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Ethyl 4-(2-furyl)-2-oxochroman-3-carboxylate

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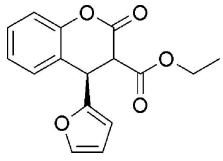
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.042; wR factor = 0.111; data-to-parameter ratio = 11.9.

The title compound, $C_{16}H_{14}O_5$, was prepared from the reaction of 3-carbethoxycoumarin with furan in the presence of $AlCl_3$ as catalyst. In the crystal, intermolecular C—H···O hydrogen-bonding interactions between four molecules lead to a tetramer in the unit cell. The furan ring is antiperiplanar [C—C—C—O = 167.9 (13)°] and the ethoxycarbonyl group is (−)anticlinal [C—C—C—O = −128.6 (14)°] to the lactone ring.

Related literature

For the medicinal and biological activity of coumarins and their derivatives, see: Borges *et al.* (2005); Kontogiorgis & Hadjipavlou-Litina (2005); Gursoy & Karali (2003); Prabhakar *et al.* (2010). For the assignment of conformations and the orientation of the substituents, see: Nardelli (1983, 1995); Klyne & Prelog (1960).



Experimental

Crystal data

$C_{16}H_{14}O_5$
M_r = 286.27
Monoclinic, $P2_1/c$
a = 10.393 (3) Å
b = 8.459 (3) Å
c = 15.819 (5) Å
β = 95.464 (5)°
V = 1384.5 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 K
0.34 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.966, T_{max} = 0.980
13767 measured reflections
2711 independent reflections
2099 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 1.04
2711 reflections
227 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O3^{i}	0.972 (15)	2.696 (15)	3.576 (2)	150.8 (11)
C16—H16B···O2^{ii}	0.96	2.70	3.549 (3)	148
C16—H16A···O2^{iii}	0.96	2.96	3.841 (3)	153
C8—H8···O3^{iv}	0.96 (2)	2.94 (2)	3.611 (3)	128.2 (15)
C11—H11···O4^{v}	0.93 (2)	2.73 (2)	3.501 (3)	140.9 (17)
C13—H13···O2^{vi}	1.01 (2)	2.54 (2)	3.456 (3)	151.0 (17)
C12—H12···O4^{vii}	0.95 (2)	2.74 (2)	3.478 (3)	134.9 (16)

Symmetry codes: (i) $-x+1, -y+2, -z+2$; (ii) $x, y-1, z$; (iii) $-x+2, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x+2, -y+2, -z+2$; (v) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (vi) $x, -y+\frac{5}{2}, z+\frac{1}{2}$; (vii) $x, y+1, z$.

Data collection: *SMART* (Bruker, 2003); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We thank the Director, Institute of Life Sciences, for support and also the Dean, School of Chemistry, University of Hyderabad, for the X-ray crystallographic data.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2028).

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supporting information

Acta Cryst. (2010). E**66**, o1310 [https://doi.org/10.1107/S1600536810016193]

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S1. Comment

We have synthesized and reported our serendipitous observations on the Diels-Alder reaction of 3-carbethoxy coumarin with furan, followed by a ring opening to yield Michael product, 3-carbethoxy-4-(2-furyl)-chroman-2-one in good yields.

S2. Experimental

3-Carbethoxy coumarin (3 m mol) was taken into 30 mmol of furan and 10 mol% of $AlCl_3$ catalyst was added. The reaction mixture was stirred at room temperature for 24 hours. After completion of the reaction, the excess of the furan was distilled off and extracted thrice with water/dichloromethane. The product was separated from flash column chromatography and recrystallized from dichloromethane.

