Synthesis and structure of 2-oxo-2H-chromen-4-yl 4-bromobenzoate: work carried out as part of the CNRS AFRAMED project

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In the title compound, $C_{16}H_9BrO_4$, the dihedral angle between the chromen-2-one ring system (r.m.s. deviation = 0.006 Å) and the bromobenzene ring is 10.29 (6)°. In the crystal, the molecules are connected through C—H···O hydrogen bonds and π–π stacking interactions. According to a Hirshfeld surface analysis, H···H (22.4%), O···H/H···O (23.6%) and C···H/H···C (21%) interactions are the most significant contributors to the crystal packing.

1. AFRAMED and chemical context

This work was carried out as part of the CNRS AFRAMED project, which aims to train African Partners (young lecturers with permanent positions) in X-ray diffraction and provide regional laboratories, which serve as focal points to assist their colleagues for remote measurements (Abdel-Aal *et al.*, 2023). Coumarin derivatives remain one of our research priorities due to their versatile range of activities, such as anticoagulant, anti-inflammatory, antiviral, antimicrobial, anticancer, antioxidant (Todorov *et al.*, 2023), anti-glaucoma (Ziki *et al.*, 2023) and anti-Parkisonian effects (Kambo *et al.*, 2024). Here we report one result of this training: the synthesis, crystal structure and Hirshfeld surface analysis of the title coumarin derivative, $C_{16}H_9BrO_4$ (**I**).
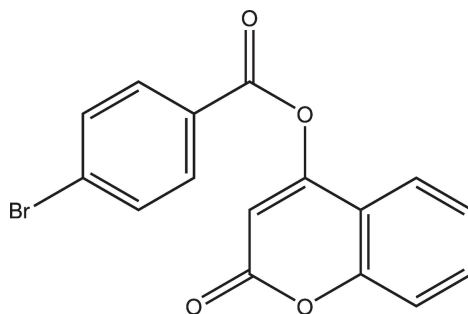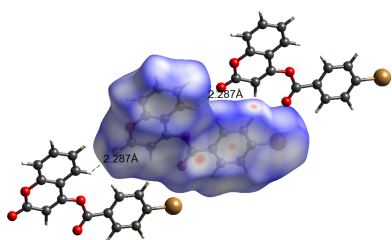
2. Structural commentary

Compound (**I**) crystallizes in the orthorhombic space group $P2_12_12_1$ with one molecule in the asymmetric unit (Fig. 1). The side chain is titled with respect to the chromen-2-one ring
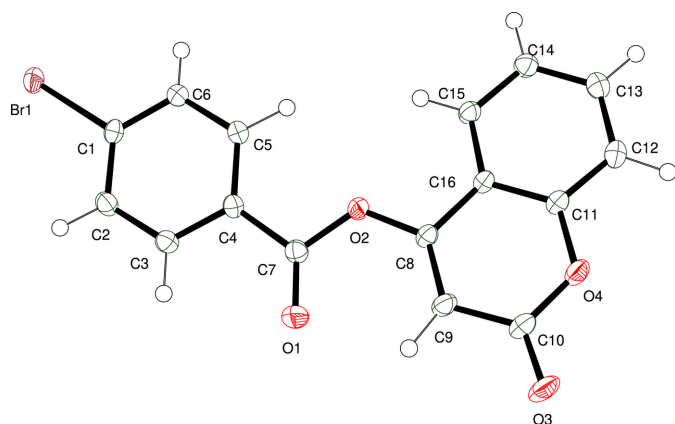
Figure 1
The molecular structure of (**I**) with displacement ellipsoids drawn at the 50% probability level.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C9—H9$\cdots$O1	1.00 (4)	2.26 (4)	2.788 (3)	111 (3)
C15—H15$\cdots$O3^i	0.97 (3)	2.36 (4)	3.079 (3)	131 (3)

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

centroids of the C8–C10/O4/C11/C16 and C11–C16 rings, respectively.

