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3-[(*E*)-1-(Benzyloxyimino)ethyl]-7-(3-methylbut-2-enyloxy)-2*H*-chromen-2-one

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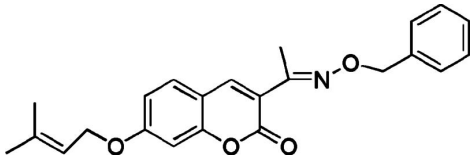
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.155; data-to-parameter ratio = 15.2.

In the title compound, $C_{23}H_{23}NO_4$, the dihedral angle beween the chromen-2-one ring system and the benzene ring is 69.73 (10)° and the molecule adopts an *E* conformation with respect to the C=N double bond. In the crystal, inversion dimers linked by pairs of C—H···O hydrogen bonds occur, generating $R_2^2(12)$ loops.

Related literature

For background to the use of Schiff bases as chemosensors, see: Li *et al.* (2009).



Experimental

Crystal data

$C_{23}H_{23}NO_4$ M_r = 377.42
Triclinic, $P\overline{1}$
a = 7.3038 (19) Å
b = 11.467 (3) Å
c = 12.184 (3) Å
α = 92.368 (3)°
β = 92.067 (3)°
γ = 102.340 (3)°
V = 995.0 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 273 K
0.26 × 0.18 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.978, T_{max} = 0.986
5604 measured reflections
3912 independent reflections
2341 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.155
S = 1.01
3912 reflections
257 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O2^{i}	0.93	2.43	3.338 (3)	167

Symmetry code: (i) $-x, -y, -z+1$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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: *SHELXL97*.

We are grateful to the Science and Technology Plan Project of Guangdong Province (No. 2008B010600008) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5875).

References

Bruker (2004). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, H. Y., Gao, S. & Xi, Z. (2009). *Inorg. Chem. Commun.* **12**, 300–303.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2011). E**67**, o1516 [doi:10.1107/S1600536811019015]

3-[(*E*)-1-(Benzyloxyimino)ethyl]-7-(3-methylbut-2-enyloxy)-2*H*-chromen-2-one

Hui Wang, Li-juan He, Wei-hua Zheng and Hua-can Song

S1. Comment

Coumarin-derived Schiff bases have attracted attention as colorimetric chemosensors (Li *et al.*, 2009). Herein, we report the crystal structure of the title compound, (I), Fig. 1, obtained by the reaction of 3-acetyl-7-(3-methylbut-2-enyloxy)-2*H*-chromen-2-one with benzyloxy-amine. Inversion dimers occur in the crystal, being linked by pairs of C—H···O hydrogen bonds (Table 1).

S2. Experimental

A mixture of 3-acetyl-7-(3-methylbut-2-enyloxy)-2*H*-chromen-2-one (1 mmol) and benzyloxy- amine hydrochloride (1.2 mmol) in ethanol (15 ml) was heated at 313 K for 0.5 h, the solution pH was then maintained at a value of 7 by the addition of sodium carbonate(0.5 mmol). The reaction mixture was refluxed for 10 h at 333 K (monitored by TLC). After completion of the reaction, the solvent was removed under a vacuum. The crude product was purified by chromatography (ethyl acetate: petroleum ether = 3:1). The eluate was evaporated to give the title compound as colourless blocks (279 mg, 74%; m. p. 372–374 K). ESI-MS (m / *z*): [(*M*+Na)+] 400, [(*M*+H)+] 378; IR (KBr, cm-1) 3065, 2971, 2877, 1722, 1603, 1491, 1458, 1357, 1259, 1216, 1129, 981, 923, 772; 1H NMR(400 MHz, CDCl3, TMS) delta 7.79 (s, 1H), 7.40 (d, 1H, J = 8.4 Hz), 7.31–7.36 (m, 5H), 6.83 (dd, 1H, J = 2.8, 8.4 Hz), 6.79(d 1H, J = 2.8 Hz), 5.45–5.50 (m, 1H), 5.23 (s, 2H), 4.56(d, 2H, J = 6.8 Hz), 2.27 (s, 3H), 1.80 (s, 3H), 1.76 (s, 3H); 13 C NMR (100 MHz, CDCl3) delta 162.45, 160.08, 155.90, 154.33, 141.40, 137.72, 129.49, 128.96, 128.41, 128.33, 127.97, 127.84,127.42, 121.30, 118.59, 113.57, 112.43, 101.08, 76.21, 65.46, 25.83,18.30,14.61.

