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Synthesis, structure and Hirshfeld surface analysis of 2-oxo-2*H*-chromen-4-yl pentanoate

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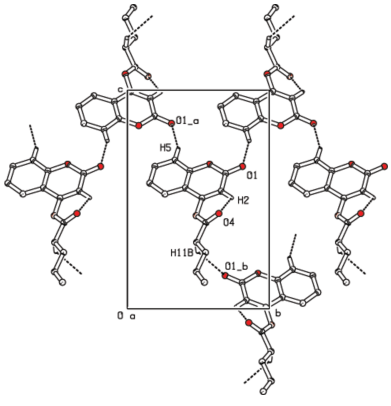
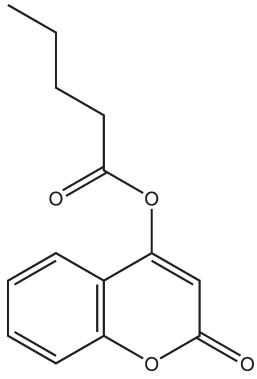
In the title compound, $C_{14}H_{14}O_4$, the dihedral angle between the coumarin ring system (r.m.s deviation = 0.016 Å) and the pentanoate ring is 36.26 (8)°. A short intramolecular C—H···O contact of 2.40 Å is observed. Hirshfeld surface analysis reveals that 46.1% of the intermolecular interactions are from H···H contacts, 28.6% are from H···O/O···H contacts and 14.7% are from H···C/C···H.

1. Chemical context

Coumarins are naturally occurring molecules with a versatile range of activities. Their structural and physicochemical characteristics make them a privileged scaffold in medicinal chemistry and chemical biology (Carneiro *et al.*, 2021). Historically, coumarins have been applied for the treatment of a variety of diseases due to their anticoagulant, anti-inflammatory, antiviral, antimicrobial, anticancer, antioxidant (Todorov *et al.*, 2023) and anti-glaucoma (Ziki *et al.*, 2023) activities. Their wide range of biological activities and the use of coumarin-containing drugs clinically have contributed to the growing interest in this class of heterocycles (Khandy *et al.*, 2024). Given their importance, coumarin derivatives continue to be our field of research (Kambo *et al.*, 2017; Hollauer *et al.*, 2023). We report herein the synthesis, crystal structure, and Hirshfeld surface analysis of the title coumarin derivative.





2. Structural commentary

The molecular structure of the title coumarin derivative is illustrated in Fig. 1. An *S*(6) ring motif arises from an intra-

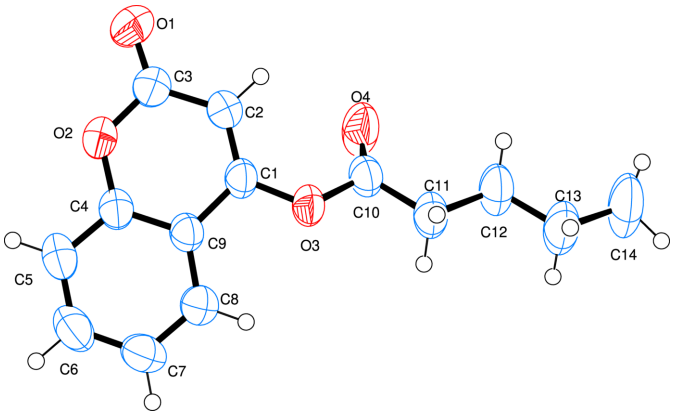


Figure 1
Molecular structure of the compound showing the atomic numbering system. Displacement ellipsoids are drawn at the 50% probability level.

molecular C2—H2···O4 hydrogen bond (Table 1). As expected, the coumarin ring system is almost planar, with a maximum deviation from the plane of 0.016 (3) Å for atom C7. An inspection of the bond lengths shows that there is a slight asymmetry of the electronic distribution around the pyrone ring: the C1—C2 [1.336 (3) Å] and C2—C3 [1.437 (3) Å] bond lengths are shorter and longer, respectively, than those excepted for a C_{ar}—C_{ar} bond. This suggests that the electron density is preferentially located in the C1—C2 bond of the pyrone ring, as seen in other coumarin derivatives (Gomes *et al.*, 2016; Ouédraogo *et al.*, 2018).

