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Methyl *N*-{4-[(4-methoxyphenoxy)methyl]-2-oxo-2*H*-chromen-7-yl}carbamate

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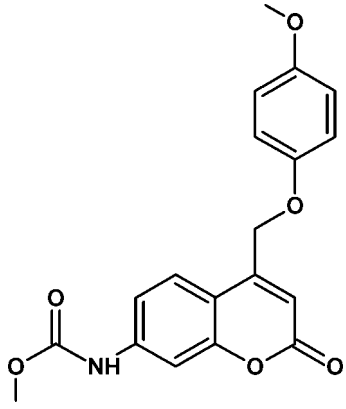
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.035; wR factor = 0.103; data-to-parameter ratio = 12.3.

In the title compound, $C_{19}H_{17}NO_6$, the dihedral angle between the 2*H*-chromene ring system and benzene ring is 5.34 (6)°. A short intramolecular C—H···O contact occurs. In the crystal, molecules are linked by N—H···O hydrogen bonds, generating $C(8)$ chains propagating in [010]. The chains are linked by C—H···O interactions and the packing also exhibits π–π stacking interactions between benzene and pyran rings, with a centroid–centroid distance of 3.676 (9) Å.

Related literature

For a related structure and background to coumarins, see: Mahabaleshwaraiah *et al.* (2012). For further synthetic details, see: Kulkarni & Patil (1981).



Experimental

Crystal data

$C_{19}H_{17}NO_6$
M_r = 355.34
Monoclinic, $P2_1/n$
a = 8.3141 (1) Å
b = 17.3978 (3) Å
c = 11.5729 (2) Å
β = 94.309 (1)°
V = 1669.25 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.770, T_{max} = 1.000
14973 measured reflections
2939 independent reflections
2374 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.103
S = 1.06
2939 reflections
238 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N7—H7···O2^{i}	0.86	1.99	2.8428 (16)	170
C15—H15···O5	0.93	2.30	2.8764 (19)	120
C19—H19A···O5^{ii}	0.97	2.53	3.476 (2)	165

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6754).

References

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supplementary materials

Acta Cryst. (2012). E**68**, o1734 [doi:10.1107/S160053681202048X]

Methyl *N*-{4-[(4-methoxyphenoxy)methyl]-2-oxo-2*H*-chromen-7-yl}carbamate

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Comment

As part of our ongoing studies of coumarin derivatives with possible biological activity (Mahabaleshwaraiah *et al.*, 2012), we now describe the structure of the title compound (Fig. 1).

The 2*H*-chromene (O1/C10–C18) and benzene (C20–C25) rings are almost coplanar; the dihedral angle between them is 5.34 (6)°. In the crystal, (Fig. 2), the molecules are connected by C19—H19A···O5 and N7—H7···O2 interaction hydrogen bonds.(Table 1) Furthermore, the crystal structure features π-π stacking interactions between pyran*Cg2* and benzene*Cg3* rings, with a centroid *Cg2* (O3/C12–C16) -centroid *Cg3* (C13/C14/C17–C20) distance of 3.676 (9) Å.

Experimental

The 4-bromomethyl coumarin required for the target molecule was synthesized according to an already reported (Kulkarni *et al.*1981) procedure involving Pechmann cyclization of phenols with 4-bromoethylacetoacetate. A mixture of 1.56 g (0.005 mol) of 7-carbonylamino-4-bromomethyl coumarin, 0.620 g (0.005 mol) of *p*-methoxy phenol and 0.70 g (0.005 mol) of powdered anhydrous K_2CO_3 in 30 ml of dry acetone were stirred at room temperature for 24 h. After completion of the reaction, the s eparated solid was filtered, washed with excess of dilute (10%) hydrochloric acid (50 ml) and then with an excess of cold water, dried and crystallized twice from ethanol & 1,4-dioxane mixture to yield colourless plates. Yield= 78%, *M*. P.475 K.

Refinement

All H atoms were positioned at calculated positions N—H = 0.86 Å, C—H = 0.93 Å for aromatic H, C—H = 0.97 Å for methelene H and C—H = 0.96 Å for methyl H and refined using a riding model with U_{iso}(H) = 1.5U_{eq}(C)for methyl H and U_{iso}(H) = 1.2U_{eq}(C,*N*)for other H.