S3. Refinement

All H atoms were found on difference maps, with C—H=0.93 Å and included in the final cycles of refinement using a riding model, with Uiso(H)=1.2Ueq(C)

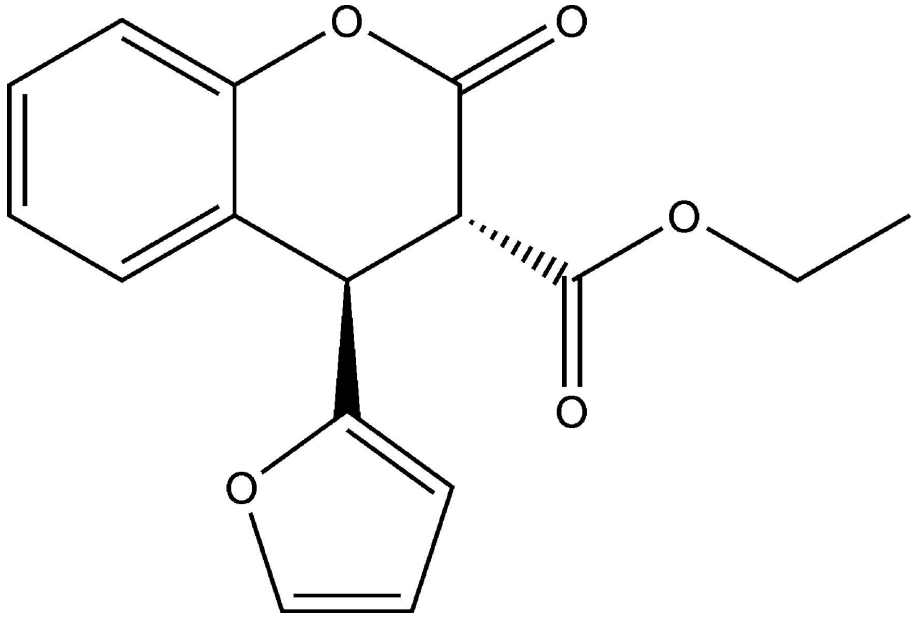

Figure 1

Chemical diagram of the title compound.