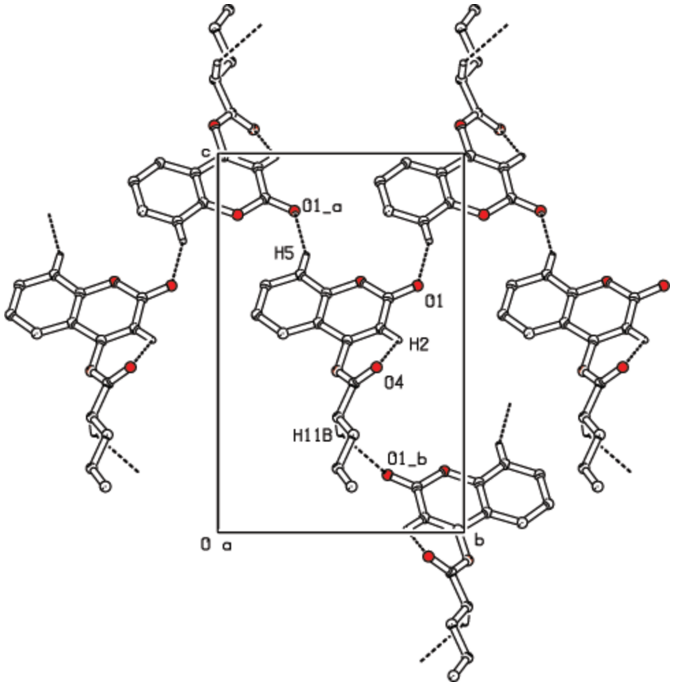


Figure 2
Part of crystalline packing of the title compound showing a parallel chain in the [001] direction. Dashed lines indicate hydrogen bonds. H atoms not involved in hydrogen-bonding interactions have been omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O4	0.93	2.40	2.855 (3)	110
C5—H5···O1^{i}	0.93	2.53	3.387 (3)	153
C11—H11B···O1^{ii}	0.97	2.65	3.446 (3)	139

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

3. Supramolecular features and Hirshfeld surface analysis

In the crystal, C5—H5···O1 hydrogen bonds link molecules into infinite chains along the [001] direction (Table 1, Fig. 2) and the C11—H11B···O1 interactions contribute to the crystal cohesion. The intermolecular interactions were quantified using Hirshfeld surface analysis. This approach is a graphical tool for visualization and understanding of intermolecular interactions. The Hirshfeld surface analysis was performed, and the two-dimensional (2D) fingerprint plots were generated with *CrystalExplorer 17* (Spackman *et al.*, 2021). Fig. 3 shows the Hirshfeld surface plotted over d_{norm} (normalized contact distance) and Fig. 4 the 2D fingerprint plots..

4. Database survey

A search of the Cambridge structural Database (CSD; Groom *et al.*, 2016; updated to April 2024) found seven coumarins structures with substituents at the 4-positions (XUFGOW, Kavitha *et al.*, 2015; NUZJOJ, Vinduvahini *et al.*, 2016; UDOGIF01, Anitha *et al.*, 2016, HUYVEE, Anitha *et al.*, 2015; NAGWAW, Ravi *et al.*, 2016; DIWPAE, Hollauer *et al.*, 2023). All seven have structural parameters very similar to this one, including essentially planar chromene portions.

