IUCrData

ISSN 2414-3146

Methyl 3,3,6,6-tetramethyl-1,8-dioxo-4,5,7,9-tetrahydro-2*H*-xanthene-9-carboxylate

Heiner Detert,* Laura Kluge and Dieter Schollmeyer

Johannes Gutenberg University Mainz, Department of Chemistry, Duesbergweg 10-14, 55099 Mainz, Germany. *Correspondence e-mail: detert@uni-mainz.de

Received 27 May 2020
Accepted 23 July 2020

Edited by J. F. Gallagher, Dublin City University, Ireland

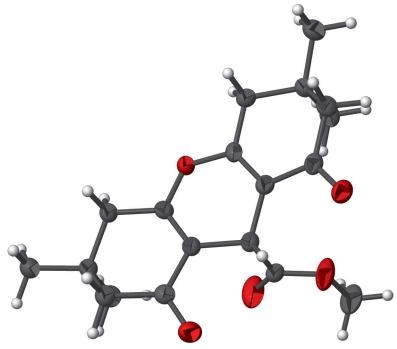
Keywords: crystal structure; heterocycles; polycyclic system.

CCDC reference: 2018468

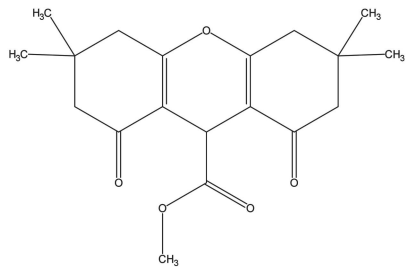
Structural data: full structural data are available from iucrdata.iucr.org

The title molecule, $C_{19}H_{24}O_5$, is built by annulation of a half-chair cyclohexenone and a twist-cyclohexenone to a flat 4-*H*-pyrane boat. In the crystal, molecules are connected *via* van der Waals interactions and C—H···O hydrogen bonds.

3D view



Chemical scheme



Structure description

The title compound was obtained as a side product during the formation of methyl methoxy(2,6-dioxo-4,4-dimethylcyclohexyl)acetate according to the procedure of Grosz & Freiberg (1966). A similar product (1,2,3,4,5,6,7,8-octahydro-3,3,6,6-tetramethyl-1,8-dioxo-9-xanthenyl acetic acid) was obtained by Gustafsson (1948) in the condensation of dimedone and glyoxalic acid. The free acid is an isomer of the title compound with a methylene group connecting the heterocyclic unit and carboxylic acid group. A short route to these compounds is the uncatalysed tandem aldol condensation/elimination/Michael addition/condensation, as discovered by Rohr & Mahrwald (2009).

The molecule is composed of two dimethylcyclohexenone units annulated to a central 4*H*-pyrane (Fig. 1). While the conformation of the latter is a flat boat, one cyclohexenone (C2–C7) forms a half-chair and the other (C9–C14) has a twist form. The pyrane boat promotes a folded shape of the molecule, the angle between the mean planes through atoms C1–C3/C6/C7/O8 and O8/C9/C10/C13//C14 being 22.42 (11)°, with maximum deviations from the mean planes at O8 [−0.1046 (18) Å] and C1 [0.051 (3) Å]. The torsion angle of the ester group (O17—C15—C1—C2) is 66.4 (3)°.

Four molecules occupy the monoclinic unit cell, the packing in the cell being dominated by van der Waals interactions and hydrogen-bonding interactions (Table 1 and Fig. 2). The C—H···O hydrogen bonds (Steiner, 1996) C18—H18*A*···O17 and C18—H18*A*···O19 form a hydrogen-bonded dimer while the C6—H6*B*···O24 interaction connects two molecules related by the *c*-glide plane.

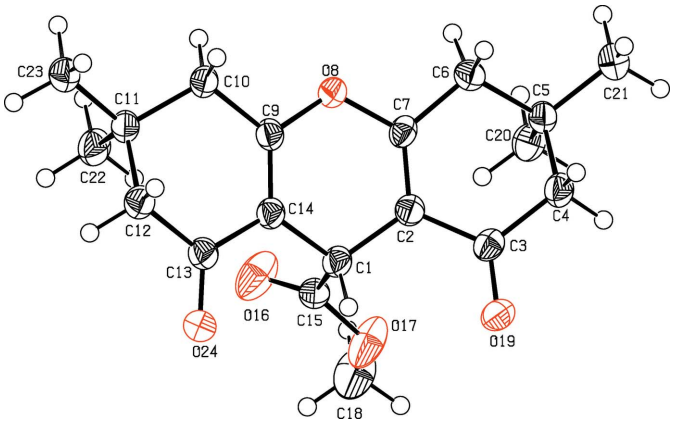


Figure 1
Perspective view of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