Computing details

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

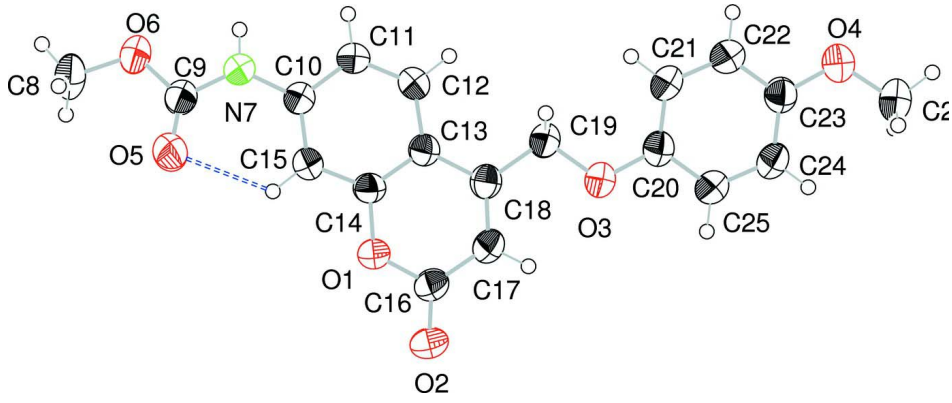

Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level. Dashed lines indicate intramolecular hydrogen bonds.

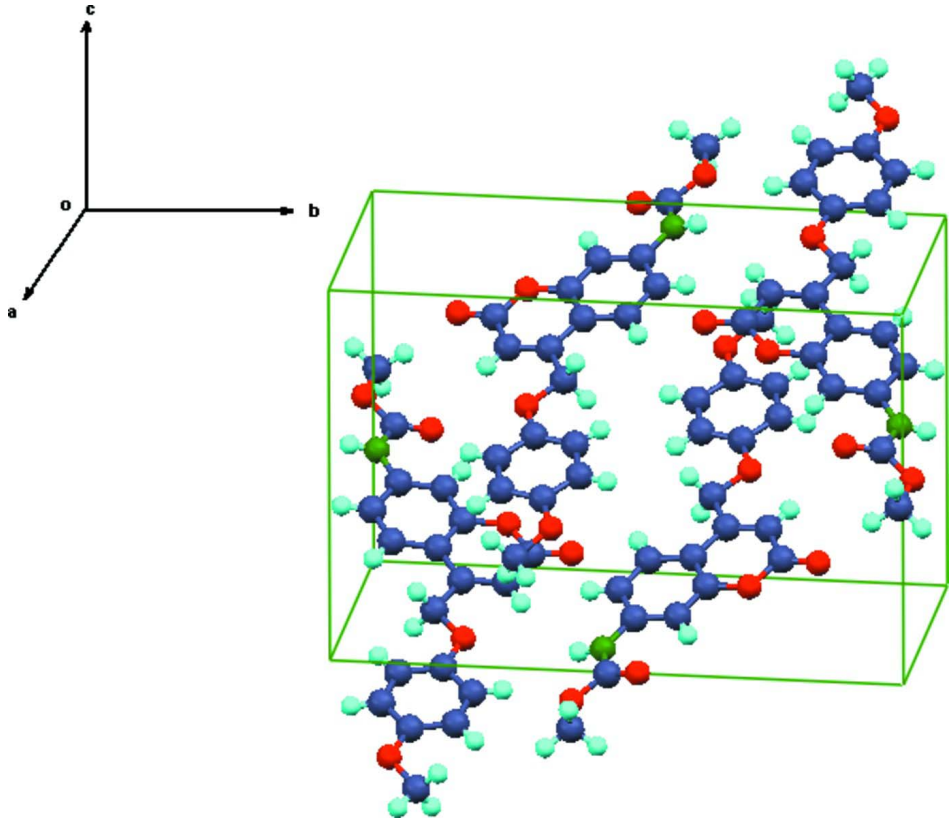

Figure 2

The packing of the molecules in the title structure.