The intermolecular interactions in (**I**) were further quantified by Hirshfeld surface analysis (Fig. 3) using *Crystal-Explorer* (Spackman *et al.*, 2023). The interactions mentioned above are confirmed by the two-dimensional fingerprint plots for (**I**) (Fig. 4). The greatest contributions are from O$\cdots$H/H$\cdots$O (23.6%), H$\cdots$H (22.4%) and C$\cdots$H/H$\cdots$C (21%) while the Br$\cdots$H/H$\cdots$Br and Br$\cdots$C/C$\cdots$Br contacts contribute 9.6 and 6.4%, respectively.

system with torsion angles C9—C8—O2—C7 = −12.3 (4)° and C16—C8—O2—C7 = 169.9 (2)°. As expected, the chromen-2-one ring (C8—H16) system is almost planar, with a maximum deviation from the mean plane of 0.030 (2) Å for atom O3. The dihedral angle between this coumarin ring and the C1–C6 phenyl group in the 4-bromobenzoate moiety is 10.29 (6)°. An inspection of the bond lengths shows that there is a slight asymmetry of the electronic distribution around the pyrone ring, as shown by the differences between C8—C9 [1.350 (3) Å] and C9—C10 [1.452 (4) Å]. This suggests that the electron density is preferentially located in the formal C8=C9 double bond of the pyrone ring, as seen in other coumarin derivatives (Gomes *et al.*, 2016; Ouédraogo *et al.*, 2018). One intramolecular short contact exists between the C9—H9 methine group and atom O1 (Table 1).

4. Database survey

A search of the Cambridge Structural Database (CSD, version 5.43; update 3, September 2022; Groom *et al.*, 2016) for
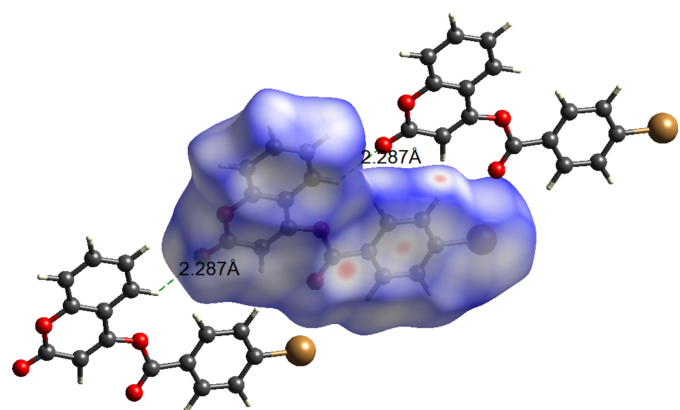
Figure 3
The Hirshfeld surface of (**I**) mapped over d_{norm}. Dotted lines (magenta) represent hydrogen bonds.

3. Supramolecular features and Hirshfeld surface analysis

In the crystal of (**I**), a weak C15—H15$\cdots$O3 hydrogen bond links the molecules into [1$\bar{1}$0] chains (Table 1, Fig. 2). The three-dimensional architecture is further consolidated by aromatic π-stacking interactions: the $Cg1\cdots Cg3(1 + x, y, z)$ separation is 3.8770 (19) Å, where $Cg1$ and $Cg3$ are the
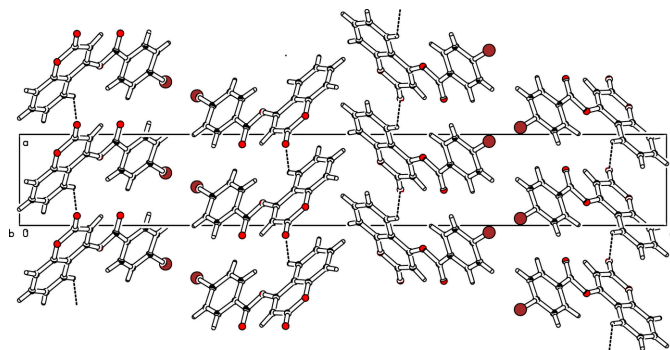

Figure 2
The unit-cell packing of (**I**) showing a hydrogen-bonded [1$\bar{1}$0] chain: dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen-bonding interactions have been omitted for clarity.

Figure 4
Fingerprint plots of (**I**): (*a*) H$\cdots$H, (*b*) O$\cdots$H, (*c*) C$\cdots$O, (*d*) C$\cdots$H, (*e*) Br$\cdots$O, (*f*) Br$\cdots$H and (*g*) Br$\cdots$C: d_i is the closest internal distance from a given point on the Hirshfeld surface and d_e is the closest external contact.