5. Synthesis and crystallization

To a solution of valeroyl chloride (6.17 mmol, ∼0.8 ml) in dried diethyl ether (16 ml) was added dried pyridine (2.31 ml;

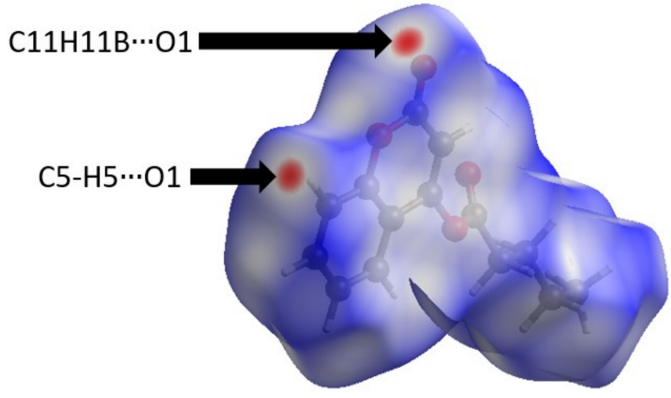


Figure 3
The Hirshfeld surface mapped over d_{norm} for visualizing the intermolecular contacts of the title compound.

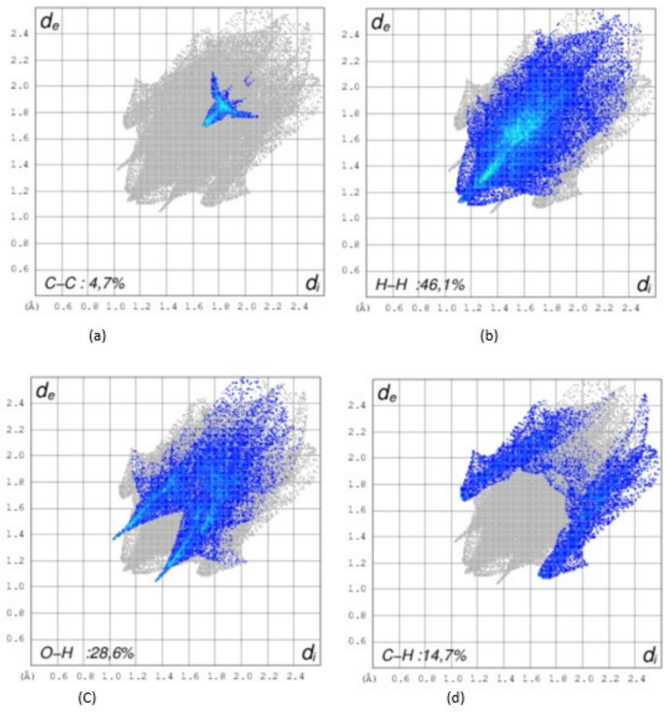


Figure 4
Fingerprint plots for the title compound showing (*a*) C···C, (*b*) H···H, (*c*) O···H/H···O and (*d*) C···H/H···C interactions. The outline of the full fingerprint is shown in grey. d_i is the closest internal distance from a given point on the Hirshfeld surface and d_e is the closest external contact.

4.7 molar equivalents) and 4-hydroxycoumarin (6.17 mmol, 1 g) in small portions over 30 min, with vigorous stirring. The reaction mixture was left stirring at room temperature for 3 h.

The mixture was then poured in a separating funnel containing 40 ml of chloroform and washed with diluted hydrochloric acid solution until the pH was 2–3. The organic layer was extracted, washed with water to neutrality, dried over $MgSO_4$ and the solvent removed. The crude product was filtered off with suction, washed with *n*-hexane and recrystallized from acetone. Dirty white crystals of the title compound were obtained in a good yield (78%), m.p. 408–409 K.

6. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms were located in a difference-Fourier map, but were positioned with idealized geometry and refined isotropically using a riding model (HFIX command), U_{iso}(H) = 1.5U_{eq}(C-methyl) and 1.2U_{eq}(C) for all other H atoms.

Acknowledgements

The authors are grateful to the Spectropôle Service of the Faculty of Sciences and Techniques (Aix-Marseille, France) for the use of the diffractometer.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{14}H_{14}O_4$
M_r	246.25
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	295
a, b, c (Å)	9.57455 (13), 9.29660 (17), 14.5761 (2)
β (°)	100.9517 (14)
V (Å^3)	1273.80 (3)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.78
Crystal size (mm)	0.26 × 0.22 × 0.18
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Atlas 2
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku OD, 2023)
T_{min}, T_{max}	0.869, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13663, 2492, 2107
R_{int}	0.024
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.056, 0.175, 1.08
No. of reflections	2492
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.27

