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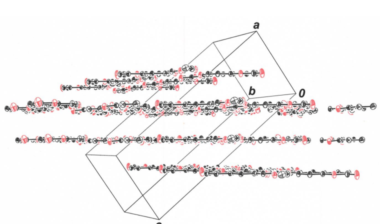

Ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate

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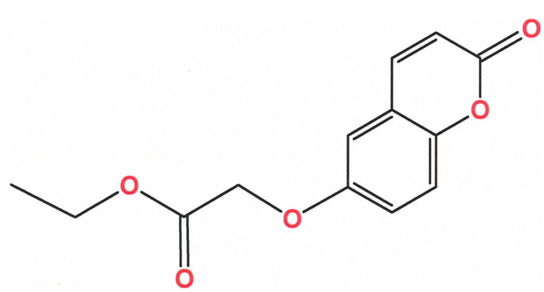
Ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate, $C_{13}H_{12}O_5$, a member of the pharmacologically important class of coumarins, crystallizes in the monoclinic *C*2/*c* space group in the form of sheets, within which molecules are related by inversion centers and 2_1 axes. Multiple C—H⋯O weak hydrogen-bonding interactions reinforce this pattern. The planes of these sheets are oriented in the approximate direction of the *ac* face diagonal. Intersheet interactions are a combination of coumarin system π–π stacking and additional C—H⋯O weak hydrogen bonds between ethyl acetoxy groups.

1. Chemical context

Chromen-2-one, also known as coumarin, and its derivatives hold considerable significance in both natural product and synthetic organic chemistry. Coumarin's structure is characterized by a benzene ring fused to an α-pyrone ring, which makes it valuable in pharmaceutical research (Murray *et al.*, 1982). Coumarin derivatives have shown biological activity as anticancer (Emami & Dadashpour, 2015), antioxidant (Matos *et al.*, 2017), anticoagulant (Satish, 2016) and antineurodegenerative agents (Jameel *et al.*, 2016). We have previously reported a number of synthetically derived molecules based on coumarin, chromene and flavone as substrates/inhibitors of several important cytochrome P450 enzymes, including P450s 1A1, 1A2, and 2A6 (Goyal *et al.*, 2023; Foroozesh *et al.*, 1997). As part of an ongoing program of research into the pharmacological properties of coumarin derivatives, we have undertaken the synthesis of ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate, the structural characterization of which we report herein.



2. Structural commentary

Ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate deposits in the form of colorless blocks by slow cooling of a 2:1 ethyl acetate: hexanes solution. The molecule crystallizes in completely ordered fashion with the appended ethyl oxyacetate group at the 6-position arranged in a fully extended, linear arrange-

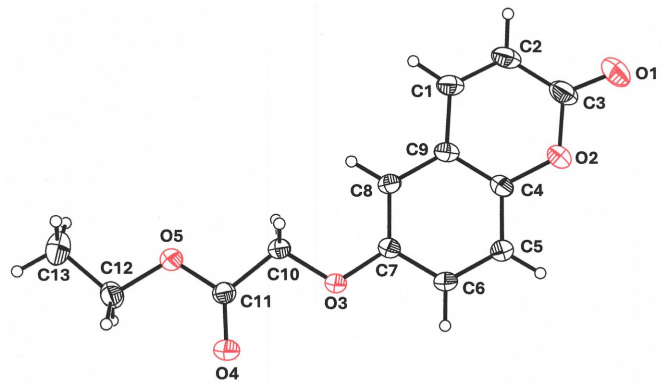


Figure 1
Displacement ellipsoid plot of ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate with complete labeling of non-hydrogen atoms. Ellipsoids are shown at the 50% probability level.

ment (Fig. 1). Thus, all non-hydrogen atoms of the molecule reside within the same plane with an average deviation of 0.0457 Å.