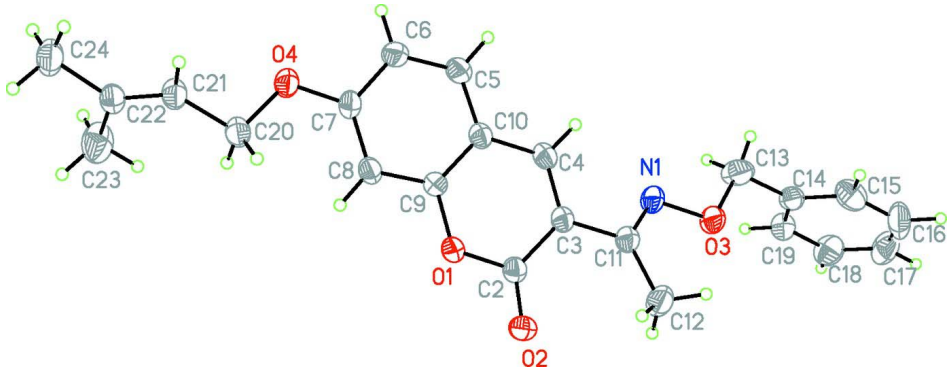


Figure 1

The molecular structure of the title compound showing the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

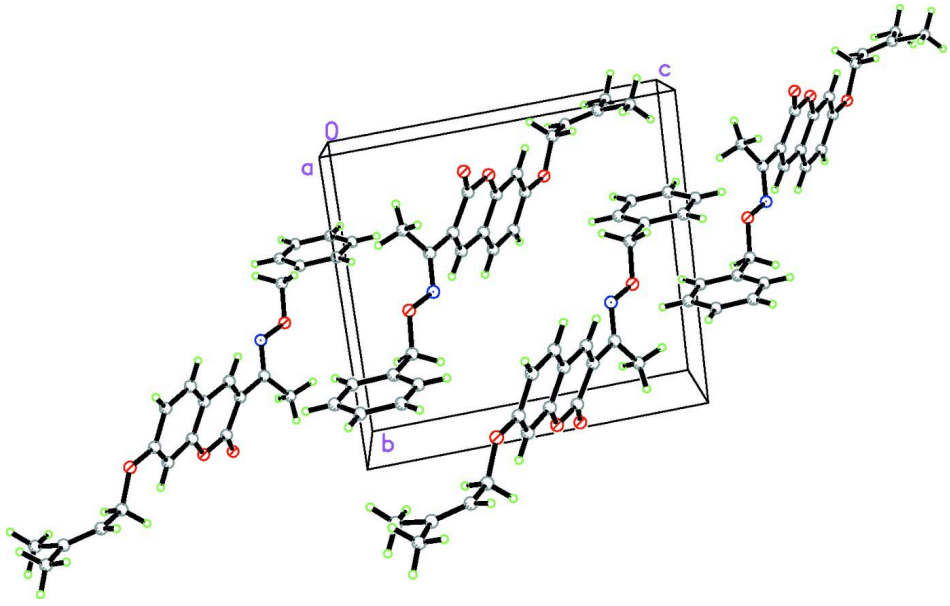


Figure 2

Crystal packing in the title compound.