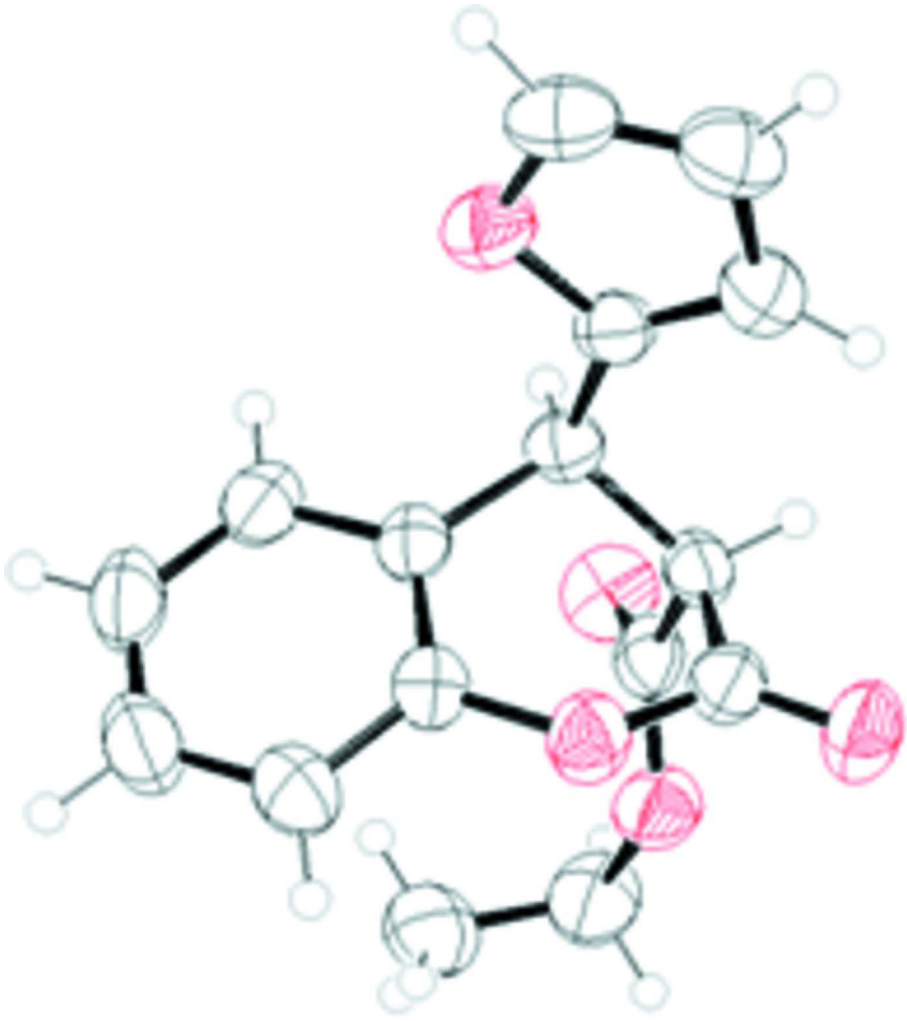

Figure 2

ORTEP diagram of the 3-carbethoxy-4-(2-furyl)-chroman-2-one. (Thermal ellipsoids are at 50% probability level).

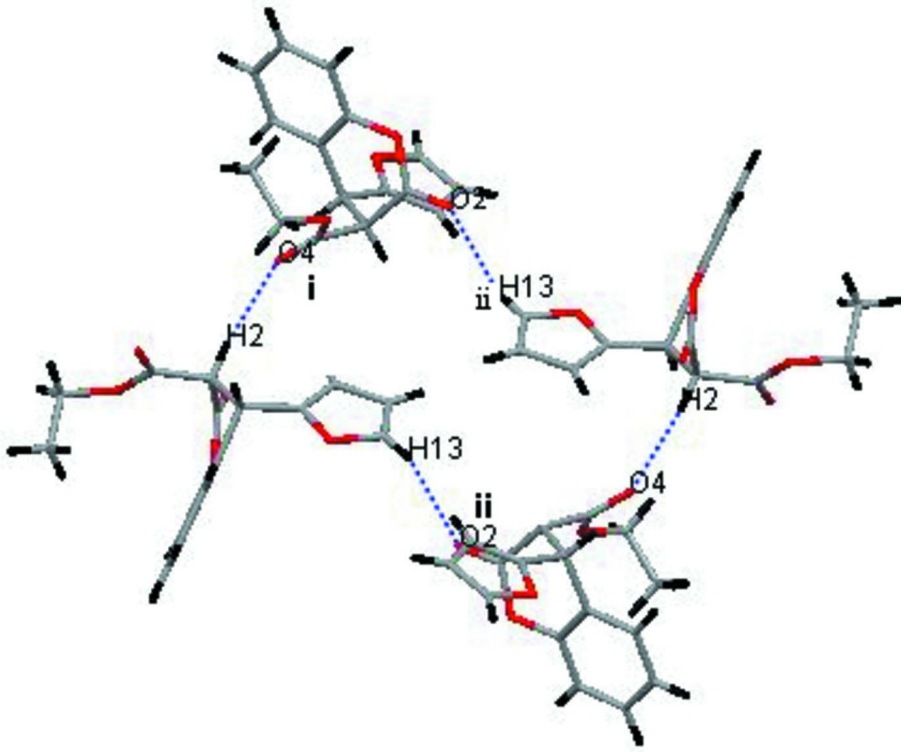

Figure 3

Crystal packing of (I) showing the formation of tetramer. The C—H•••O contacts are shown as dashed lines. Symmetry code: (i) 1-x,-1/2+y, 1/2-z (ii) x, -1/2-y, -1/2+z

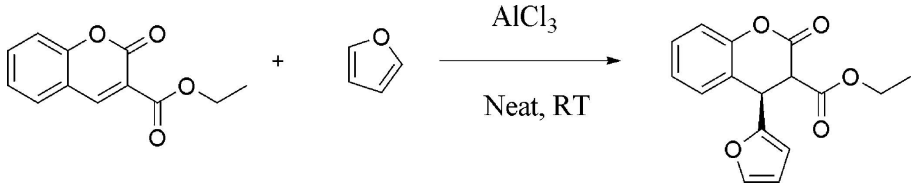

Figure 4

The formation of the title compound.