structures having a coumarin motif similar to (**I**) returned thirteen hits, including (7-chloro-2-oxo-2*H*-chromen-4-yl) methyldimethylcarbamodithioate (CSD refcode XUFGOW; Kavitha *et al.*, 2015); (6-bromo-2-oxo-2*H*-chromen-4-yl) methyl diethylcarbamodithioate (NUZJOJ; Vinduvahini *et al.*, 2016); (5,7-dimethyl-2-oxo-2*H*-chromen-4-yl)methyl morpholine-4-carbodithioate (UDOGIF01; Anitha *et al.*, 2016); (7-fluoro-2-oxo-2*H*-chromen-4-yl)methyl morpholine-4-carbodithioate (UYVEE; Anitha *et al.*, 2015); (7,8-dimethyl-2-oxo-2*H*-chromen-4-yl)methyl piperidine-1-carbodithioate (NAGWAW; Ravi *et al.*, 2016); methyl 2-[(2-oxo-2*H*-1-benzopyran-4-yl)amino]benzoate (DIWPAE; Hollauer *et al.*, 2023). In 2-oxo-2*H*-chromen-4-yl 4-(dimetylamino)benzoate (AYOXAO; Abou *et al.*, 2011), the benzoate ring is oriented at a dihedral angle of 43.43 (6)° with the chromene ring system while in 2-oxo-2*H*-chromen-4-yl 4-*tert*-butylbenzoate (GARHAK; Abou et *et al.*, 2012*b*), the benzene ring of the benzoate group is oriented at a dihedral angle of 60.70 (7)° with the chromene ring system. In 2-oxo-2*H*-chromen-4-yl 4-methoxybenzoate (PECVUQ; Abou *et al.*, 2012*a*), the chromen-2-one ring and the 4-methoxybenzoate side chain are inclined to one another at a dihedral angle of 69.82 (9)° and in 2-oxo-2*H*-chromen-4-yl 4-methylbenzoate (AFOQET; Abou *et al.*, 2013), the chromene-2-one ring and the 4-methylbenzoate side chain are inclined to one another at a dihedral angle of 64.79 (10)° in one molecule and 88.3 (1)° in the other. In 2-oxo-2*H*-chromen-4-yl propionate (AGAREH; Bibila Mayaya Bisseyou *et al.*, 2013), the 2-oxo-2*H*-chromene ring system and the non-H atom of the 4-substituent all lie on a crystallographic mirror plane. In 2-oxo-2*H*-1-benzopyran-4-yl 3,3-dimethylbutanoate (JOMHUS; Bationo *et al.*, 2024*a*), the 2-oxo-2*H*-1-benzopyran-4-yl 3,3-dimethylbutanoate the coumarin ring system is oriented at a dihedral angle of 56.24 (18)° with the butanoate moiety. In 2-oxo-2*H*-chromen-4-yl pentanoate (PONXUP; Bationo *et al.*, 2024*b*), the dihedral angle between the coumarin ring system and the pentanoate is 36.26 (8)°.

5. Synthesis and crystallization

To a solution of 4-bromobenzoyl chloride (6.2 mmol, 1.35 g) in dried tetrahydrofurn (30 ml) were added dried triethylamine (3 molar equivalents, 2.6 ml) and 4-hydroxycoumarin (6.17 mmol, 1.00 g) in small portions over 30 min. The mixture was then refluxed for 4 h and poured into 40 ml of chloroform. The solution was acidified with dilute (5%) hydrochloric acid until its discoloration was complete. The organic layers were extracted, concentrated under vacuum until a slight cloudiness was obtained and left in an ice bath. The resulting crude product was filtered off with suction, washed with petroleum ether and purified by recrystallization from a chloroform–hexane solvent mixture: yield 71%.

Colorless crystals of (**I**) suitable for data collection were obtained by recrystallization from acetone solution. The melting point was measured in an open capillary with a Cole–Parmer STUART MP. 800D Series-Melting S apparatus and is thus uncorrected, m.p. 423–425 K.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_9BrO_4$
M_r	345.14
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	100
a, b, c (Å)	5.4003 (11), 6.367 (2), 38.404 (8)
V (Å^3)	1320.4 (6)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	3.13
Crystal size (mm)	$0.27 \times 0.21 \times 0.13$
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.731, 0.895
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37156, 4975, 4880
R_{int}	0.034
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.768
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.064, 1.11
No. of reflections	4975
No. of parameters	226
No. of restraints	2
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.89, −0.87
Absolute structure	Flack x determined using 1967 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.008 (3)

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2013* (Sheldrick, 2015*b*).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms were located in difference-Fourier maps and their positions and U_{iso} values were freely refined.

