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2-Oxo-2H-chromen-4-yl propionate

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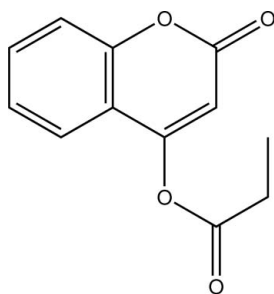
Received 7 June 2013; accepted 12 June 2013

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(I) = 0.000$ Å; disorder in main residue; R factor = 0.056; wR factor = 0.146; data-to-parameter ratio = 14.6.

In the title compound, $C_{12}H_{10}O_4$, the atoms of the 2-oxo-2H-chromene ring system and the non-H atoms of the 4-substituent all lie on a crystallographic mirror plane. The molecular structure exhibits an intramolecular $C-H \cdots O$ hydrogen bond, which generates an $S(6)$ ring. In the crystal, molecules form $R_3^2(12)$ trimeric units via $C-H \cdots O$ interactions which propagate into layers parallel to the ac plane. These layers are linked by weak $C-H \cdots O$ interactions along the $[010]$ direction, generating a three-dimensional network.

Related literature

For the biological activity of coumarin derivatives, see: Abernethy (1969); Wang *et al.* (2001); Yu *et al.* (2003, 2007); Vukovic *et al.* (2010). For industrial applications, see: O'Kennedy & Thornes (1997); Lakshmi *et al.* (1995). For a related structure, see: Abou *et al.* (2012). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For thermal motion of carbonyl group oxygen atoms, see: Braga & Koetzle (1988).



Experimental

Crystal data

$C_{12}H_{10}O_4$
 $M_r = 218.20$

Orthorhombic, $Pnma$
 $a = 9.2834$ (3) Å

$b = 6.7081$ (2) Å
 $c = 16.8068$ (6) Å
 $V = 1046.63$ (6) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 298$ K
 $0.40 \times 0.40 \times 0.20$ mm

Data collection

Nonius KappaCCD diffractometer
8092 measured reflections
1431 independent reflections

1155 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.146$
 $S = 1.06$
1431 reflections

98 parameters
H-atom parameters constrained
 $\Delta\rho_{max} = 0.24$ e Å⁻³
 $\Delta\rho_{min} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C2-H2 \cdots O4$	0.93	2.21	2.800 (3)	121
$C6-H6 \cdots O2^i$	0.93	2.48	3.376 (3)	161
$C8-H8 \cdots O2^{ii}$	0.93	2.71	3.394 (3)	131

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

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *pubCIF* (Westrip, 2010) and *WinGX* (Farrugia, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2203).

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supporting information

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2-Oxo-2H-chromen-4-yl propionate

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S1. Comment

The 2-Oxo-2H-chromene ring system derivatives commonly called coumarin derivatives attracted significant attention because of their interesting biological profile including anti-HIV (Yu *et al.*, 2003; Yu *et al.*, 2007), anti-coagulant (Abernethy, 1969), anti-oxidant (Vukovic *et al.*, 2010), anti-tumor (Wang *et al.*, 2001) properties.

They found applications in cosmetic and food industries (O'Kennedy & Thornes, 1997) and are also potential laser dyes (Lakshmi *et al.*, 1995). Owing its versatile properties, coumarin ring system has become a hub nucleus in the developing of new molecules in organic, medicinal and material chemistry. We have synthesized novel coumarin derivatives substituted at position 4 in order to explore the new properties of this compound class. Herein, we report single-crystal structure of title compound.

The molecular structure of title compound (and its atomic numbering scheme) is illustrated in Fig. 1. As expected, the coumarin moiety is planar as shown in the recent X-ray diffraction analysis of 4-substituted coumarin derivative (Abou *et al.*, 2012). Analysis of bond length values of aromatic ring indicate the existence of a delocalized π -electron cloud in this one. In this structure, except the H atoms of the substituent groups, all other atoms lie in a crystallographic mirror plane ($x, y = 1/4, z$). We also note the existence of an intramolecular C—H $\cdots$ O hydrogen bond which generates a S(6) ring motif (Bernstein *et al.*, 1995).