Synthesis and crystallization

Dimedone (7.01 g, 0.05 mol, 1 eq.) and triethylamine (5.05 g, 50 mmol, 6.9 ml, 1 eq.) were dissolved in dichloromethane (25 ml) in a 250 ml flask under nitrogen. Methyl chloromethoxyacetate (5.9 ml, 6.49 g, 0.525 mol, 1.05 eq.) was added dropwise to the ice-cooled mixture under stirring and the stirring was continued for 75 min at room temperature and a further 3 h under reflux conditions. The solvent was evaporated, methyl *tert*-butyl ether was added to the suspension and triethyl ammonium chloride was removed *via* filtration. The etheral layer was washed with aqueous sodium carbonate and brine, and dried over sodium sulfate. Evaporation of the solvent and chromatography (silica gel, petroleum ether/ethyl acetate = 3/1, R_f = 3:1) yielded 0.83 g (2.5 mmol, 5%) of the title compound as colourless crystals with m.p. = 474–478 K. The main product yield was 83%. Crystals of the title compound were obtained from a solution in ethyl acetate. IR: 2959, 2875, 1728, 1663, 1368, 1193, 995. ^{1}H NMR (300 MHz, $CDCl_3$) δ/p.p.m.: 4.47–4.46 (*s*, 1H), 3.68 (*s*, 3H), 2.43 (2**d*, 2*2 *gem* H, J = 18 Hz), 4H), 2.27 (2**d*, 2*2 *gem* H, J = 18 Hz), 1.11 (*s*, 12H).

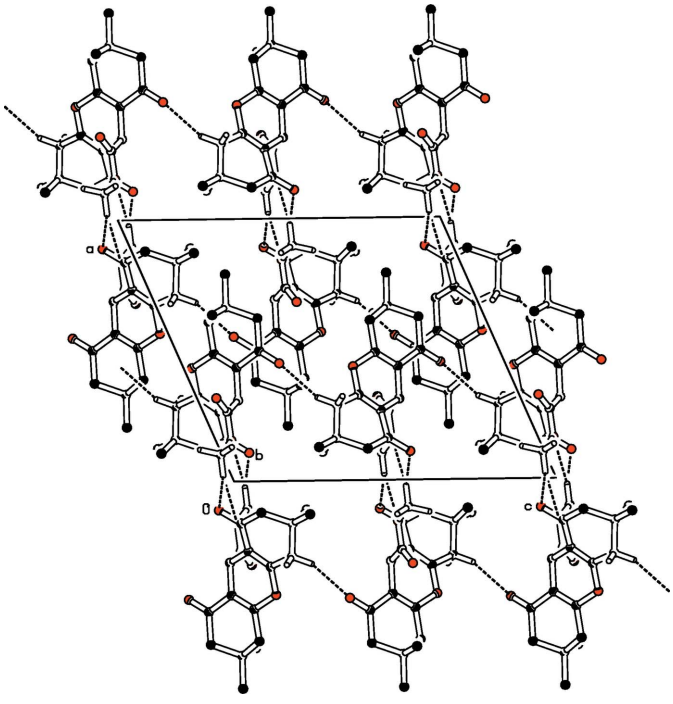


Figure 2
Partial packing diagram of the title compound with a view along the *b* axis. Most of the hydrogen atoms omitted for clarity. Hydrogen bonds are depicted with dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6B···O24^{i}	0.99	2.50	3.324 (4)	140
C18—H18A···O17^{ii}	0.98	2.53	3.451 (5)	156
C18—H18A···O19^{ii}	0.98	2.41	3.093 (4)	126

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{19}H_{24}O_5$
M_r	332.38
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	120
a, b, c (Å)	13.1494 (10), 9.6899 (6), 14.8185 (13)
β (°)	113.295 (6)
V (Å^3)	1734.2 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.09
Crystal size (mm)	0.22 × 0.11 × 0.06
Data collection	
Diffractometer	Stoe IPDS 2T
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8583, 4123, 2784
R_{int}	0.036
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.659
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.072, 0.185, 1.05
No. of reflections	4123
No. of parameters	222
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.30

Computer programs: *X-AREA WinXpose*, *X-AREA Recipe* and *X-AREA Integrate* (Stoe & Cie, 2019), *SIR2004* (Burla *et al.*, 2005), *SHELXL2018/3* (Sheldrick, 2015) and *PLATON* (Spek, 2020).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

References

Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Gross, H. & Freiberg, J. (1966). *Chem. Ber.* **99**, 3260–3267.
Gustafsson, C. (1948). *Suomen Kemistilehti B*, **21**, 3–5.