Coplanar pairs of ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate molecules are organized in a head-to-tail fashion by apparent C5—H5···O4 and C6—H6···O3 weak hydrogen-bonding interactions around an inversion center (Table 1, Fig. 2). The adjoining rows of molecules above and below those shown in Fig. 2 are related by 2_1 screw axes to those in these centrosymmetric dyads, with which they form C8—H8···O1 and C1—H1···O1 hydrogen bonds (Fig. 3). The replication of these rows of molecules, which are alternately related by inversion centers and 2_1 axes, creates sheets whose planes lie approximately in the direction of the *ac* face diagonal of the unit cell (Fig. 4). Molecules between sheets are also related by inversion centers (Fig. 5) and enjoy pairs of C12—H12*A*···O3 hydrogen-bond contacts. The layered packing arrangement is guided by π–π stacking between the coumarin ring systems, with a separation of 3.4460 (6) Å between the centroids of the α-pyrone rings (C1–C3/O2/C4/C9) of adjacent molecules, as assessed by *PLATON* (Spek, 2020). This distance is only modestly greater than the 3.35 Å separation between the sheets of carbon atoms in graphite (Chung, 2002) and is reinforced by the hydrogen bonding between extended ethyl oxyacetate chains in adjacent layers (Fig. 5).

A Hirshfeld surface, generated by use of *CrystalExplorer* 21.5 (Spackman *et al.*, 2021) for ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate is presented in Fig. 6 with a normalized

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O4^{i}	0.95	2.47	3.1973 (12)	133
C6—H6···O3^{i}	0.95	2.65	3.5965 (11)	172
C1—H1···O1^{ii}	0.95	2.61	3.3951 (15)	140
C8—H8···O1^{ii}	0.95	2.27	3.1536 (12)	154
C12—H12*A*···O3^{iii}	0.99	2.57	3.5156 (13)	159

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+1, -y+1, -z+1$.

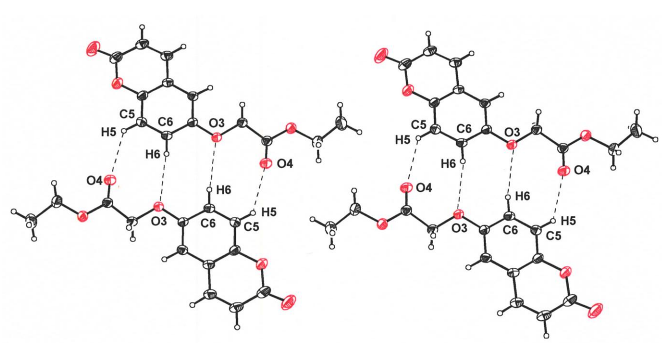


Figure 2
Planar centrosymmetric dyads of ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate showing the C—H···O weak interactions that guide the packing arrangement. The H5···O4 and H6···O3 distances are 2.47 and 2.65 Å, respectively. The symmetry transformation relating molecules through these hydrogen bonds is $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$. Displacement ellipsoids are presented at the 50% probability level.

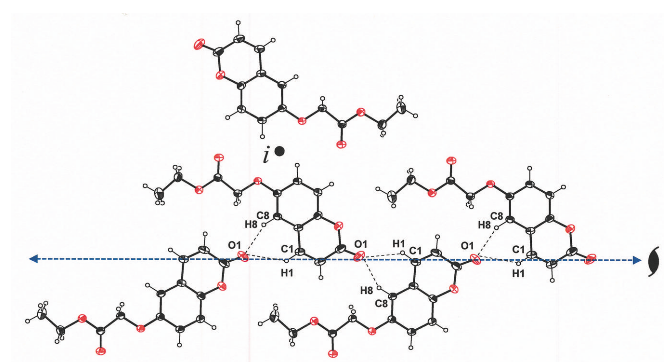


Figure 3
Rows of ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate molecules to both sides of, and in the same plane as, the centrosymmetric diads in Fig. 2. These molecules are related to those in the centrosymmetric dyads by a 2_1 operation, the position for one such axis being shown. This patterned arrangement is assisted by C8—H8···O1 and by C1—H1···O1 close contacts, in which the corresponding H8···O1 and H1···O1 distances are 2.27 and 2.61 Å. The symmetry transformation whereby one molecule is converted to the other across these hydrogen bonds is $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$. Ellipsoids are shown at the 50% probability level.