In the three-dimensional crystal packing, molecules form cyclic trimers of R_3^2 (12) motifs (Bernstein *et al.*, 1995) via two independent intermolecular C—H $\cdots$ O hydrogen bond interactions along the a axis (Fig. 2). These trimolecular aggregates propagate into parallel layers to the ac plane (Fig. 3). These layers are additionally stabilized by weak intermolecular C—H $\cdots$ O interactions along [010] direction.

S2. Experimental

To a solution of propionic chloride (125 mmol) in dried diethyl ether (300 ml) was added dried pyridine (4 ml) and 4-hydroxycoumarin (120 mmol) in small portions over 30 min. The mixture was then refluxed for 3 h and poured in 300 ml of chloroform. The solution was acidified with dilute hydrochloric acid until the pH was 2–3. The organic layer was extracted, washed with water, dried over MgSO_4 and the solvent removed. The crude product was recrystallized from acetone. Colourless single crystals of the title compound were obtained in a good yield: 69.7%; m.p. 358–359 K.

S3. Refinement

The structure solution program *SIR2004* (Burla *et al.*, 2005) was used to solve the structure. The .res file shows that all non H atoms lie on special positions ($x, 1/4, z$) with a site-occupancy factor of 0.5. As the program *SHELXL97* (Sheldrick, 2008) will automatically work out and apply the appropriate positional, s.o.f. and U^{ij} constraints for any special positions, we have not refined these non H atoms parameters with further constraints. However, omitted from the refinement

because of bad disagreements were (7 0 4), (7 2 4), (4 1 1), (1 0 1), (0 0 2).

H atoms were placed in calculated positions [$C-H = 0.93$ (aromatic), 0.96 (methyl group) or 0.97 Å (methylene group)] and refined using a riding model approximation with $U_{iso}(H)$ constrained to 1.2 (aromatic and methylene group) or 1.5 (methyl group) times U_{eq} of the respective parent atom.