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Computing details

2-Oxo-2*H*-chromen-4-yl 4-bromobenzoate

Crystal data

$C_{16}H_9BrO_4$	$D_x = 1.736$ Mg m^{-3}
$M_r = 345.14$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Orthorhombic, $P2_12_12_1$	Cell parameters from 4940 reflections
$a = 5.4003\ (11)$ Å	$\theta = 2.3$–$30.6°$
$b = 6.367\ (2)$ Å	$\mu = 3.13$ mm^{-1}
$c = 38.404\ (8)$ Å	$T = 100$ K
$V = 1320.4\ (6)$ Å^3	Prism, colourless
$Z = 4$	$0.27 \times 0.21 \times 0.13$ mm
$F(000) = 688$	

Data collection

Bruker D8 Venture diffractometer	4975 independent reflections
	4880 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.034$
Absorption correction: multi-scan (SADBAS; Krause *et al.*, 2015)	$\theta_{max} = 33.1°$, $\theta_{min} = 3.2°$
	$h = -7 \rightarrow 8$
$T_{min} = 0.731$, $T_{max} = 0.895$	$k = -9 \rightarrow 9$
37156 measured reflections	$l = -58 \rightarrow 58$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0042P)^2 + 1.5505P]$
Least-squares matrix: full	where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.029$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.064$	$\Delta\rho_{max} = 0.89$ e Å^{-3}
$S = 1.11$	$\Delta\rho_{min} = -0.87$ e Å^{-3}
4975 reflections	Absolute structure: Flack x determined using
226 parameters	1967 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et*
2 restraints	*al.*, 2013)
Hydrogen site location: difference Fourier map	Absolute structure parameter: 0.008 (3)
All H-atom parameters refined	

Special details

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{iso}*/U_{eq}$
Br1	0.42360 (5)	−0.17161 (4)	0.27424 (2)	0.02298 (6)
C5	0.3903 (4)	0.3282 (4)	0.34326 (6)	0.0189 (4)
O2	0.2448 (3)	0.6887 (3)	0.37643 (4)	0.0196 (3)
C2	0.0989 (5)	0.1701 (5)	0.28876 (6)	0.0209 (4)
O3	−0.1051 (4)	1.3465 (3)	0.41125 (5)	0.0301 (4)
C8	0.2098 (5)	0.8636 (4)	0.39677 (6)	0.0173 (4)
O4	0.2035 (4)	1.2090 (3)	0.44169 (5)	0.0231 (4)
O1	−0.1096 (4)	0.7132 (3)	0.34419 (6)	0.0291 (5)
C7	0.0804 (6)	0.6245 (4)	0.35106 (6)	0.0189 (4)
C3	0.0272 (4)	0.3517 (4)	0.30635 (6)	0.0192 (4)
C6	0.4630 (5)	0.1452 (4)	0.32592 (6)	0.0188 (4)
C15	0.5666 (5)	0.7219 (4)	0.43182 (6)	0.0190 (4)
C4	0.1718 (5)	0.4307 (4)	0.33372 (6)	0.0175 (4)
C9	0.0415 (5)	1.0175 (4)	0.39187 (7)	0.0209 (5)
C12	0.5369 (5)	1.0775 (4)	0.47484 (7)	0.0225 (5)
C11	0.3749 (5)	1.0522 (4)	0.44687 (6)	0.0183 (4)
C10	0.0358 (5)	1.1996 (4)	0.41461 (7)	0.0219 (5)
C16	0.3865 (5)	0.8749 (4)	0.42525 (6)	0.0162 (4)
C14	0.7299 (5)	0.7462 (4)	0.45953 (7)	0.0217 (5)
C1	0.3163 (5)	0.0707 (4)	0.29888 (6)	0.0179 (4)
C13	0.7138 (5)	0.9227 (5)	0.48089 (7)	0.0228 (5)
H15	0.579 (7)	0.600 (5)	0.4167 (8)	0.019 (8)*
H9	−0.096 (8)	1.018 (6)	0.3743 (9)	0.029 (9)*
H2	−0.004 (4)	0.106 (4)	0.2698 (6)	0.026 (9)*
H13	0.823 (6)	0.936 (5)	0.5001 (9)	0.021 (9)*
H3	−0.124 (6)	0.423 (5)	0.2999 (8)	0.015 (8)*
H12	0.518 (6)	1.206 (6)	0.4896 (9)	0.027 (9)*
H6	0.597 (7)	0.074 (5)	0.3328 (8)	0.016 (8)*
H14	0.851 (7)	0.640 (6)	0.4629 (9)	0.026 (9)*
H5	0.492 (7)	0.386 (6)	0.3636 (10)	0.037 (11)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02724 (11)	0.02177 (10)	0.01994 (10)	0.00231 (11)	−0.00180 (10)	−0.00603 (9)
C5	0.0192 (10)	0.0202 (10)	0.0172 (9)	−0.0005 (10)	−0.0020 (8)	−0.0020 (9)
O2	0.0223 (8)	0.0163 (8)	0.0201 (8)	0.0048 (7)	−0.0044 (6)	−0.0035 (7)
C2	0.0224 (11)	0.0223 (10)	0.0181 (9)	0.0002 (12)	−0.0044 (8)	0.0000 (9)
O3	0.0362 (11)	0.0234 (9)	0.0308 (9)	0.0166 (10)	0.0030 (8)	−0.0011 (8)