Rohr, K. & Mahrwald, R. (2009). *Bioorg. Med. Chem. Lett.* **19**, 3949–3951.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.
Steiner, T. (1996). *Crystallogr. Rev.* **6**, 1–51.
Stoe & Cie (2019). *X-RED* and *X-AREA*. Stoe & Cie, Darmstadt, Germany.

full crystallographic data

IUCrData (2020). **5**, x201018 [https://doi.org/10.1107/S2414314620010184]

Methyl 3,3,6,6-tetramethyl-1,8-dioxo-4,5,7,9-tetrahydro-2*H*-xanthene-9-carboxylate

Heiner Detert, Laura Kluge and Dieter Schollmeyer

Methyl 3,3,6,6-tetramethyl-1,8-dioxo-4,5,7,9-tetrahydro-2*H*-xanthene-9-carboxylate

Crystal data

$C_{19}H_{24}O_5$
M_r = 332.38
Monoclinic, $P2_1/c$
a = 13.1494 (10) Å
b = 9.6899 (6) Å
c = 14.8185 (13) Å
β = 113.295 (6)°
V = 1734.2 (2) Å^3
Z = 4
$F(000)$ = 712
D_x = 1.273 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9146 reflections
θ = 2.6–28.2°
μ = 0.09 mm^{-1}
T = 120 K
Plate, colourless
0.22 × 0.11 × 0.06 mm

Data collection

STOE IPDS 2T diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Detector resolution: 6.67 pixels mm^{-1}
rotation method, ω scans
8583 measured reflections
4123 independent reflections
2784 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 28.0°, θ_{min} = 2.6°
h = −16→17
k = −12→12
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.185
S = 1.05
4123 reflections
222 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 2.4014P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

Special details

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. Hydrogen atoms attached to carbons were placed at calculated positions and were refined in the riding-model approximation with C–H = 0.95 Å, and with U_{iso}(H) = 1.2 U_{eq}(C).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6910 (2)	0.3522 (3)	0.3759 (2)	0.0290 (5)
H1	0.715526	0.337644	0.320767	0.035*
C2	0.7343 (2)	0.2359 (3)	0.4490 (2)	0.0284 (5)
C3	0.8403 (2)	0.1694 (3)	0.4650 (2)	0.0292 (5)
C4	0.8801 (2)	0.0564 (3)	0.5415 (2)	0.0327 (6)
H4A	0.962100	0.056545	0.570806	0.039*
H4B	0.855649	−0.033835	0.508675	0.039*
C5	0.8385 (2)	0.0705 (3)	0.6237 (2)	0.0302 (6)
C6	0.7113 (2)	0.0808 (3)	0.5759 (2)	0.0313 (6)
H6A	0.679951	−0.010095	0.548221	0.038*
H6B	0.683107	0.105346	0.626632	0.038*
C7	0.67398 (19)	0.1865 (3)	0.49633 (19)	0.0270 (5)
O8	0.56625 (14)	0.22547 (18)	0.47408 (13)	0.0293 (4)
C9	0.5126 (2)	0.2955 (3)	0.38683 (19)	0.0283 (5)
C10	0.3910 (2)	0.3002 (3)	0.3590 (2)	0.0301 (6)
H10A	0.375949	0.313286	0.418829	0.036*
H10B	0.358002	0.211094	0.328896	0.036*
C11	0.3362 (2)	0.4177 (3)	0.2862 (2)	0.0312 (6)
C12	0.3794 (2)	0.4120 (3)	0.2043 (2)	0.0329 (6)
H12A	0.354823	0.324715	0.167285	0.040*
H12B	0.347003	0.489267	0.158080	0.040*
C13	0.5040 (2)	0.4207 (3)	0.2430 (2)	0.0302 (5)
C14	0.5668 (2)	0.3511 (3)	0.33598 (19)	0.0276 (5)
C15	0.7354 (2)	0.4908 (3)	0.4252 (2)	0.0307 (6)
O16	0.68175 (18)	0.5800 (2)	0.4402 (2)	0.0579 (7)
O17	0.84313 (17)	0.5003 (2)	0.4521 (2)	0.0606 (8)
C18	0.8958 (3)	0.6248 (3)	0.5039 (3)	0.0611 (11)
H18A	0.976272	0.616654	0.525273	0.092*
H18B	0.877556	0.637293	0.561397	0.092*
H18C	0.869276	0.704400	0.460044	0.092*
O19	0.89116 (15)	0.19842 (19)	0.41368 (14)	0.0334 (4)
C20	0.8901 (2)	0.1983 (3)	0.6859 (2)	0.0369 (6)
H20A	0.971000	0.190656	0.712677	0.055*
H20B	0.865283	0.204928	0.740014	0.055*
H20C	0.867042	0.281133	0.644830	0.055*
C21	0.8723 (2)	−0.0569 (3)	0.6899 (2)	0.0407 (7)
H21A	0.841283	−0.139699	0.650705	0.061*
H21B	0.844160	−0.048635	0.741779	0.061*
H21C	0.953273	−0.063927	0.719617	0.061*
C22	0.3647 (2)	0.5576 (3)	0.3387 (2)	0.0386 (7)
H22A	0.332418	0.631853	0.290953	0.058*
H22B	0.445306	0.568687	0.369190	0.058*
H22C	0.334574	0.561723	0.389470	0.058*
C23	0.2108 (2)	0.3990 (3)	0.2434 (2)	0.0386 (7)
H23A	0.175703	0.475736	0.198843	0.058*