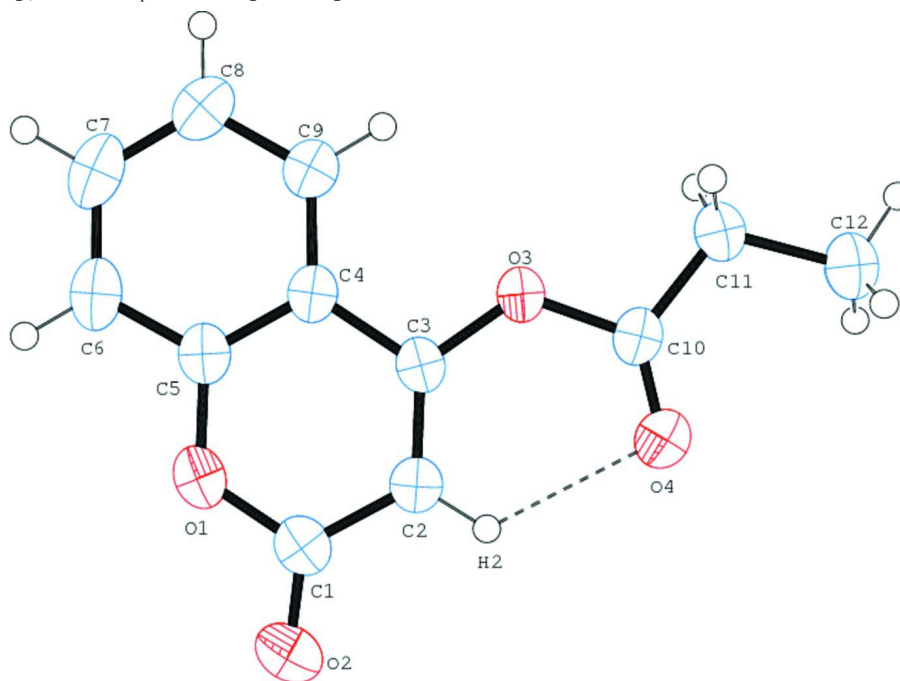
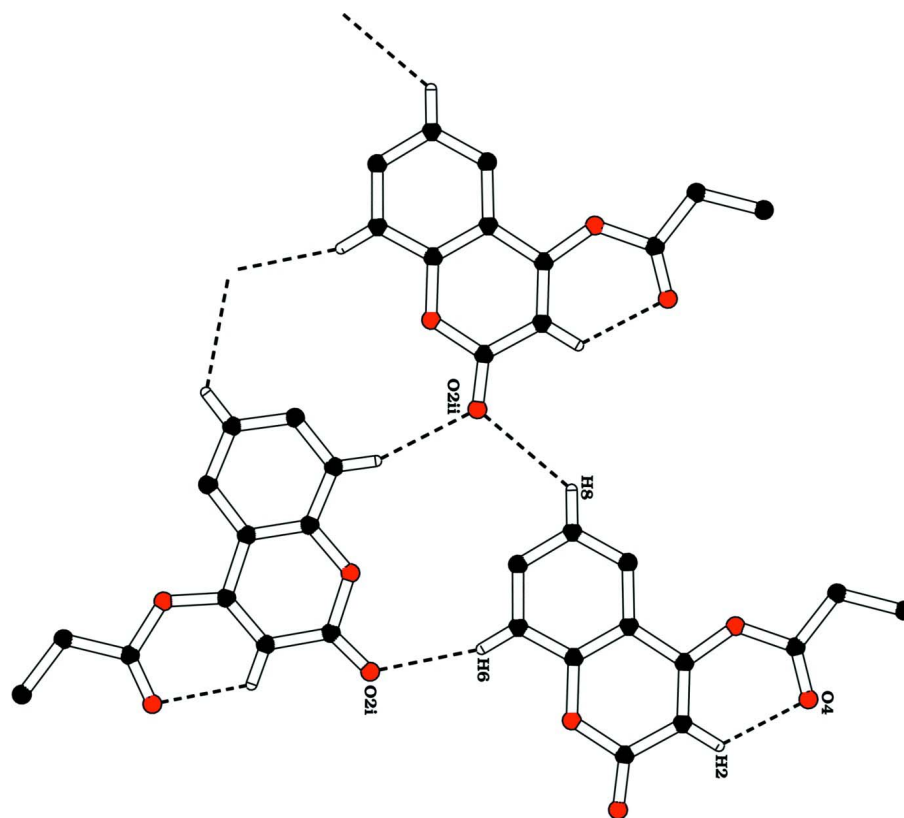


Figure 1

The molecular structure of the title compound and the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius. Dashed lines indicate a hydrogen bond.

**Figure 2**

Part of the crystal packing of the title compound showing the formation of $R_3^2(12)$ cyclic trimers. Dashed lines indicate hydrogen bond contacts. H atoms not involved in hydrogen bond interactions have been omitted for clarity.