Computer programs: *CrysAlis PRO* (Rigaku OD, 2023), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2013* (Sheldrick, 2015*b*), *PLATON* (Spek, 2020), *ORTEP*-III Burnett & Johnson, 1996), *WinGX* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

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Acta Cryst. (2024). E**80**, 767-770 [https://doi.org/10.1107/S205698902400584X]

Synthesis, structure and Hirshfeld surface analysis of 2-oxo-2*H*-chromen-4-yl pentanoate

Valentin Bationo, Konan René Kambo, Charles Bavouma Sombié, Rasmané Semdé, Pierre Francotte and Abdoulaye Djandé

Computing details

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

Crystal data

$C_{14}H_{14}O_4$
$M_r = 246.25$
Monoclinic, $P2_1/c$
$a = 9.57455$ (13) Å
$b = 9.29660$ (17) Å
$c = 14.5761$ (2) Å
$\beta = 100.9517$ (14)°
$V = 1273.80$ (3) Å^3
$Z = 4$
$F(000) = 520$
$D_x = 1.284$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 5988 reflections
$\theta = 6.2$–72.3°
$\mu = 0.78$ mm^{-1}
$T = 295$ K
Prism, colourless
0.26 × 0.22 × 0.18 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Atlas 2 diffractometer
Radiation source: micro-focus sealed X-ray tube
Mirror monochromator
Detector resolution: 5.3045 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (CrysAlisPro; Rigaku OD, 2023)
$T_{min} = 0.869$, $T_{max} = 1.000$
13663 measured reflections
2492 independent reflections
2107 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 72.4°$, $\theta_{min} = 4.7°$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 9$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.175$
$S = 1.08$
2492 reflections
164 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0899P)^2 + 0.3573P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Experimental. CrysAlisPro 1.171.42.102a (Rigaku Oxford Diffraction, 2023) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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}
O1	0.8531 (2)	0.18982 (18)	0.34863 (13)	0.0878 (6)
O2	0.89842 (16)	0.42028 (16)	0.33853 (10)	0.0657 (4)
O3	0.73548 (14)	0.51487 (15)	0.57413 (9)	0.0611 (4)
O4	0.5562 (2)	0.3555 (2)	0.56376 (12)	0.0961 (7)
C1	0.78028 (18)	0.4741 (2)	0.49394 (12)	0.0521 (4)
C2	0.7829 (2)	0.3396 (2)	0.46205 (14)	0.0601 (5)
H2	0.7447	0.2654	0.4923	0.072*
C3	0.8442 (2)	0.3080 (2)	0.38166 (15)	0.0637 (5)
C4	0.89359 (19)	0.5596 (2)	0.36974 (13)	0.0552 (5)
C5	0.9492 (2)	0.6653 (3)	0.32029 (15)	0.0686 (6)
H5	0.9891	0.6417	0.2689	0.082*
C6	0.9443 (3)	0.8055 (3)	0.34855 (18)	0.0770 (7)
H6	0.9807	0.8776	0.3155	0.092*
C7	0.8862 (3)	0.8417 (2)	0.42546 (19)	0.0769 (6)
H7	0.8837	0.9373	0.4439	0.092*
C8	0.8321 (2)	0.7356 (2)	0.47459 (15)	0.0640 (5)
H8	0.7930	0.7600	0.5263	0.077*
C9	0.83527 (18)	0.5928 (2)	0.44771 (13)	0.0514 (4)
C10	0.6263 (2)	0.4471 (2)	0.60627 (13)	0.0588 (5)
C11	0.6104 (3)	0.5117 (3)	0.69639 (16)	0.0763 (7)
H11A	0.5905	0.6134	0.6862	0.092*
H11B	0.7010	0.5039	0.7392	0.092*
C12	0.4994 (3)	0.4499 (3)	0.74266 (16)	0.0776 (7)
H12A	0.4091	0.4569	0.6995	0.093*
H12B	0.5199	0.3484	0.7531	0.093*
C13	0.4818 (4)	0.5132 (4)	0.8311 (2)	0.1043 (11)
H13A	0.4636	0.6150	0.8203	0.125*
H13B	0.5723	0.5050	0.8739	0.125*
C14	0.3720 (4)	0.4574 (4)	0.8790 (2)	0.1154 (12)
H14A	0.2833	0.4508	0.8355	0.173*
H14B	0.3614	0.5211	0.9291	0.173*
H14C	0.3992	0.3637	0.9038	0.173*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1124 (13)	0.0674 (10)	0.0945 (12)	−0.0079 (9)	0.0470 (10)	−0.0231 (9)