Methyl *N*-{4-[(4-methoxyphenoxy)methyl]-2-oxo-2*H*-chromen-7-yl}carbamate

Crystal data

$C_{19}H_{17}NO_6$	a = 8.3141 (1) Å
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Monoclinic, $P2_1/n$	c = 11.5729 (2) Å
Hall symbol: -P 2yn	β = 94.309 (1)°

V = 1669.25 (5) Å^3
Z = 4
F(000) = 744
D_x = 1.414 Mg m^{-3}
Melting point: 475 K
Mo $K\alpha$ radiation, λ = 0.71073 Å

Cell parameters from 2939 reflections
θ = 2.1–25.0°
μ = 0.11 mm^{-1}
T = 293 K
Plate, colourless
0.24 × 0.20 × 0.12 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2007)
T_{min} = 0.770, T_{max} = 1.000

14973 measured reflections
2939 independent reflections
2374 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.0°, θ_{min} = 2.1°
h = −9→9
k = −20→20
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.103
S = 1.06
2939 reflections
238 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0518P)^2 + 0.2287P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0028 (9)

Special details

Experimental. IR(KBr): 1067 cm^{-1}(C—O—C), 1697 cm^{-1} (NH—C=O), 1727 cm^{-1} (Coumarin C=O), 3261 cm^{-1} (NH). GCMS: m/e: 355.1H NMR (500 MHz, DMSO.D6, \?, p.p.m.): 3.34 (s,3*H*, C_{13}), 3.696(s,3*H*, C_1), 5.2 (s,2*H*, C_6), 6.38 (s 1H, C_{17}), 6.88 (d,2*H*, C_3 & C_{15}), 7.04 (s,2*H*, C_4 & C_{14}), 7.38 (s,1*H*, C_{19}), 7.56 (s,1*H*, C_{10}),7.74 (s,1*H*, C_{18}). Elemental analysis: C, 64.20; H, 4.78; N, 3.91; O, 27.19.

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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.22869 (12)	0.25228 (5)	0.17803 (9)	0.0546 (3)
O2	0.29518 (15)	0.36749 (6)	0.12426 (11)	0.0719 (4)
O3	0.75136 (12)	0.21340 (6)	0.00648 (10)	0.0625 (3)
O4	1.32758 (14)	0.12805 (7)	−0.16477 (12)	0.0738 (4)
O5	−0.16647 (14)	0.08777 (7)	0.31601 (12)	0.0798 (4)

O6	−0.15645 (12)	−0.02844 (7)	0.40139 (10)	0.0651 (3)
N7	0.05554 (14)	0.01046 (7)	0.31564 (11)	0.0551 (3)
H7	0.0887	−0.0347	0.3359	0.066*
C8	−0.3183 (2)	−0.01795 (12)	0.43264 (17)	0.0758 (5)
H8A	−0.3253	0.0291	0.4753	0.114*
H8B	−0.3480	−0.0603	0.4798	0.114*
H8C	−0.3901	−0.0157	0.3638	0.114*
C9	−0.09510 (18)	0.02930 (9)	0.34198 (14)	0.0547 (4)
C10	0.16316 (16)	0.05609 (8)	0.25940 (12)	0.0482 (3)
C11	0.30076 (18)	0.02083 (9)	0.22107 (14)	0.0556 (4)
H11	0.3157	−0.0318	0.2316	0.067*
C12	0.41361 (18)	0.06269 (9)	0.16825 (14)	0.0548 (4)
H12	0.5041	0.0380	0.1435	0.066*
C13	0.39578 (16)	0.14200 (8)	0.15063 (12)	0.0465 (3)
C14	0.25774 (17)	0.17482 (8)	0.18963 (12)	0.0468 (3)
C15	0.14222 (17)	0.13397 (8)	0.24295 (13)	0.0503 (4)
H15	0.0515	0.1586	0.2675	0.060*
C16	0.33358 (18)	0.30026 (9)	0.12863 (14)	0.0544 (4)
C17	0.47631 (18)	0.26706 (9)	0.08719 (14)	0.0534 (4)
H17	0.5491	0.2987	0.0527	0.064*
C18	0.50789 (16)	0.19144 (8)	0.09682 (12)	0.0486 (4)
C19	0.65617 (17)	0.15584 (9)	0.05426 (14)	0.0543 (4)
H19A	0.7178	0.1306	0.1179	0.065*
H19B	0.6260	0.1175	−0.0042	0.065*
C20	0.89376 (17)	0.18941 (9)	−0.03564 (13)	0.0515 (4)
C21	0.94292 (19)	0.11408 (9)	−0.04124 (15)	0.0598 (4)
H21	0.8782	0.0751	−0.0152	0.072*
C22	1.08779 (19)	0.09621 (9)	−0.08528 (15)	0.0612 (4)
H22	1.1200	0.0451	−0.0885	0.073*
C23	1.18506 (18)	0.15266 (9)	−0.12443 (13)	0.0547 (4)
C24	1.13658 (18)	0.22857 (9)	−0.11907 (13)	0.0563 (4)
H24	1.2015	0.2674	−0.1453	0.068*
C25	0.99133 (18)	0.24652 (9)	−0.07463 (13)	0.0552 (4)
H25	0.9591	0.2976	−0.0710	0.066*
C26	1.4187 (2)	0.18198 (11)	−0.22267 (18)	0.0783 (5)
H26A	1.3534	0.2034	−0.2866	0.118*
H26B	1.5110	0.1570	−0.2509	0.118*
H26C	1.4538	0.2223	−0.1700	0.118*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0545 (6)	0.0394 (6)	0.0714 (7)	0.0001 (4)	0.0157 (5)	−0.0011 (5)
O2	0.0771 (8)	0.0378 (6)	0.1031 (10)	−0.0030 (5)	0.0219 (7)	−0.0017 (6)
O3	0.0518 (6)	0.0560 (6)	0.0820 (8)	−0.0025 (5)	0.0206 (6)	0.0081 (6)
O4	0.0598 (7)	0.0671 (8)	0.0982 (9)	0.0010 (6)	0.0296 (6)	0.0054 (6)
O5	0.0574 (7)	0.0748 (9)	0.1098 (10)	0.0136 (6)	0.0223 (7)	0.0293 (7)
O6	0.0536 (6)	0.0648 (7)	0.0789 (8)	−0.0073 (5)	0.0184 (5)	0.0110 (6)
N7	0.0505 (7)	0.0444 (7)	0.0720 (9)	0.0020 (5)	0.0150 (6)	0.0087 (6)
C8	0.0518 (10)	0.0929 (14)	0.0848 (13)	−0.0122 (9)	0.0191 (9)	0.0062 (10)