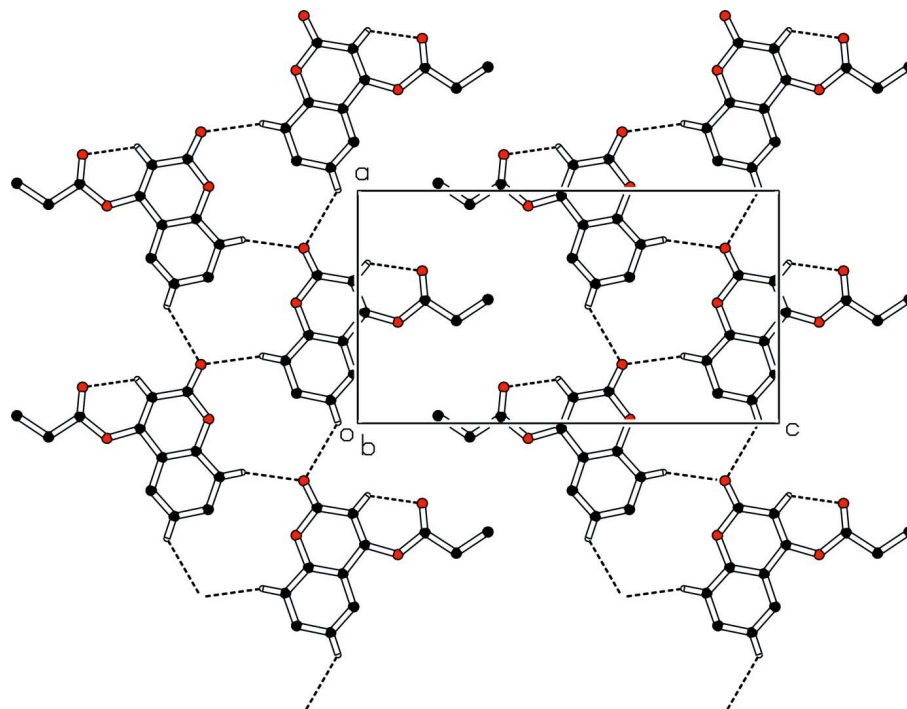


Figure 3

Crystal packing of the title compound viewed down the *b* axis, showing parallel layers in the *ac* plane. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen bond interactions have been omitted for clarity.

2-Oxo-2H-chromen-4-yl propionic acid

Crystal data

$C_{12}H_{10}O_4$

$M_r = 218.20$

Orthorhombic, *Pnma*

Hall symbol: -P 2ac 2n

$a = 9.2834(3) \text{ \AA}$

$b = 6.7081(2) \text{ \AA}$

$c = 16.8068(6) \text{ \AA}$

$V = 1046.63(6) \text{ \AA}^3$

$Z = 4$

$F(000) = 456$

$D_x = 1.385 \text{ Mg m}^{-3}$

Melting point = 358–359 K

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

Cell parameters from 8092 reflections

$\theta = 3.3\text{--}29.0^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Parallelepiped, colourless

$0.40 \times 0.40 \times 0.20 \text{ mm}$

Data collection

Nonius KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

8092 measured reflections

1431 independent reflections

1155 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\text{max}} = 29.0^\circ$, $\theta_{\text{min}} = 3.3^\circ$

$h = -12 \rightarrow 12$

$k = -8 \rightarrow 8$

$l = -22 \rightarrow 22$

*Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.056$ $wR(F^2) = 0.146$ $S = 1.06$

1431 reflections

98 parameters

0 restraints

88 constraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.3235P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$ $\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$ *Special details*

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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. The *DENZO* image processing package used during process data may have problems with certain strong reflections. These reflections are often excluded from the data set to result in _diffn_measured_fraction_theta_full Low (0.974 in our investigation study). However, it presents no problem in the refinement since the data-to-parameter ratio is superior to 10.

In the initial refinement, extinction correction (EXTI) has been applied because *SHELXL* has suggested it; but in the last cycles of the refinement, the EXTI instruction has been removed because of *PLATON checkCIF* reports mentioning extinction parameter within range (2.20σ).

The non H atoms lie in the mirror plane at $y = 1/4$. Therefore the U^{ij} constraints ($U_{12} = U_{23} = 0$) generated automatically by *SHELXL* for this special positions ($x, 1/4, z$) in the space group *Pnma* is responsible for the elongated thermal ellipsoids in the $[010]$ direction causing a large U_3/U_1 ratio for the average $U(i,j)$ tensor (2.4).