O2	0.0772 (9)	0.0669 (9)	0.0606 (8)	−0.0033 (7)	0.0327 (7)	−0.0066 (6)
O3	0.0666 (8)	0.0655 (9)	0.0593 (8)	−0.0121 (6)	0.0321 (6)	−0.0060 (6)
O4	0.0994 (12)	0.1166 (15)	0.0855 (11)	−0.0483 (11)	0.0512 (10)	−0.0319 (10)
C1	0.0498 (8)	0.0594 (11)	0.0511 (9)	−0.0023 (7)	0.0197 (7)	0.0002 (8)
C2	0.0676 (11)	0.0539 (11)	0.0645 (11)	−0.0058 (9)	0.0274 (9)	0.0005 (9)
C3	0.0685 (11)	0.0610 (12)	0.0661 (12)	−0.0034 (9)	0.0237 (9)	−0.0052 (9)
C4	0.0546 (9)	0.0614 (11)	0.0523 (9)	0.0002 (8)	0.0174 (7)	0.0041 (8)
C5	0.0726 (12)	0.0783 (14)	0.0605 (11)	−0.0048 (10)	0.0269 (10)	0.0116 (10)
C6	0.0829 (15)	0.0721 (15)	0.0812 (15)	−0.0046 (11)	0.0286 (12)	0.0243 (12)
C7	0.0876 (15)	0.0513 (12)	0.0967 (17)	−0.0010 (10)	0.0302 (13)	0.0088 (11)
C8	0.0666 (11)	0.0582 (12)	0.0728 (12)	0.0017 (9)	0.0276 (10)	0.0010 (10)
C9	0.0479 (8)	0.0549 (11)	0.0545 (9)	0.0003 (7)	0.0175 (7)	0.0036 (8)
C10	0.0585 (10)	0.0657 (12)	0.0568 (10)	−0.0064 (9)	0.0224 (8)	0.0035 (9)
C11	0.0903 (15)	0.0834 (16)	0.0652 (12)	−0.0196 (12)	0.0402 (11)	−0.0079 (11)
C12	0.0740 (13)	0.1044 (18)	0.0614 (12)	−0.0191 (13)	0.0304 (10)	−0.0062 (12)
C13	0.131 (2)	0.116 (2)	0.0846 (17)	−0.0419 (19)	0.0664 (17)	−0.0261 (16)
C14	0.117 (2)	0.167 (3)	0.0780 (17)	−0.043 (2)	0.0579 (17)	−0.0214 (19)

Geometric parameters (Å, º)

O1—C3	1.209 (3)	C7—H7	0.9300
O2—C3	1.371 (2)	C8—C9	1.387 (3)
O2—C4	1.377 (2)	C8—H8	0.9300
O3—C1	1.373 (2)	C10—C11	1.478 (3)
O3—C10	1.377 (2)	C11—C12	1.479 (3)
O4—C10	1.184 (3)	C11—H11A	0.9700
C1—C2	1.336 (3)	C11—H11B	0.9700
C1—C9	1.443 (2)	C12—C13	1.455 (3)
C2—C3	1.437 (3)	C12—H12A	0.9700
C2—H2	0.9300	C12—H12B	0.9700
C4—C5	1.384 (3)	C13—C14	1.464 (3)
C4—C9	1.393 (3)	C13—H13A	0.9700
C5—C6	1.370 (3)	C13—H13B	0.9700
C5—H5	0.9300	C14—H14A	0.9600
C6—C7	1.384 (4)	C14—H14B	0.9600
C6—H6	0.9300	C14—H14C	0.9600
C7—C8	1.376 (3)		