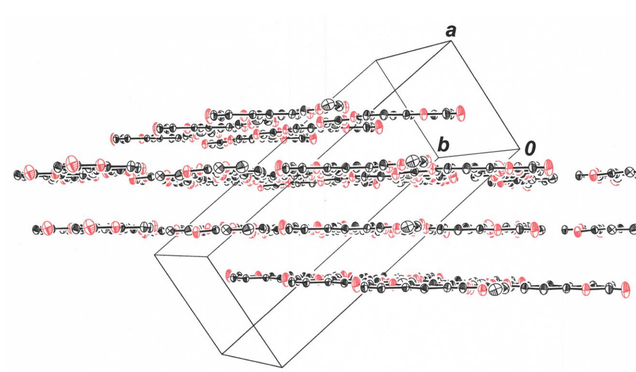


Figure 4
Packing diagram for ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate illustrating the arrangement of molecules into sheets in the approximate direction of the *ac* face diagonal of the unit cell. All H atoms are omitted for clarity, and displacement ellipsoids are drawn at 50% probability.

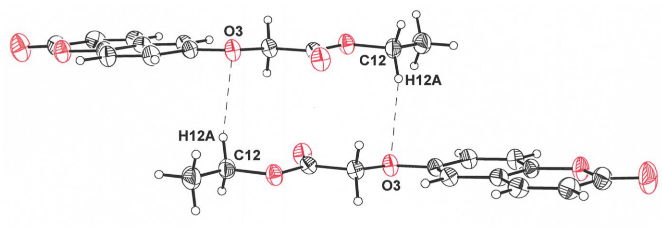


Figure 5
Weak C—H···O interactions between centrosymmetric pairs of molecules of ethyl 2-[(2-oxo-2H-chromen-6-yl)oxy]acetate in different sheets. The H12A···O3 distance is 2.57 Å, and the symmetry transformation relating these molecules is $-x + 1$, $-y + 1$, $-z + 1$. Displacement ellipsoids are presented at the 50% probability level.

contact distance ($d_{\rm norm}$) set between −0.3446 and 1.3365. Adjacent molecules, both within the plane and above the plane of that depicted with the Hirshfeld surface, are shown along with close C—H···O contacts. The C—H···O hydrogen bonds that are separately illustrated in Figs. 2, 3 and 5 are collectively shown in Fig. 6 and emphasize the packing efficiency enabled by the abundance of such juxtapositions. Fig. 7 illustrates a fingerprint plot with all intermolecular contacts presented in the upper left panel and the O···H/H···O, C···H/H···C, and H···H contacts parsed into separate panels (clockwise, respectively). Of these contacts, the O···H/H···O contribute most importantly to the packing energetics, both because they represent the greatest percentage of the total and because they account for the closest intermolecular contacts. The distinctive blue fingers observed in the $d_{\rm e} + d_{\rm i} \simeq$ 2.2–2.6 territory of Fig. 7, upper right, have their origin in these non-classical C—H···O hydrogen bonds.

3. Database survey

A variety of chromen-2-ones that are substituted in the 6-position of the ring system have been characterized

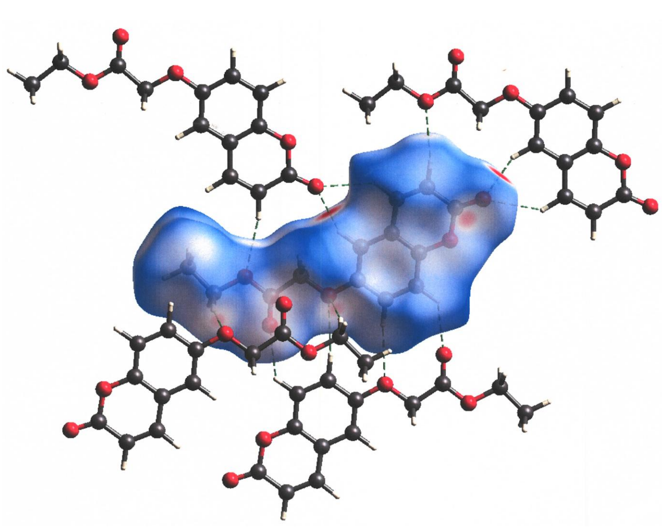


Figure 6
Hirshfeld surface for ethyl 2-[(2-oxo-2H-chromen-6-yl)oxy]acetate with $d_{\rm norm}$ set between −0.3446 and 1.3365. Close intermolecular contacts are depicted with dashed lines.