Ethyl 4-(2-furyl)-2-oxochroman-3-carboxylate

Crystal data

$C_{16}H_{14}O_5$
$M_r = 286.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.393\ (3)$ Å
$b = 8.459\ (3)$ Å
$c = 15.819\ (5)$ Å
$\beta = 95.464\ (5)°$
$V = 1384.5\ (8)$ Å^3
$Z = 4$

$F(000) = 600$
$D_x = 1.373$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5174 reflections
$\theta = 2.5–25.8°$
$\mu = 0.10$ mm^{-1}
$T = 298$ K
Block, colourless
0.34 × 0.24 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2003)
T_{min} = 0.966, T_{max} = 0.980
13767 measured reflections
2711 independent reflections
2099 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 25.9°, θ_{min} = 2.0°
h = −12→12
k = −10→10
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.111
S = 1.04
2711 reflections
227 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.1619P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
H2	0.5893 (16)	0.9788 (19)	0.7769 (10)	0.045 (4)*
H13	0.648 (2)	1.325 (3)	1.0884 (14)	0.084 (7)*
H11	0.598 (2)	1.251 (2)	0.8313 (14)	0.076 (6)*
H12	0.598 (2)	1.472 (3)	0.9435 (13)	0.081 (6)*
H7	1.082 (2)	0.672 (3)	1.0318 (14)	0.085 (7)*
H6	0.8933 (19)	0.630 (2)	1.0962 (13)	0.073 (6)*
H8	1.077 (2)	0.828 (2)	0.9093 (13)	0.078 (6)*
H5	0.6973 (18)	0.7462 (19)	1.0400 (11)	0.048 (5)*
H3	0.5753 (15)	0.8783 (17)	0.9186 (9)	0.033 (4)*
C6	0.8921 (2)	0.6956 (2)	1.04771 (12)	0.0626 (5)
O3	0.65502 (12)	1.13244 (14)	1.01627 (7)	0.0545 (3)
O1	0.88812 (11)	0.98037 (14)	0.83531 (7)	0.0523 (3)
O5	0.74734 (13)	0.71312 (14)	0.73664 (7)	0.0598 (4)
O2	0.78518 (13)	1.08381 (15)	0.72090 (7)	0.0635 (4)
C3	0.65067 (15)	0.93666 (18)	0.90397 (9)	0.0404 (4)
O4	0.57089 (13)	0.66307 (15)	0.80304 (8)	0.0651 (4)
C4	0.77275 (15)	0.86004 (17)	0.94453 (9)	0.0388 (4)
C1	0.77894 (17)	1.00313 (19)	0.78254 (10)	0.0460 (4)
C9	0.88546 (15)	0.88312 (18)	0.90688 (10)	0.0429 (4)
C2	0.65772 (16)	0.92533 (19)	0.80739 (10)	0.0425 (4)
C14	0.65204 (17)	0.7516 (2)	0.78227 (10)	0.0465 (4)
C13	0.63873 (19)	1.2923 (2)	1.02691 (14)	0.0597 (5)
C8	1.00056 (18)	0.8154 (2)	0.93811 (12)	0.0576 (5)
C5	0.77720 (19)	0.7640 (2)	1.01532 (10)	0.0502 (4)