C8	0.0202 (10)	0.0147 (10)	0.0169 (9)	0.0023 (8)	0.0016 (8)	0.0000 (7)
O4	0.0279 (9)	0.0174 (9)	0.0238 (8)	0.0066 (7)	0.0018 (7)	−0.0035 (7)
O1	0.0261 (10)	0.0266 (10)	0.0345 (10)	0.0087 (8)	−0.0092 (8)	−0.0063 (8)
C7	0.0213 (10)	0.0174 (10)	0.0182 (9)	0.0006 (9)	−0.0019 (9)	0.0013 (7)
C3	0.0190 (10)	0.0192 (11)	0.0194 (9)	0.0003 (9)	−0.0020 (8)	0.0010 (8)
C6	0.0185 (11)	0.0193 (11)	0.0185 (9)	0.0014 (9)	−0.0014 (8)	−0.0014 (8)
C15	0.0213 (10)	0.0177 (10)	0.0180 (9)	0.0037 (9)	−0.0007 (9)	−0.0007 (7)
C4	0.0199 (10)	0.0177 (10)	0.0148 (9)	−0.0009 (9)	−0.0008 (8)	−0.0002 (8)
C9	0.0231 (12)	0.0187 (10)	0.0210 (10)	0.0073 (9)	0.0002 (9)	0.0000 (8)
C12	0.0266 (14)	0.0215 (11)	0.0195 (10)	−0.0011 (10)	0.0015 (9)	−0.0045 (9)
C11	0.0207 (12)	0.0162 (10)	0.0181 (10)	0.0020 (8)	0.0019 (8)	−0.0005 (8)
C10	0.0248 (12)	0.0177 (11)	0.0231 (10)	0.0057 (9)	0.0043 (9)	0.0003 (8)
C16	0.0190 (11)	0.0144 (9)	0.0150 (9)	0.0017 (8)	0.0021 (8)	−0.0002 (7)
C14	0.0228 (11)	0.0225 (11)	0.0199 (11)	0.0037 (10)	−0.0014 (9)	0.0002 (9)
C1	0.0216 (11)	0.0178 (10)	0.0141 (9)	−0.0010 (9)	0.0029 (8)	−0.0009 (8)
C13	0.0242 (12)	0.0247 (12)	0.0195 (11)	−0.0020 (10)	−0.0020 (9)	−0.0015 (9)

Geometric parameters (Å, º)

Br1—C1	1.900 (3)	C3—C4	1.403 (3)
C5—C4	1.397 (4)	C3—H3	0.97 (3)
C5—C6	1.398 (4)	C6—C1	1.389 (3)
C5—H5	1.02 (4)	C6—H6	0.89 (4)
O2—C8	1.373 (3)	C15—C14	1.391 (4)
O2—C7	1.380 (3)	C15—C16	1.400 (3)
C2—C1	1.389 (4)	C15—H15	0.97 (3)
C2—C3	1.394 (4)	C9—C10	1.452 (4)
C2—H2	1.002 (12)	C9—H9	1.00 (4)
O3—C10	1.213 (3)	C12—C13	1.392 (4)
C8—C9	1.350 (3)	C12—C11	1.394 (4)
C8—C16	1.453 (3)	C12—H12	1.00 (4)
O4—C11	1.376 (3)	C11—C16	1.403 (3)
O4—C10	1.380 (3)	C14—C13	1.394 (4)
O1—C7	1.200 (3)	C14—H14	0.95 (4)
C7—C4	1.487 (3)	C13—H13	0.95 (3)
C4—C5—C6	120.1 (2)	C3—C4—C7	116.6 (2)
C4—C5—H5	119 (2)	C8—C9—C10	120.7 (2)
C6—C5—H5	121 (2)	C8—C9—H9	127 (2)
C8—O2—C7	123.6 (2)	C10—C9—H9	113 (2)
C1—C2—C3	118.5 (2)	C13—C12—C11	118.5 (2)
C1—C2—H2	119.2 (9)	C13—C12—H12	124 (2)
C3—C2—H2	122.3 (9)	C11—C12—H12	118 (2)
C9—C8—O2	127.0 (2)	O4—C11—C12	116.7 (2)
C9—C8—C16	120.7 (2)	O4—C11—C16	121.9 (2)
O2—C8—C16	112.2 (2)	C12—C11—C16	121.4 (2)
C11—O4—C10	121.3 (2)	O3—C10—O4	117.3 (2)
O1—C7—O2	124.5 (2)	O3—C10—C9	124.4 (3)