H23B	0.184611	0.397602	0.296837	0.058*
H23C	0.191455	0.311806	0.207127	0.058*
O24	0.55164 (16)	0.4811 (2)	0.19806 (15)	0.0394 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0238 (11)	0.0255 (12)	0.0370 (14)	−0.0006 (10)	0.0111 (10)	0.0008 (10)
C2	0.0257 (12)	0.0230 (12)	0.0356 (14)	−0.0012 (10)	0.0110 (11)	−0.0004 (10)
C3	0.0251 (12)	0.0255 (12)	0.0373 (15)	−0.0023 (10)	0.0125 (11)	−0.0049 (11)
C4	0.0301 (13)	0.0271 (13)	0.0409 (16)	0.0045 (11)	0.0142 (12)	0.0013 (11)
C5	0.0253 (12)	0.0290 (13)	0.0340 (14)	0.0027 (10)	0.0095 (11)	0.0048 (11)
C6	0.0294 (12)	0.0268 (12)	0.0375 (15)	−0.0010 (11)	0.0131 (11)	0.0041 (11)
C7	0.0209 (11)	0.0234 (11)	0.0349 (14)	−0.0004 (9)	0.0091 (10)	−0.0030 (10)
O8	0.0231 (8)	0.0295 (9)	0.0348 (10)	0.0014 (7)	0.0109 (7)	0.0041 (8)
C9	0.0268 (12)	0.0237 (12)	0.0326 (14)	0.0025 (10)	0.0100 (10)	0.0026 (10)
C10	0.0240 (12)	0.0266 (13)	0.0385 (15)	0.0018 (10)	0.0110 (11)	0.0034 (11)
C11	0.0251 (12)	0.0267 (13)	0.0402 (15)	0.0024 (10)	0.0113 (11)	0.0050 (11)
C12	0.0279 (12)	0.0315 (13)	0.0380 (15)	0.0011 (11)	0.0115 (11)	0.0021 (11)
C13	0.0297 (12)	0.0256 (12)	0.0351 (14)	0.0012 (10)	0.0125 (11)	0.0017 (11)
C14	0.0244 (11)	0.0229 (12)	0.0344 (14)	0.0005 (10)	0.0103 (10)	0.0008 (10)
C15	0.0262 (12)	0.0258 (12)	0.0396 (15)	−0.0022 (10)	0.0124 (11)	0.0033 (11)
O16	0.0392 (12)	0.0427 (13)	0.094 (2)	−0.0050 (10)	0.0282 (13)	−0.0255 (13)
O17	0.0286 (11)	0.0317 (11)	0.116 (2)	−0.0079 (9)	0.0229 (13)	−0.0240 (13)
C18	0.0410 (17)	0.0309 (16)	0.104 (3)	−0.0120 (14)	0.0204 (19)	−0.0201 (18)
O19	0.0305 (9)	0.0317 (10)	0.0425 (11)	−0.0013 (8)	0.0193 (9)	−0.0026 (8)
C20	0.0311 (13)	0.0402 (16)	0.0355 (15)	0.0013 (12)	0.0089 (12)	−0.0025 (12)
C21	0.0368 (15)	0.0381 (16)	0.0460 (18)	0.0077 (13)	0.0151 (13)	0.0103 (13)
C22	0.0377 (14)	0.0295 (14)	0.0512 (18)	0.0048 (12)	0.0204 (14)	−0.0010 (13)
C23	0.0289 (13)	0.0376 (15)	0.0461 (17)	0.0024 (12)	0.0112 (12)	0.0106 (13)
O24	0.0367 (10)	0.0391 (11)	0.0470 (12)	0.0054 (9)	0.0214 (10)	0.0129 (9)