The low U_{eq} as compared to neighbors for atom C10 may be caused by the carbonyl bond in which the oxygen atom vibrates more than the carbon atom (Braga & Koetzle, 1988). Moreover, the decrease of U_{eq} from C12 to C10 of the propanoate substituent originates from the minor unresolved disordered H atoms bonded to the non disordered carbon atom C12 (split H atoms) revealed by manual inspection of *PLATON* (Spek, 2009) may partly justify this low U_{eq} of C10.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}	Occ. (<1)
O3	0.06438 (15)	0.2500	0.59531 (8)	0.0595 (5)	
O1	−0.01804 (18)	0.2500	0.35401 (8)	0.0607 (5)	
C4	0.1469 (2)	0.2500	0.46462 (11)	0.0434 (4)	
C3	0.0233 (2)	0.2500	0.51696 (10)	0.0428 (4)	
C2	−0.1113 (2)	0.2500	0.48839 (12)	0.0481 (5)	
H2	−0.1890	0.2500	0.5233	0.058*	
C10	−0.0290 (2)	0.2500	0.65979 (11)	0.0460 (5)	
C5	0.1204 (2)	0.2500	0.38352 (12)	0.0461 (5)	
O4	−0.15508 (17)	0.2500	0.65323 (9)	0.0695 (5)	
C11	0.0580 (2)	0.2500	0.73405 (11)	0.0616 (7)	
H11A	0.1197	0.3668	0.7343	0.074*	0.50
H11B	0.1197	0.1332	0.7343	0.074*	0.50

C9	0.2892 (2)	0.2500	0.49029 (13)	0.0633 (7)	
H9	0.3096	0.2500	0.5445	0.076*	
O2	−0.25262 (19)	0.2500	0.37225 (11)	0.0921 (8)	
C1	−0.1362 (2)	0.2500	0.40383 (13)	0.0579 (6)	
C8	0.4004 (3)	0.2500	0.43576 (16)	0.0765 (9)	
H8	0.4954	0.2500	0.4532	0.092*	
C12	−0.0325 (3)	0.2500	0.80905 (13)	0.0723 (8)	
H12A	−0.0996	0.1412	0.8072	0.108*	0.50
H12B	−0.0842	0.3735	0.8129	0.108*	0.50
H12C	0.0290	0.2352	0.8546	0.108*	0.50
C6	0.2310 (3)	0.2500	0.32839 (13)	0.0603 (6)	
H6	0.2111	0.2500	0.2742	0.072*	
C7	0.3703 (3)	0.2500	0.35516 (15)	0.0687 (7)	
H7	0.4457	0.2500	0.3187	0.082*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0399 (8)	0.1082 (14)	0.0304 (7)	0.000	0.0025 (5)	0.000
O1	0.0549 (9)	0.0936 (12)	0.0336 (7)	0.000	−0.0011 (6)	0.000
C4	0.0423 (10)	0.0528 (11)	0.0351 (9)	0.000	0.0050 (7)	0.000
C3	0.0420 (10)	0.0554 (11)	0.0311 (8)	0.000	0.0007 (7)	0.000
C2	0.0401 (10)	0.0665 (13)	0.0376 (9)	0.000	0.0013 (7)	0.000
C10	0.0409 (10)	0.0616 (12)	0.0354 (9)	0.000	0.0062 (7)	0.000
C5	0.0485 (11)	0.0532 (11)	0.0368 (9)	0.000	0.0036 (8)	0.000
O4	0.0419 (8)	0.1227 (16)	0.0439 (8)	0.000	0.0053 (6)	0.000
C11	0.0468 (12)	0.1050 (19)	0.0331 (10)	0.000	0.0013 (8)	0.000
C9	0.0432 (11)	0.104 (2)	0.0432 (10)	0.000	0.0039 (9)	0.000
O2	0.0540 (10)	0.173 (2)	0.0496 (9)	0.000	−0.0147 (8)	0.000
C1	0.0493 (12)	0.0841 (16)	0.0402 (10)	0.000	−0.0031 (9)	0.000
C8	0.0440 (12)	0.126 (3)	0.0589 (15)	0.000	0.0111 (10)	0.000
C12	0.0577 (14)	0.123 (2)	0.0363 (10)	0.000	0.0053 (9)	0.000
C6	0.0654 (14)	0.0757 (16)	0.0397 (10)	0.000	0.0133 (10)	0.000
C7	0.0603 (14)	0.0908 (18)	0.0550 (13)	0.000	0.0232 (11)	0.000