C3—O2—C4	121.71 (15)	C4—C9—C1	116.78 (17)
C1—O3—C10	122.95 (15)	O4—C10—O3	122.85 (18)
C2—C1—O3	125.75 (17)	O4—C10—C11	127.85 (18)
C2—C1—C9	121.34 (17)	O3—C10—C11	109.25 (17)
O3—C1—C9	112.80 (16)	C10—C11—C12	116.9 (2)
C1—C2—C3	120.85 (18)	C10—C11—H11A	108.1
C1—C2—H2	119.6	C12—C11—H11A	108.1
C3—C2—H2	119.6	C10—C11—H11B	108.1
O1—C3—O2	116.60 (19)	C12—C11—H11B	108.1
O1—C3—C2	125.5 (2)	H11A—C11—H11B	107.3

O2—C3—C2	117.86 (18)	C13—C12—C11	117.4 (2)
O2—C4—C5	117.11 (17)	C13—C12—H12A	107.9
O2—C4—C9	121.44 (17)	C11—C12—H12A	107.9
C5—C4—C9	121.45 (19)	C13—C12—H12B	107.9
C6—C5—C4	118.7 (2)	C11—C12—H12B	107.9
C6—C5—H5	120.6	H12A—C12—H12B	107.2
C4—C5—H5	120.6	C12—C13—C14	119.6 (3)
C5—C6—C7	121.1 (2)	C12—C13—H13A	107.4
C5—C6—H6	119.5	C14—C13—H13A	107.4
C7—C6—H6	119.5	C12—C13—H13B	107.4
C8—C7—C6	119.8 (2)	C14—C13—H13B	107.4
C8—C7—H7	120.1	H13A—C13—H13B	107.0
C6—C7—H7	120.1	C13—C14—H14A	109.5
C7—C8—C9	120.5 (2)	C13—C14—H14B	109.5
C7—C8—H8	119.7	H14A—C14—H14B	109.5
C9—C8—H8	119.7	C13—C14—H14C	109.5
C8—C9—C4	118.44 (17)	H14A—C14—H14C	109.5
C8—C9—C1	124.78 (17)	H14B—C14—H14C	109.5

C10—O3—C1—C2	−34.4 (3)	C7—C8—C9—C1	−179.4 (2)
C10—O3—C1—C9	149.38 (17)	O2—C4—C9—C8	−179.15 (17)
O3—C1—C2—C3	−174.29 (19)	C5—C4—C9—C8	0.9 (3)
C9—C1—C2—C3	1.6 (3)	O2—C4—C9—C1	0.0 (3)
C4—O2—C3—O1	179.69 (19)	C5—C4—C9—C1	179.97 (17)
C4—O2—C3—C2	−0.8 (3)	C2—C1—C9—C8	177.8 (2)
C1—C2—C3—O1	178.9 (2)	O3—C1—C9—C8	−5.9 (3)
C1—C2—C3—O2	−0.5 (3)	C2—C1—C9—C4	−1.3 (3)
C3—O2—C4—C5	−178.94 (18)	O3—C1—C9—C4	175.07 (16)
C3—O2—C4—C9	1.1 (3)	C1—O3—C10—O4	−5.5 (3)
O2—C4—C5—C6	179.07 (19)	C1—O3—C10—C11	176.86 (19)
C9—C4—C5—C6	−0.9 (3)	O4—C10—C11—C12	3.8 (4)
C4—C5—C6—C7	0.5 (4)	O3—C10—C11—C12	−178.7 (2)
C5—C6—C7—C8	−0.1 (4)	C10—C11—C12—C13	−179.5 (3)
C6—C7—C8—C9	0.0 (4)	C11—C12—C13—C14	179.1 (3)
C7—C8—C9—C4	−0.4 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O4	0.93	2.40	2.855 (3)	110
C5—H5···O1^{i}	0.93	2.53	3.387 (3)	153
C11—H11*B*···O1^{ii}	0.97	2.65	3.446 (3)	139

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