C10	0.63742 (14)	1.10518 (19)	0.93083 (9)	0.0419 (4)
C11	0.61161 (18)	1.2410 (2)	0.89003 (12)	0.0550 (5)
C12	0.61250 (19)	1.3618 (2)	0.95281 (13)	0.0593 (5)
C15	0.7673 (2)	0.5456 (2)	0.71929 (12)	0.0718 (6)
H15A	0.8029	0.5339	0.6652	0.086*
H15B	0.6853	0.4902	0.7162	0.086*
C7	1.0035 (2)	0.7225 (2)	1.00924 (13)	0.0656 (5)
C16	0.8571 (2)	0.4771 (2)	0.78765 (14)	0.0738 (6)
H16A	0.9356	0.5376	0.7935	0.111*
H16B	0.8763	0.3698	0.7737	0.111*
H16C	0.8177	0.4795	0.8401	0.111*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C6	0.0812 (15)	0.0569 (11)	0.0475 (10)	0.0109 (10)	−0.0047 (10)	0.0124 (9)
O3	0.0658 (8)	0.0534 (7)	0.0449 (7)	0.0049 (6)	0.0093 (5)	−0.0084 (5)
O1	0.0463 (7)	0.0581 (7)	0.0526 (7)	−0.0034 (5)	0.0059 (5)	0.0137 (6)
O5	0.0819 (9)	0.0466 (7)	0.0524 (7)	0.0094 (6)	0.0151 (7)	−0.0060 (5)
O2	0.0824 (9)	0.0614 (8)	0.0474 (7)	0.0061 (7)	0.0110 (6)	0.0171 (6)
C3	0.0377 (8)	0.0423 (9)	0.0416 (8)	−0.0022 (7)	0.0052 (7)	−0.0030 (7)
O4	0.0666 (8)	0.0592 (8)	0.0684 (9)	−0.0165 (7)	0.0007 (7)	−0.0116 (6)
C4	0.0459 (9)	0.0342 (8)	0.0357 (8)	−0.0005 (6)	0.0000 (6)	−0.0043 (6)
C1	0.0571 (10)	0.0409 (9)	0.0403 (9)	0.0072 (7)	0.0058 (8)	0.0004 (7)
C9	0.0453 (9)	0.0417 (8)	0.0410 (8)	−0.0012 (7)	0.0005 (7)	0.0016 (7)
C2	0.0446 (9)	0.0423 (9)	0.0392 (8)	0.0078 (7)	−0.0041 (7)	−0.0021 (7)
C14	0.0539 (10)	0.0482 (9)	0.0352 (8)	0.0012 (8)	−0.0077 (7)	−0.0034 (7)
C13	0.0629 (12)	0.0556 (11)	0.0622 (12)	−0.0001 (9)	0.0144 (10)	−0.0197 (10)
C8	0.0456 (10)	0.0652 (12)	0.0609 (11)	0.0047 (8)	−0.0008 (9)	0.0030 (9)
C5	0.0623 (11)	0.0471 (10)	0.0420 (9)	−0.0002 (8)	0.0081 (8)	0.0010 (8)
C10	0.0384 (8)	0.0479 (9)	0.0394 (8)	0.0024 (7)	0.0044 (6)	−0.0057 (7)
C11	0.0628 (12)	0.0512 (10)	0.0508 (11)	0.0106 (8)	0.0039 (9)	−0.0008 (9)
C12	0.0629 (12)	0.0441 (10)	0.0723 (13)	0.0059 (9)	0.0138 (10)	−0.0066 (9)
C15	0.1093 (17)	0.0474 (11)	0.0597 (12)	0.0141 (11)	0.0133 (12)	−0.0108 (9)
C7	0.0622 (13)	0.0673 (13)	0.0641 (12)	0.0171 (10)	−0.0104 (10)	0.0071 (10)
C16	0.0813 (15)	0.0552 (11)	0.0863 (15)	0.0111 (10)	0.0158 (12)	0.0064 (11)

Geometric parameters (Å, º)

C6—C7	1.377 (3)	C2—C14	1.522 (2)
C6—C5	1.381 (3)	C2—H2	0.937 (17)
C6—H6	0.94 (2)	C13—C12	1.317 (3)
O3—C10	1.3663 (19)	C13—H13	1.01 (2)
O3—C13	1.375 (2)	C8—C7	1.371 (3)
O1—C1	1.357 (2)	C8—H8	0.96 (2)
O1—C9	1.4020 (19)	C5—H5	0.962 (18)
O5—C14	1.321 (2)	C10—C11	1.333 (2)
O5—C15	1.462 (2)	C11—C12	1.425 (3)