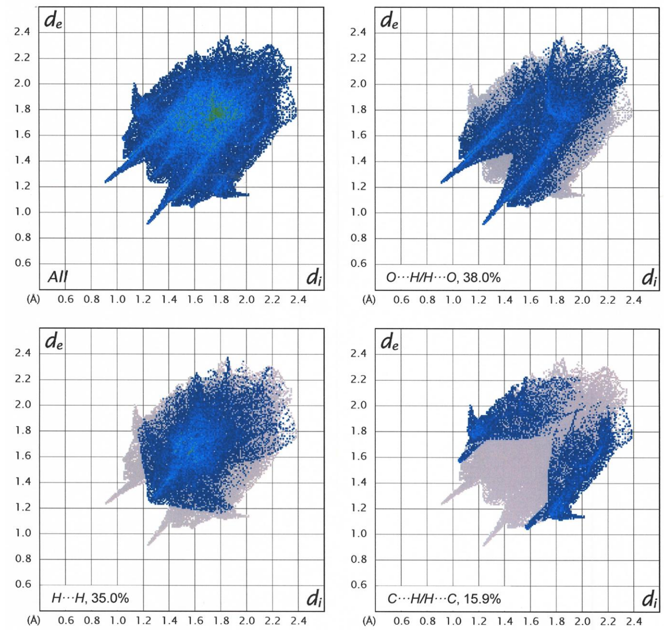


Figure 7
Fingerprint plot for ethyl 2-[(2-oxo-2H-chromen-6-yl)oxy]acetate with all intermolecular contacts presented in the upper left panel and the O···H/H···O, C···H/H···C, and H···H contacts illustrated in separate panels (clockwise, respectively).

structurally by X-ray diffraction. Examples include 6-methoxycoumarin (Baures *et al.*, 2002), 6-benzyloxycoumarin (Adfa *et al.*, 2010), 6-acetoxycoumarin (Murthy *et al.*, 1988), 6-(quinoxalin-2-yl)coumarin (Bandaru *et al.*, 2019), 6-(4-*tert*-butylbenzoate)coumarin (Kenfack Tsobnang *et al.*, 2024), and 6-(2-iodophenoxy)coumarin (Wang *et al.*, 2022). Of these, only 6-methoxycoumarin has a planar molecular structure and therefore a sheetlike packing arrangement in the crystalline state that is analogous to that observed for ethyl 2-[(2-oxo-2H-chromen-6-yl)oxy]acetate. Because aryloxy substituents in the 6-position of the coumarin ring system are typically not oriented to be in the same plane as the coumarin core, a pattern that such derivatives display is packing as centrosymmetric dyads with with parallel coplanar arrangement of the coumarin cores.

4. Synthesis and crystallization

Potassium carbonate (0.512 g, 3.70 mmol) was added to a stirred solution of 6-hydroxy-2H-chromen-2-one (0.200 g, 1.233 mmol) in 10 mL of acetone, and stirring was continued for 30 minutes at 298 K. Bromoethyl acetate (0.309 g, 1.850 mmol) was added slowly to the reaction mixture, and upon completion, the temperature was elevated to 313 K with stirring for 12 h. The reaction mixture was filtered, and the filtrate was concentrated to dryness under reduced pressure. The resulting crude solid was purified *via* flash chromatography on silica gel with 20:80 ethyl acetate:hexanes as the eluting solvent to yield ethyl 2-[(2-oxo-2H-chrome-6-yl)oxy] acetate as a white solid, m.p. 377–380 K. ^{1}H NMR [300 MHz, δ (ppm, in $CDCl_3$)]: 7.53 (d, J = 8.0 Hz, 1 H), 7.21 (d, J = 8.0 Hz, 1 H), 7.14–7.10 (*m*, 1 H), 6.93 (*d*, *J* = 6.9 Hz, 1 H), 6.39 (*d*, *J* = 7.0 Hz, 1 H), 4.64 (*s*, 2 H), 4.28 (*q*, *J* = 7.4 Hz, 2 H), 1.28 (*t*, *J* = 7.2 Hz, 3 H). ^{13}C NMR (75 MHz, δ (ppm, in $CDCl_3$)): 168.4, 160.7, 154.3, 149.0, 143.0, 119.8, 119.2, 117.9, 117.2, 111.6, 65.9, 61.5, 14.1. Diffraction-quality white needle-shaped crystals were obtained by slow cooling of a warm solution of the product in 2:1 ethyl acetate:hexanes.

5. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All hydrogen atoms were refined isotropically with displacement parameters 1.2–1.5 times those of the carbon atoms to which they are attached.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{12}O_5$
M_r	248.23
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	150
a, *b*, *c* (Å)	8.2188 (4), 13.8709 (7), 20.8370 (11)
β (°)	96.062 (2)
V (Å^3)	2362.2 (2)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.22 × 0.11 × 0.07
Data collection	
Diffractometer	Bruker D8 QUEST PHOTON 3 diffractometer
Absorption correction	Numerical (*SADABS*; Krause et al., 2015)
T_{min}, T_{max}	0.95, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	30310, 3031, 2517
R_{int}	0.031
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.677
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.038, 0.115, 1.08
No. of reflections	3031
No. of parameters	164
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.18

Computer programs: *APEX4* and *SAINT* (Bruker, 2021), *SHELXT/5* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *SHELXTL* (Sheldrick, 2008).

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Computing details

Ethyl 2-[(2-oxo-2*H*-chromen-6-yl)oxy]acetate

Crystal data

$C_{13}H_{12}O_5$
M_r = 248.23
Monoclinic, $C2/c$
a = 8.2188 (4) Å
b = 13.8709 (7) Å
c = 20.8370 (11) Å
β = 96.062 (2)°
V = 2362.2 (2) Å^3
Z = 8
F(000) = 1040
D_x = 1.396 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9847 reflections
θ = 2.9–28.7°
μ = 0.11 mm^{-1}
T = 150 K
Block, clear colourless
0.22 × 0.11 × 0.07 mm

Data collection

Bruker D8 QUEST PHOTON 3 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.3910 pixels mm^{-1}
φ and ω scans
Absorption correction: numerical (*SADABS*; Krause et al., 2015)
T_{min} = 0.95, T_{max} = 0.99
30310 measured reflections
3031 independent reflections
2517 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 28.8°, θ_{min} = 2.9°
h = −11→11
k = −18→18
l = −28→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.115
S = 1.08
3031 reflections
164 parameters
0 restraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0593P)^2 + 0.7919P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Experimental. The diffraction data were obtained from 6 sets of frames, each of width 0.50 ° in ω or φ, collected with scan parameters determined by the "strategy" routine in *APEX4*. The scan time was 10.00 sec/frame.