C9	0.0506 (9)	0.0549 (10)	0.0594 (9)	−0.0030 (7)	0.0082 (7)	0.0061 (7)
C10	0.0459 (8)	0.0465 (8)	0.0527 (8)	−0.0009 (6)	0.0060 (6)	0.0028 (6)
C11	0.0523 (8)	0.0436 (8)	0.0720 (10)	0.0045 (6)	0.0119 (7)	0.0063 (7)
C12	0.0474 (8)	0.0506 (9)	0.0677 (10)	0.0064 (6)	0.0129 (7)	0.0040 (7)
C13	0.0432 (7)	0.0459 (8)	0.0504 (8)	−0.0012 (6)	0.0038 (6)	−0.0007 (6)
C14	0.0480 (8)	0.0402 (8)	0.0522 (8)	0.0009 (6)	0.0038 (6)	−0.0015 (6)
C15	0.0456 (8)	0.0475 (9)	0.0591 (9)	0.0016 (6)	0.0122 (7)	−0.0011 (7)
C16	0.0574 (9)	0.0429 (9)	0.0633 (10)	−0.0069 (7)	0.0070 (7)	−0.0035 (7)
C17	0.0507 (8)	0.0481 (9)	0.0619 (9)	−0.0091 (7)	0.0088 (7)	0.0007 (7)
C18	0.0432 (8)	0.0526 (9)	0.0499 (8)	−0.0042 (6)	0.0016 (6)	−0.0008 (6)
C19	0.0473 (8)	0.0540 (9)	0.0627 (9)	−0.0038 (7)	0.0109 (7)	0.0067 (7)
C20	0.0457 (8)	0.0563 (9)	0.0529 (8)	−0.0042 (7)	0.0058 (6)	0.0023 (7)
C21	0.0552 (9)	0.0511 (9)	0.0746 (11)	−0.0092 (7)	0.0153 (8)	0.0063 (8)
C22	0.0600 (10)	0.0493 (9)	0.0753 (11)	−0.0024 (7)	0.0126 (8)	0.0024 (8)
C23	0.0493 (8)	0.0577 (10)	0.0576 (9)	−0.0024 (7)	0.0077 (7)	0.0005 (7)
C24	0.0536 (9)	0.0550 (9)	0.0612 (9)	−0.0106 (7)	0.0093 (7)	0.0059 (7)
C25	0.0557 (9)	0.0488 (9)	0.0615 (9)	−0.0045 (7)	0.0076 (7)	0.0037 (7)
C26	0.0662 (11)	0.0834 (13)	0.0892 (13)	−0.0052 (9)	0.0309 (10)	0.0102 (10)

Geometric parameters (Å, º)