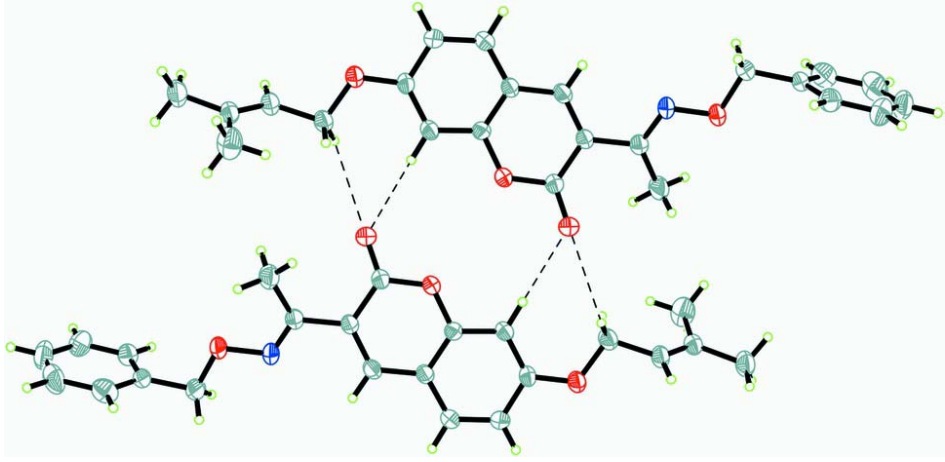


Figure 3

Part of the crystal structure of the title compound showing weak C—H···O hydrogen bonds as dashed lines.

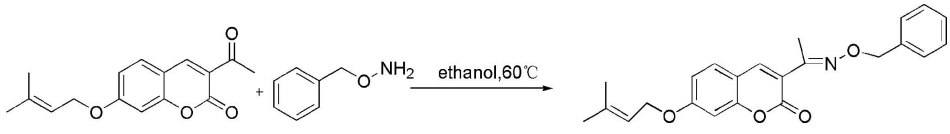


Figure 4

The formation of the title compound.

3-[(*E*)-1-(Benzyloxyimino)ethyl]-7-(3-methylbut-2-enyloxy)- 2*H*-chromen-2-one

Crystal data

$C_{23}H_{23}NO_4$
$M_r = 377.42$

Triclinic, $P\overline{1}$
Hall symbol: -P 1

a = 7.3038 (19) Å
b = 11.467 (3) Å
c = 12.184 (3) Å
α = 92.368 (3)°
β = 92.067 (3)°
γ = 102.340 (3)°
V = 995.0 (5) Å^3
Z = 2
F(000) = 430.0

D_x = 1.260 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1391 reflections
θ = 2.4–24.9°
μ = 0.09 mm^{-1}
T = 273 K
Block, colorless
0.26 × 0.18 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.978, T_{max} = 0.986

5604 measured reflections
3912 independent reflections
2341 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 26.3°, θ_{min} = 1.8°
h = −9→8
k = −10→14
l = −14→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.155
S = 1.01
3912 reflections
257 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
$w = 1/[\sigma^2(F_o^2) + (0.0644P)^2 + 0.1016P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.028
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.049 (5)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'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 $F^2 > \sigma(F^2)$ 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}
N1	0.4410 (2)	0.43803 (16)	0.26381 (14)	0.0548 (5)
O3	0.59306 (19)	0.46642 (13)	0.19438 (12)	0.0611 (4)
O2	0.3010 (2)	0.10766 (13)	0.38943 (12)	0.0622 (4)
O1	0.04470 (18)	0.14660 (12)	0.45666 (10)	0.0492 (4)
O4	−0.50894 (19)	0.21552 (13)	0.61925 (11)	0.0558 (4)
C19	0.9868 (3)	0.6503 (2)	0.19649 (18)	0.0627 (6)