O2—C1	1.1969 (19)	C11—H11	0.93 (2)
C3—C10	1.498 (2)	C12—H12	0.95 (2)
C3—C4	1.512 (2)	C15—C16	1.478 (3)
C3—C2	1.539 (2)	C15—H15A	0.9700
C3—H3	0.972 (15)	C15—H15B	0.9700
O4—C14	1.197 (2)	C7—H7	0.95 (2)
C4—C9	1.378 (2)	C16—H16A	0.9600
C4—C5	1.381 (2)	C16—H16B	0.9600
C1—C2	1.506 (2)	C16—H16C	0.9600
C9—C8	1.375 (2)		

C7—C6—C5	120.05 (18)	O3—C13—H13	113.0 (13)
C7—C6—H6	120.5 (13)	C7—C8—C9	118.97 (19)
C5—C6—H6	119.4 (13)	C7—C8—H8	119.9 (13)
C10—O3—C13	106.29 (14)	C9—C8—H8	121.0 (13)
C1—O1—C9	120.03 (12)	C6—C5—C4	120.55 (18)
C14—O5—C15	117.97 (15)	C6—C5—H5	122.2 (10)
C10—C3—C4	112.56 (13)	C4—C5—H5	117.2 (10)
C10—C3—C2	110.80 (13)	C11—C10—O3	109.59 (14)
C4—C3—C2	106.09 (12)	C11—C10—C3	134.68 (15)
C10—C3—H3	108.5 (8)	O3—C10—C3	115.73 (13)
C4—C3—H3	110.1 (8)	C10—C11—C12	107.06 (17)
C2—C3—H3	108.7 (8)	C10—C11—H11	124.6 (13)
C9—C4—C5	118.03 (15)	C12—C11—H11	128.3 (13)
C9—C4—C3	117.99 (13)	C13—C12—C11	106.68 (17)
C5—C4—C3	123.94 (15)	C13—C12—H12	126.2 (13)
O2—C1—O1	118.36 (16)	C11—C12—H12	127.1 (13)
O2—C1—C2	124.98 (16)	O5—C15—C16	109.51 (16)
O1—C1—C2	116.65 (13)	O5—C15—H15A	109.8
C8—C9—C4	122.14 (15)	C16—C15—H15A	109.8
C8—C9—O1	116.82 (15)	O5—C15—H15B	109.8
C4—C9—O1	121.02 (13)	C16—C15—H15B	109.8
C1—C2—C14	111.48 (13)	H15A—C15—H15B	108.2
C1—C2—C3	110.60 (13)	C8—C7—C6	120.24 (18)
C14—C2—C3	108.36 (13)	C8—C7—H7	120.9 (14)
C1—C2—H2	105.5 (10)	C6—C7—H7	118.8 (14)
C14—C2—H2	108.9 (10)	C15—C16—H16A	109.5
C3—C2—H2	112.1 (10)	C15—C16—H16B	109.5
O4—C14—O5	125.46 (16)	H16A—C16—H16B	109.5
O4—C14—C2	122.91 (16)	C15—C16—H16C	109.5
O5—C14—C2	111.62 (15)	H16A—C16—H16C	109.5
C12—C13—O3	110.37 (17)	H16B—C16—H16C	109.5
C12—C13—H13	136.6 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3^{i}	0.972 (15)	2.696 (15)	3.576 (2)	150.8 (11)

C16—H16*B*···O2^{ii}	0.96	2.70	3.549 (3)	148
C16—H16*A*···O2^{iii}	0.96	2.96	3.841 (3)	153
C8—H8···O3^{iv}	0.96 (2)	2.94 (2)	3.611 (3)	128.2 (15)
C11—H11···O4^{v}	0.93 (2)	2.73 (2)	3.501 (3)	140.9 (17)
C13—H13···O2^{vi}	1.01 (2)	2.54 (2)	3.456 (3)	151.0 (17)
C12—H12···O4^{vii}	0.95 (2)	2.74 (2)	3.478 (3)	134.9 (16)

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+2; (ii) *x*, *y*−1, *z*; (iii) −*x*+2, *y*−1/2, −*z*+3/2; (iv) −*x*+2, −*y*+2, −*z*+2; (v) −*x*+1, *y*+1/2, −*z*+3/2; (vi) *x*, −*y*+5/2, *z*+1/2; (vii) *x*, *y*+1, *z*.