O1—C16	1.3636 (17)	C13—C18	1.4435 (19)
O1—C14	1.3738 (16)	C14—C15	1.3774 (19)
O2—C16	1.2124 (18)	C15—H15	0.9300
O3—C20	1.3786 (18)	C16—C17	1.434 (2)
O3—C19	1.4144 (17)	C17—C18	1.345 (2)
O4—C23	1.3742 (18)	C17—H17	0.9300
O4—C26	1.407 (2)	C18—C19	1.496 (2)
O5—C9	1.2042 (19)	C19—H19A	0.9700
O6—C9	1.3397 (18)	C19—H19B	0.9700
O6—C8	1.4309 (19)	C20—C21	1.376 (2)
N7—C9	1.3514 (19)	C20—C25	1.380 (2)
N7—C10	1.3935 (18)	C21—C22	1.378 (2)
N7—H7	0.8600	C21—H21	0.9300
C8—H8A	0.9600	C22—C23	1.371 (2)
C8—H8B	0.9600	C22—H22	0.9300
C8—H8C	0.9600	C23—C24	1.384 (2)
C10—C15	1.377 (2)	C24—C25	1.383 (2)
C10—C11	1.3993 (19)	C24—H24	0.9300
C11—C12	1.368 (2)	C25—H25	0.9300
C11—H11	0.9300	C26—H26A	0.9600
C12—C13	1.401 (2)	C26—H26B	0.9600
C12—H12	0.9300	C26—H26C	0.9600
C13—C14	1.3874 (19)		
C16—O1—C14	121.89 (11)	C18—C17—C16	121.85 (14)
C20—O3—C19	116.39 (12)	C18—C17—H17	119.1
C23—O4—C26	117.57 (13)	C16—C17—H17	119.1
C9—O6—C8	115.81 (13)	C17—C18—C13	119.37 (13)
C9—N7—C10	127.28 (13)	C17—C18—C19	122.58 (13)

C9—N7—H7	116.4	C13—C18—C19	118.05 (13)
C10—N7—H7	116.4	O3—C19—C18	109.56 (12)
O6—C8—H8A	109.5	O3—C19—H19A	109.8
O6—C8—H8B	109.5	C18—C19—H19A	109.8
H8A—C8—H8B	109.5	O3—C19—H19B	109.8
O6—C8—H8C	109.5	C18—C19—H19B	109.8
H8A—C8—H8C	109.5	H19A—C19—H19B	108.2
H8B—C8—H8C	109.5	C21—C20—O3	124.84 (13)
O5—C9—O6	124.20 (14)	C21—C20—C25	119.12 (14)
O5—C9—N7	126.60 (14)	O3—C20—C25	116.05 (14)
O6—C9—N7	109.20 (13)	C20—C21—C22	120.22 (15)
C15—C10—N7	123.09 (13)	C20—C21—H21	119.9
C15—C10—C11	119.03 (13)	C22—C21—H21	119.9
N7—C10—C11	117.86 (13)	C23—C22—C21	120.96 (15)
C12—C11—C10	120.86 (14)	C23—C22—H22	119.5
C12—C11—H11	119.6	C21—C22—H22	119.5
C10—C11—H11	119.6	C22—C23—O4	115.74 (14)
C11—C12—C13	121.39 (14)	C22—C23—C24	119.20 (14)
C11—C12—H12	119.3	O4—C23—C24	125.05 (14)
C13—C12—H12	119.3	C25—C24—C23	119.85 (14)
C14—C13—C12	116.04 (13)	C25—C24—H24	120.1
C14—C13—C18	118.16 (13)	C23—C24—H24	120.1
C12—C13—C18	125.80 (13)	C20—C25—C24	120.66 (15)
O1—C14—C15	115.25 (12)	C20—C25—H25	119.7
O1—C14—C13	121.04 (12)	C24—C25—H25	119.7
C15—C14—C13	123.71 (13)	O4—C26—H26A	109.5
C10—C15—C14	118.97 (13)	O4—C26—H26B	109.5
C10—C15—H15	120.5	H26A—C26—H26B	109.5
C14—C15—H15	120.5	O4—C26—H26C	109.5
O2—C16—O1	115.68 (14)	H26A—C26—H26C	109.5
O2—C16—C17	126.62 (14)	H26B—C26—H26C	109.5
O1—C16—C17	117.70 (13)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7···O2^{i}	0.86	1.99	2.8428 (16)	170
C15—H15···O5	0.93	2.30	2.8764 (19)	120
C19—H19*A*···O5^{ii}	0.97	2.53	3.476 (2)	165

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