H19	0.9821	0.6483	0.2726	0.075*
C18	1.1555 (3)	0.6776 (2)	0.1505 (2)	0.0776 (8)
H18	1.2648	0.6934	0.1952	0.093*
C17	1.1667 (4)	0.6820 (3)	0.0413 (3)	0.0852 (9)
H17	1.2835	0.7011	0.0107	0.102*
C16	1.0072 (5)	0.6584 (3)	−0.0255 (2)	0.0898 (9)
H16	1.0149	0.6616	−0.1014	0.108*
C15	0.8327 (4)	0.6296 (2)	0.0215 (2)	0.0743 (7)
H15	0.7235	0.6132	−0.0232	0.089*
C14	0.8222 (3)	0.62545 (18)	0.13341 (18)	0.0539 (6)
C13	0.6388 (4)	0.5931 (2)	0.1861 (3)	0.0872 (9)
H13B	0.6471	0.6325	0.2587	0.105*
H13A	0.5422	0.6184	0.1423	0.105*
C11	0.3842 (3)	0.32488 (19)	0.26814 (16)	0.0485 (5)
C12	0.4680 (4)	0.2348 (2)	0.2070 (2)	0.0742 (7)
H12C	0.5063	0.2642	0.1369	0.111*
H12A	0.3765	0.1611	0.1961	0.111*
H12B	0.5750	0.2214	0.2486	0.111*
C3	0.2208 (3)	0.28938 (17)	0.33767 (15)	0.0449 (5)
C2	0.1984 (3)	0.17731 (18)	0.39329 (15)	0.0462 (5)
C9	−0.0825 (3)	0.21796 (17)	0.47168 (14)	0.0418 (5)
C4	0.0943 (3)	0.35857 (18)	0.35299 (15)	0.0481 (5)
H4	0.1091	0.4300	0.3175	0.058*
C10	−0.0610 (3)	0.32596 (17)	0.42160 (15)	0.0437 (5)
C5	−0.1943 (3)	0.39482 (19)	0.44254 (17)	0.0530 (5)
H5	−0.1839	0.4680	0.4104	0.064*
C6	−0.3389 (3)	0.35666 (19)	0.50912 (17)	0.0529 (5)
H6	−0.4246	0.4042	0.5231	0.064*
C8	−0.2289 (3)	0.17562 (18)	0.53828 (15)	0.0460 (5)
H8	−0.2400	0.1022	0.5699	0.055*
C7	−0.3581 (3)	0.24560 (18)	0.55642 (15)	0.0458 (5)
C20	−0.5503 (3)	0.0961 (2)	0.65844 (17)	0.0564 (6)
H20B	−0.4576	0.0878	0.7150	0.068*
H20A	−0.5481	0.0381	0.5985	0.068*
C21	−0.7401 (3)	0.07576 (19)	0.70411 (16)	0.0536 (5)
H21	−0.8390	0.0794	0.6550	0.064*
C22	−0.7839 (3)	0.05328 (19)	0.80605 (16)	0.0516 (5)
C24	−0.6452 (4)	0.0473 (3)	0.89720 (19)	0.0908 (9)
H24C	−0.5213	0.0617	0.8696	0.136*
H24A	−0.6738	−0.0304	0.9268	0.136*
H24B	−0.6510	0.1069	0.9539	0.136*
C23	−0.9843 (3)	0.0309 (2)	0.8392 (2)	0.0740 (7)
H23B	−1.0643	0.0390	0.7770	0.111*
H23C	−0.9965	0.0879	0.8969	0.111*
H23A	−1.0200	−0.0484	0.8648	0.111*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0458 (10)	0.0545 (12)	0.0642 (11)	0.0066 (9)	0.0195 (8)	0.0119 (9)
