2	106.8 (2)	C10—C11—H11A	109.5
C3—C4—H4	109.1	C10—C11—H11B	109.5
C5—C4—H4	109.1	H11A—C11—H11B	109.5
Br2—C4—H4	109.1	C10—C11—H11C	109.5
O3—C5—C6	122.5 (4)	H11A—C11—H11C	109.5
O3—C5—C4	118.6 (4)	H11B—C11—H11C	109.5
C6—C5—C4	118.9 (3)	C10—C12—H12A	109.5
C5—C6—C7	113.9 (3)	C10—C12—H12B	109.5
C5—C6—H6B	108.8	H12A—C12—H12B	109.5
C7—C6—H6B	108.8	C10—C12—H12C	109.5
C5—C6—H6A	108.8	H12A—C12—H12C	109.5
C7—C6—H6A	108.8	H12B—C12—H12C	109.5
H6B—C6—H6A	107.7	C10—C13—H13A	109.5
C8—C7—C6	114.1 (4)	C10—C13—H13B	109.5
C8—C7—H7B	108.7	H13A—C13—H13B	109.5
C6—C7—H7B	108.7	C10—C13—H13C	109.5
C8—C7—H7A	108.7	H13A—C13—H13C	109.5
C6—C7—H7A	108.7	H13B—C13—H13C	109.5
H7B—C7—H7A	107.6		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3ⁱ	0.98	2.57	3.525 (5)	165
C8—H8*A*···O3ⁱ	0.97	2.63	3.590 (6)	172

Symmetry code: (i) −*x*+2, −*y*+2, *z*−1/2.