Geometric parameters (Å, °)

O3—C3	1.371 (2)	C11—H11A	0.9700
O3—C10	1.388 (2)	C11—H11B	0.9700
O1—C5	1.378 (3)	C9—C8	1.380 (3)
O1—C1	1.380 (3)	C9—H9	0.9300
C4—C5	1.385 (3)	O2—C1	1.204 (3)
C4—C9	1.390 (3)	C8—C7	1.383 (4)
C4—C3	1.446 (3)	C8—H8	0.9300
C3—C2	1.339 (3)	C12—H12A	0.9600
C2—C1	1.440 (3)	C12—H12B	0.9600
C2—H2	0.9300	C12—H12C	0.9600
C10—O4	1.176 (3)	C6—C7	1.370 (4)

C10—C11	1.487 (3)	C6—H6	0.9300
C5—C6	1.383 (3)	C7—H7	0.9300
C11—C12	1.515 (3)		
C3—O3—C10	125.20 (15)	C12—C11—H11A	108.9
C5—O1—C1	121.52 (16)	C10—C11—H11B	108.9
C5—C4—C9	118.32 (18)	C12—C11—H11B	108.9
C5—C4—C3	117.24 (17)	H11A—C11—H11B	107.7
C9—C4—C3	124.44 (18)	C8—C9—C4	120.3 (2)
C2—C3—O3	127.17 (17)	C8—C9—H9	119.9
C2—C3—C4	121.50 (17)	C4—C9—H9	119.9
O3—C3—C4	111.33 (16)	O2—C1—O1	116.48 (19)
C3—C2—C1	120.26 (18)	O2—C1—C2	125.4 (2)
C3—C2—H2	119.9	O1—C1—C2	118.13 (18)
C1—C2—H2	119.9	C9—C8—C7	120.0 (2)
O4—C10—O3	123.27 (18)	C9—C8—H8	120.0
O4—C10—C11	128.30 (18)	C7—C8—H8	120.0
O3—C10—C11	108.43 (16)	C7—C6—C5	118.8 (2)
O1—C5—C6	116.82 (19)	C7—C6—H6	120.6
O1—C5—C4	121.34 (18)	C5—C6—H6	120.6
C6—C5—C4	121.8 (2)	C6—C7—C8	120.8 (2)
C10—C11—C12	113.39 (19)	C6—C7—H7	119.6
C10—C11—H11A	108.9	C8—C7—H7	119.6
C10—O3—C3—C2	0.0	C3—C4—C5—C6	180.0
C10—O3—C3—C4	180.0	O4—C10—C11—C12	0.0
C5—C4—C3—C2	0.0	O3—C10—C11—C12	180.0
C9—C4—C3—C2	180.0	C5—C4—C9—C8	0.0
C5—C4—C3—O3	180.0	C3—C4—C9—C8	180.0
C9—C4—C3—O3	0.0	C5—O1—C1—O2	180.0
O3—C3—C2—C1	180.0	C5—O1—C1—C2	0.0
C4—C3—C2—C1	0.0	C3—C2—C1—O2	180.0
C3—O3—C10—O4	0.0	C3—C2—C1—O1	0.0
C3—O3—C10—C11	180.0	C4—C9—C8—C7	0.0
C1—O1—C5—C6	180.0	O1—C5—C6—C7	180.0
C1—O1—C5—C4	0.0	C4—C5—C6—C7	0.0
C9—C4—C5—O1	180.0	C5—C6—C7—C8	0.0
C3—C4—C5—O1	0.0	C9—C8—C7—C6	0.0
C9—C4—C5—C6	0.0		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2 $\cdots$ O4	0.93	2.21	2.800 (3)	121
C6—H6 $\cdots$ O2 ⁱ	0.93	2.48	3.376 (3)	161
C8—H8 $\cdots$ O2 ⁱⁱ	0.93	2.71	3.394 (3)	131

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