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.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.75633 (12)	1.09654 (7)	0.74589 (4)	0.0550 (3)
O2	0.61257 (9)	1.00599 (5)	0.67405 (3)	0.0340 (2)
O3	0.39172 (8)	0.65701 (5)	0.57181 (3)	0.02737 (17)
O4	0.30429 (9)	0.48767 (5)	0.51199 (3)	0.03335 (19)
O5	0.43669 (8)	0.40506 (5)	0.59491 (3)	0.02889 (18)
C1	0.72524 (12)	0.84028 (8)	0.74031 (5)	0.0296 (2)
H1	0.764445	0.783825	0.762671	0.036*
C2	0.77358 (13)	0.92769 (8)	0.76270 (5)	0.0341 (2)
H2	0.846404	0.932070	0.801150	0.041*
C3	0.71754 (14)	1.01526 (8)	0.72973 (5)	0.0366 (3)
C4	0.56065 (11)	0.91657 (7)	0.65061 (4)	0.0261 (2)
C5	0.45308 (12)	0.91484 (7)	0.59435 (5)	0.0288 (2)
H5	0.417946	0.973141	0.573336	0.035*
C6	0.39835 (11)	0.82698 (7)	0.56963 (4)	0.0268 (2)
H6	0.324788	0.824656	0.531298	0.032*
C7	0.45074 (11)	0.74109 (7)	0.60079 (4)	0.0238 (2)
C8	0.55704 (11)	0.74335 (7)	0.65691 (4)	0.0246 (2)
H8	0.591389	0.685046	0.678111	0.030*
C9	0.61368 (11)	0.83234 (7)	0.68224 (4)	0.0246 (2)
C10	0.45751 (12)	0.57113 (7)	0.60081 (4)	0.0258 (2)
H10A	0.430090	0.566985	0.645869	0.031*
H10B	0.578154	0.571536	0.601579	0.031*
C11	0.38763 (11)	0.48537 (7)	0.56293 (4)	0.0249 (2)
C12	0.38869 (13)	0.31449 (7)	0.56288 (5)	0.0316 (2)
H12A	0.426499	0.312769	0.519332	0.038*
H12B	0.268095	0.307832	0.558270	0.038*
C13	0.46617 (15)	0.23374 (8)	0.60369 (6)	0.0411 (3)
H13A	0.434714	0.171748	0.583483	0.062*
H13B	0.428707	0.236471	0.646769	0.062*
H13C	0.585489	0.240449	0.607364	0.062*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0740 (6)	0.0406 (5)	0.0461 (5)	−0.0193 (4)	−0.0137 (4)	−0.0108 (4)
O2	0.0400 (4)	0.0273 (4)	0.0323 (4)	−0.0061 (3)	−0.0069 (3)	−0.0044 (3)
O3	0.0308 (4)	0.0226 (3)	0.0261 (3)	−0.0025 (2)	−0.0093 (3)	0.0009 (2)
O4	0.0369 (4)	0.0320 (4)	0.0283 (4)	−0.0025 (3)	−0.0101 (3)	0.0005 (3)
O5	0.0350 (4)	0.0239 (3)	0.0263 (3)	−0.0031 (3)	−0.0039 (3)	0.0020 (3)
C1	0.0275 (5)	0.0371 (5)	0.0229 (4)	−0.0028 (4)	−0.0034 (4)	0.0001 (4)
C2	0.0325 (5)	0.0440 (6)	0.0242 (4)	−0.0084 (4)	−0.0044 (4)	−0.0043 (4)
C3	0.0395 (6)	0.0400 (6)	0.0291 (5)	−0.0112 (4)	−0.0029 (4)	−0.0079 (4)
C4	0.0261 (4)	0.0259 (5)	0.0254 (4)	−0.0044 (3)	−0.0014 (3)	−0.0038 (3)
C5	0.0306 (5)	0.0256 (5)	0.0285 (5)	0.0000 (4)	−0.0059 (4)	0.0020 (4)
C6	0.0263 (4)	0.0277 (5)	0.0245 (4)	−0.0011 (3)	−0.0071 (3)	0.0017 (3)
C7	0.0226 (4)	0.0244 (4)	0.0235 (4)	−0.0025 (3)	−0.0024 (3)	−0.0005 (3)
C8	0.0244 (4)	0.0265 (4)	0.0218 (4)	−0.0006 (3)	−0.0029 (3)	0.0027 (3)
C9	0.0220 (4)	0.0309 (5)	0.0203 (4)	−0.0022 (3)	−0.0015 (3)	−0.0009 (3)
C10	0.0288 (5)	0.0245 (4)	0.0226 (4)	−0.0024 (3)	−0.0043 (3)	0.0025 (3)
C11	0.0251 (4)	0.0257 (4)	0.0233 (4)	−0.0024 (3)	−0.0001 (3)	0.0022 (3)
C12	0.0384 (5)	0.0247 (5)	0.0317 (5)	−0.0034 (4)	0.0036 (4)	−0.0026 (4)
C13	0.0418 (6)	0.0289 (5)	0.0534 (7)	0.0043 (4)	0.0082 (5)	0.0041 (5)

Geometric parameters (Å, º)

O1—C3	1.2096 (13)	C5—H5	0.9500
O2—C3	1.3769 (12)	C6—C7	1.4026 (13)
O2—C4	1.3838 (11)	C6—H6	0.9500
O3—C7	1.3778 (11)	C7—C8	1.3842 (12)
O3—C10	1.4169 (11)	C8—C9	1.4025 (13)
O4—C11	1.2013 (11)	C8—H8	0.9500
O5—C11	1.3383 (11)	C10—C11	1.5074 (12)
O5—C12	1.4573 (11)	C10—H10A	0.9900
C1—C2	1.3440 (14)	C10—H10B	0.9900
C1—C9	1.4432 (12)	C12—C13	1.5062 (15)
C1—H1	0.9500	C12—H12A	0.9900
C2—C3	1.4467 (16)	C12—H12B	0.9900
C2—H2	0.9500	C13—H13A	0.9800
C4—C9	1.3888 (13)	C13—H13B	0.9800
C4—C5	1.3920 (12)	C13—H13C	0.9800
C5—C6	1.3798 (13)		
C3—O2—C4	121.57 (8)	C9—C8—H8	120.2
C7—O3—C10	115.08 (7)	C4—C9—C8	119.14 (8)
C11—O5—C12	115.90 (7)	C4—C9—C1	118.25 (9)
C2—C1—C9	119.89 (9)	C8—C9—C1	122.61 (9)
C2—C1—H1	120.1	O3—C10—C11	109.44 (7)
C9—C1—H1	120.1	O3—C10—H10A	109.8
C1—C2—C3	121.67 (8)	C11—C10—H10A	109.8