O3	0.0547 (9)	0.0495 (10)	0.0799 (10)	0.0063 (7)	0.0285 (8)	0.0151 (7)
O2	0.0598 (9)	0.0552 (10)	0.0791 (10)	0.0235 (8)	0.0188 (8)	0.0198 (8)
O1	0.0493 (8)	0.0467 (9)	0.0545 (8)	0.0125 (7)	0.0135 (6)	0.0161 (6)
O4	0.0584 (9)	0.0500 (9)	0.0602 (9)	0.0102 (7)	0.0212 (7)	0.0094 (7)
C19	0.0675 (15)	0.0652 (16)	0.0569 (13)	0.0157 (13)	0.0105 (11)	0.0086 (11)
C18	0.0535 (15)	0.089 (2)	0.0876 (19)	0.0105 (14)	0.0042 (13)	0.0007 (15)
C17	0.0681 (18)	0.089 (2)	0.096 (2)	0.0059 (15)	0.0369 (16)	0.0044 (16)
C16	0.122 (3)	0.093 (2)	0.0524 (15)	0.0134 (19)	0.0287 (17)	0.0138 (14)
C15	0.0760 (18)	0.0710 (18)	0.0716 (16)	0.0083 (14)	−0.0149 (13)	0.0084 (13)
C14	0.0487 (13)	0.0434 (13)	0.0715 (14)	0.0097 (10)	0.0176 (11)	0.0137 (10)
C13	0.0692 (17)	0.0507 (16)	0.150 (3)	0.0176 (13)	0.0500 (17)	0.0331 (16)
C11	0.0480 (12)	0.0480 (13)	0.0491 (11)	0.0080 (10)	0.0081 (9)	0.0058 (9)
C12	0.0839 (18)	0.0575 (16)	0.0832 (17)	0.0131 (13)	0.0391 (14)	0.0051 (13)
C3	0.0458 (11)	0.0430 (12)	0.0453 (10)	0.0064 (9)	0.0075 (8)	0.0072 (9)
C2	0.0449 (11)	0.0469 (13)	0.0467 (11)	0.0082 (10)	0.0057 (9)	0.0072 (9)
C9	0.0435 (11)	0.0416 (12)	0.0412 (10)	0.0108 (9)	0.0031 (8)	0.0047 (8)
C4	0.0508 (12)	0.0435 (12)	0.0489 (11)	0.0060 (10)	0.0046 (9)	0.0111 (9)
C10	0.0431 (11)	0.0433 (12)	0.0436 (10)	0.0059 (9)	0.0032 (8)	0.0075 (9)
C5	0.0536 (13)	0.0454 (13)	0.0616 (13)	0.0110 (10)	0.0085 (10)	0.0146 (10)
C6	0.0519 (12)	0.0448 (13)	0.0649 (13)	0.0144 (10)	0.0097 (10)	0.0085 (10)
C8	0.0511 (12)	0.0427 (12)	0.0436 (10)	0.0069 (10)	0.0064 (9)	0.0084 (9)
C7	0.0465 (11)	0.0465 (12)	0.0425 (10)	0.0042 (9)	0.0101 (8)	0.0046 (9)
C20	0.0637 (14)	0.0540 (14)	0.0522 (12)	0.0108 (11)	0.0156 (10)	0.0106 (10)
C21	0.0486 (12)	0.0603 (14)	0.0501 (12)	0.0054 (10)	0.0072 (9)	0.0111 (10)
C22	0.0539 (13)	0.0507 (13)	0.0490 (11)	0.0066 (10)	0.0091 (9)	0.0062 (9)
C24	0.0810 (19)	0.136 (3)	0.0548 (14)	0.0192 (18)	0.0014 (13)	0.0171 (15)
C23	0.0656 (16)	0.0839 (19)	0.0709 (16)	0.0066 (14)	0.0209 (12)	0.0184 (13)