O1—C7—C4	125.2 (2)	O4—C10—C9	118.3 (2)
O2—C7—C4	110.4 (2)	C15—C16—C11	119.0 (2)
C2—C3—C4	120.4 (2)	C15—C16—C8	123.9 (2)
C2—C3—H3	120.1 (19)	C11—C16—C8	117.2 (2)
C4—C3—H3	119.5 (19)	C15—C14—C13	120.0 (3)
C1—C6—C5	118.7 (2)	C15—C14—H14	118 (2)
C1—C6—H6	120 (2)	C13—C14—H14	122 (2)
C5—C6—H6	121 (2)	C2—C1—C6	122.4 (2)
C14—C15—C16	120.1 (2)	C2—C1—Br1	119.22 (19)
C14—C15—H15	120 (2)	C6—C1—Br1	118.39 (19)
C16—C15—H15	120 (2)	C12—C13—C14	121.0 (3)
C5—C4—C3	119.9 (2)	C12—C13—H13	120 (2)
C5—C4—C7	123.4 (2)	C14—C13—H13	119 (2)
C7—O2—C8—C9	−12.3 (4)	C11—O4—C10—C9	1.0 (4)
C7—O2—C8—C16	169.9 (2)	C8—C9—C10—O3	177.9 (3)
C8—O2—C7—O1	−0.3 (4)	C8—C9—C10—O4	−1.3 (4)
C8—O2—C7—C4	179.9 (2)	C14—C15—C16—C11	0.3 (4)
C1—C2—C3—C4	−0.3 (4)	C14—C15—C16—C8	179.5 (2)
C4—C5—C6—C1	1.1 (4)	O4—C11—C16—C15	179.5 (2)
C6—C5—C4—C3	−1.0 (4)	C12—C11—C16—C15	−0.7 (4)
C6—C5—C4—C7	179.4 (2)	O4—C11—C16—C8	0.3 (3)
C2—C3—C4—C5	0.6 (4)	C12—C11—C16—C8	−179.9 (2)
C2—C3—C4—C7	−179.8 (2)	C9—C8—C16—C15	−179.7 (2)
O1—C7—C4—C5	−177.8 (3)	O2—C8—C16—C15	−1.8 (3)
O2—C7—C4—C5	2.0 (3)	C9—C8—C16—C11	−0.6 (4)
O1—C7—C4—C3	2.5 (4)	O2—C8—C16—C11	177.3 (2)
O2—C7—C4—C3	−177.7 (2)	C16—C15—C14—C13	0.2 (4)
O2—C8—C9—C10	−176.5 (2)	C3—C2—C1—C6	0.4 (4)
C16—C8—C9—C10	1.0 (4)	C3—C2—C1—Br1	−177.87 (19)
C10—O4—C11—C12	179.6 (2)	C5—C6—C1—C2	−0.8 (4)
C10—O4—C11—C16	−0.6 (4)	C5—C6—C1—Br1	177.48 (19)
C13—C12—C11—O4	−179.7 (2)	C11—C12—C13—C14	0.1 (4)
C13—C12—C11—C16	0.5 (4)	C15—C14—C13—C12	−0.5 (4)
C11—O4—C10—O3	−178.2 (2)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···O1	1.00 (4)	2.26 (4)	2.788 (3)	111 (3)
C15—H15···O3[i]	0.97 (3)	2.36 (4)	3.079 (3)	131 (3)

Symmetry code: (i) x+1, y−1, z.