C1—C2—H2	119.2	O3—C10—H10B	109.8
C3—C2—H2	119.2	C11—C10—H10B	109.8
O1—C3—O2	116.42 (10)	H10A—C10—H10B	108.2
O1—C3—C2	126.09 (10)	O4—C11—O5	125.15 (9)
O2—C3—C2	117.48 (9)	O4—C11—C10	126.30 (9)
O2—C4—C9	121.13 (8)	O5—C11—C10	108.54 (7)
O2—C4—C5	117.22 (9)	O5—C12—C13	107.74 (8)
C9—C4—C5	121.64 (9)	O5—C12—H12A	110.2
C6—C5—C4	118.87 (9)	C13—C12—H12A	110.2
C6—C5—H5	120.6	O5—C12—H12B	110.2
C4—C5—H5	120.6	C13—C12—H12B	110.2
C5—C6—C7	120.35 (8)	H12A—C12—H12B	108.5
C5—C6—H6	119.8	C12—C13—H13A	109.5
C7—C6—H6	119.8	C12—C13—H13B	109.5
O3—C7—C8	123.44 (8)	H13A—C13—H13B	109.5
O3—C7—C6	116.07 (7)	C12—C13—H13C	109.5
C8—C7—C6	120.49 (8)	H13A—C13—H13C	109.5
C7—C8—C9	119.51 (9)	H13B—C13—H13C	109.5
C7—C8—H8	120.2		

C9—C1—C2—C3	−0.47 (16)	C6—C7—C8—C9	−0.63 (14)
C4—O2—C3—O1	179.55 (10)	O2—C4—C9—C8	179.62 (8)
C4—O2—C3—C2	0.72 (15)	C5—C4—C9—C8	−0.16 (15)
C1—C2—C3—O1	−178.93 (12)	O2—C4—C9—C1	−0.21 (14)
C1—C2—C3—O2	−0.23 (16)	C5—C4—C9—C1	180.00 (8)
C3—O2—C4—C9	−0.50 (15)	C7—C8—C9—C4	0.50 (14)
C3—O2—C4—C5	179.29 (9)	C7—C8—C9—C1	−179.67 (8)
O2—C4—C5—C6	−179.84 (9)	C2—C1—C9—C4	0.69 (14)
C9—C4—C5—C6	−0.05 (15)	C2—C1—C9—C8	−179.14 (9)
C4—C5—C6—C7	−0.08 (15)	C7—O3—C10—C11	−177.82 (7)
C10—O3—C7—C8	−4.68 (13)	C12—O5—C11—O4	2.60 (14)
C10—O3—C7—C6	174.59 (8)	C12—O5—C11—C10	−176.17 (7)
C5—C6—C7—O3	−178.87 (8)	O3—C10—C11—O4	8.06 (14)
C5—C6—C7—C8	0.43 (15)	O3—C10—C11—O5	−173.20 (7)
O3—C7—C8—C9	178.61 (8)	C11—O5—C12—C13	175.57 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C5—H5···O4^{i}	0.95	2.47	3.1973 (12)	133
C6—H6···O3^{i}	0.95	2.65	3.5965 (11)	172
C1—H1···O1^{ii}	0.95	2.61	3.3951 (15)	140
C8—H8···O1^{ii}	0.95	2.27	3.1536 (12)	154
C12—H12*A*···O3^{iii}	0.99	2.57	3.5156 (13)	159

Symmetry codes: (i) −*x*+1/2, −*y*+3/2, −*z*+1; (ii) −*x*+3/2, *y*−1/2, −*z*+3/2; (iii) −*x*+1, −*y*+1, −*z*+1.