Geometric parameters (Å, º)

N1—C11	1.279 (3)	C3—C4	1.354 (3)
N1—O3	1.4111 (19)	C3—C2	1.459 (3)
O3—C13	1.428 (3)	C9—C8	1.382 (2)
O2—C2	1.207 (2)	C9—C10	1.385 (3)
O1—C9	1.375 (2)	C4—C10	1.428 (3)
O1—C2	1.377 (2)	C4—H4	0.9300
O4—C7	1.356 (2)	C10—C5	1.402 (3)
O4—C20	1.443 (2)	C5—C6	1.363 (3)
C19—C18	1.353 (3)	C5—H5	0.9300
C19—C14	1.373 (3)	C6—C7	1.401 (3)
C19—H19	0.9300	C6—H6	0.9300
C18—C17	1.338 (3)	C8—C7	1.381 (3)
C18—H18	0.9300	C8—H8	0.9300
C17—C16	1.368 (4)	C20—C21	1.488 (3)

C17—H17	0.9300	C20—H20B	0.9700
C16—C15	1.396 (4)	C20—H20A	0.9700
C16—H16	0.9300	C21—C22	1.316 (3)
C15—C14	1.371 (3)	C21—H21	0.9300
C15—H15	0.9300	C22—C24	1.490 (3)
C14—C13	1.488 (3)	C22—C23	1.504 (3)
C13—H13B	0.9700	C24—H24C	0.9600
C13—H13A	0.9700	C24—H24A	0.9600
C11—C3	1.483 (3)	C24—H24B	0.9600
C11—C12	1.495 (3)	C23—H23B	0.9600
C12—H12C	0.9600	C23—H23C	0.9600
C12—H12A	0.9600	C23—H23A	0.9600
C12—H12B	0.9600		

C11—N1—O3	111.18 (16)	O1—C9—C10	120.44 (16)
N1—O3—C13	108.13 (15)	C8—C9—C10	123.15 (18)
C9—O1—C2	123.03 (15)	C3—C4—C10	122.46 (18)
C7—O4—C20	117.49 (15)	C3—C4—H4	118.8
C18—C19—C14	121.5 (2)	C10—C4—H4	118.8
C18—C19—H19	119.2	C9—C10—C5	117.12 (17)
C14—C19—H19	119.2	C9—C10—C4	117.73 (18)
C17—C18—C19	120.6 (3)	C5—C10—C4	125.15 (18)
C17—C18—H18	119.7	C6—C5—C10	121.29 (19)
C19—C18—H18	119.7	C6—C5—H5	119.4
C18—C17—C16	120.4 (2)	C10—C5—H5	119.4
C18—C17—H17	119.8	C5—C6—C7	119.89 (19)
C16—C17—H17	119.8	C5—C6—H6	120.1
C17—C16—C15	119.2 (2)	C7—C6—H6	120.1
C17—C16—H16	120.4	C7—C8—C9	118.06 (18)
C15—C16—H16	120.4	C7—C8—H8	121.0
C14—C15—C16	120.1 (2)	C9—C8—H8	121.0
C14—C15—H15	119.9	O4—C7—C8	124.63 (18)
C16—C15—H15	119.9	O4—C7—C6	114.90 (17)
C15—C14—C19	118.1 (2)	C8—C7—C6	120.47 (18)
C15—C14—C13	121.5 (2)	O4—C20—C21	107.58 (17)
C19—C14—C13	120.3 (2)	O4—C20—H20B	110.2
O3—C13—C14	108.12 (18)	C21—C20—H20B	110.2
O3—C13—H13B	110.1	O4—C20—H20A	110.2
C14—C13—H13B	110.1	C21—C20—H20A	110.2
O3—C13—H13A	110.1	H20B—C20—H20A	108.5
C14—C13—H13A	110.1	C22—C21—C20	127.4 (2)
H13B—C13—H13A	108.4	C22—C21—H21	116.3
N1—C11—C3	113.69 (18)	C20—C21—H21	116.3
N1—C11—C12	124.18 (19)	C21—C22—C24	124.4 (2)
C3—C11—C12	122.1 (2)	C21—C22—C23	121.23 (19)
C11—C12—H12C	109.5	C24—C22—C23	114.42 (18)
C11—C12—H12A	109.5	C22—C24—H24C	109.5
H12C—C12—H12A	109.5	C22—C24—H24A	109.5

C11—C12—H12B	109.5	H24C—C24—H24A	109.5
H12C—C12—H12B	109.5	C22—C24—H24B	109.5
H12A—C12—H12B	109.5	H24C—C24—H24B	109.5
C4—C3—C2	118.98 (17)	H24A—C24—H24B	109.5
C4—C3—C11	122.25 (18)	C22—C23—H23B	109.5
C2—C3—C11	118.75 (17)	C22—C23—H23C	109.5
O2—C2—O1	116.02 (17)	H23B—C23—H23C	109.5
O2—C2—C3	126.66 (18)	C22—C23—H23A	109.5
O1—C2—C3	117.31 (17)	H23B—C23—H23A	109.5
O1—C9—C8	116.41 (16)	H23C—C23—H23A	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O2^{i}	0.93	2.43	3.338 (3)	167

Symmetry code: (i) −*x*, −*y*, −*z*+1.