Geometric parameters (Å, º)

C1—C14	1.501 (3)	C11—C12	1.532 (4)
C1—C2	1.510 (4)	C11—C22	1.534 (4)
C1—C15	1.530 (4)	C12—C13	1.508 (3)
C1—H1	1.0000	C12—H12A	0.9900
C2—C7	1.337 (3)	C12—H12B	0.9900
C2—C3	1.468 (3)	C13—O24	1.229 (3)
C3—O19	1.228 (3)	C13—C14	1.461 (4)
C3—C4	1.513 (4)	C15—O16	1.191 (3)
C4—C5	1.526 (4)	C15—O17	1.314 (3)
C4—H4A	0.9900	O17—C18	1.450 (4)
C4—H4B	0.9900	C18—H18A	0.9800
C5—C21	1.529 (4)	C18—H18B	0.9800
C5—C20	1.533 (4)	C18—H18C	0.9800
C5—C6	1.541 (3)	C20—H20A	0.9800

C6—C7	1.490 (4)	C20—H20B	0.9800
C6—H6A	0.9900	C20—H20C	0.9800
C6—H6B	0.9900	C21—H21A	0.9800
C7—O8	1.374 (3)	C21—H21B	0.9800
O8—C9	1.381 (3)	C21—H21C	0.9800
C9—C14	1.338 (3)	C22—H22A	0.9800
C9—C10	1.486 (3)	C22—H22B	0.9800
C10—C11	1.538 (4)	C22—H22C	0.9800
C10—H10A	0.9900	C23—H23A	0.9800
C10—H10B	0.9900	C23—H23B	0.9800
C11—C23	1.526 (3)	C23—H23C	0.9800

C14—C1—C2	108.7 (2)	C22—C11—C10	110.2 (2)
C14—C1—C15	110.2 (2)	C13—C12—C11	112.6 (2)
C2—C1—C15	110.4 (2)	C13—C12—H12A	109.1
C14—C1—H1	109.2	C11—C12—H12A	109.1
C2—C1—H1	109.2	C13—C12—H12B	109.1
C15—C1—H1	109.2	C11—C12—H12B	109.1
C7—C2—C3	118.7 (2)	H12A—C12—H12B	107.8
C7—C2—C1	120.7 (2)	O24—C13—C14	120.7 (2)
C3—C2—C1	120.5 (2)	O24—C13—C12	122.0 (2)
O19—C3—C2	121.0 (2)	C14—C13—C12	117.2 (2)
O19—C3—C4	121.2 (2)	C9—C14—C13	119.2 (2)
C2—C3—C4	117.7 (2)	C9—C14—C1	121.3 (2)
C3—C4—C5	114.0 (2)	C13—C14—C1	119.4 (2)
C3—C4—H4A	108.8	O16—C15—O17	122.7 (3)
C5—C4—H4A	108.8	O16—C15—C1	125.7 (2)
C3—C4—H4B	108.8	O17—C15—C1	111.6 (2)
C5—C4—H4B	108.8	C15—O17—C18	116.8 (2)
H4A—C4—H4B	107.7	O17—C18—H18A	109.5
C4—C5—C21	109.6 (2)	O17—C18—H18B	109.5
C4—C5—C20	109.8 (2)	H18A—C18—H18B	109.5
C21—C5—C20	108.6 (2)	O17—C18—H18C	109.5
C4—C5—C6	107.8 (2)	H18A—C18—H18C	109.5
C21—C5—C6	109.6 (2)	H18B—C18—H18C	109.5
C20—C5—C6	111.5 (2)	C5—C20—H20A	109.5
C7—C6—C5	111.5 (2)	C5—C20—H20B	109.5
C7—C6—H6A	109.3	H20A—C20—H20B	109.5
C5—C6—H6A	109.3	C5—C20—H20C	109.5
C7—C6—H6B	109.3	H20A—C20—H20C	109.5
C5—C6—H6B	109.3	H20B—C20—H20C	109.5
H6A—C6—H6B	108.0	C5—C21—H21A	109.5
C2—C7—O8	123.0 (2)	C5—C21—H21B	109.5
C2—C7—C6	125.6 (2)	H21A—C21—H21B	109.5
O8—C7—C6	111.4 (2)	C5—C21—H21C	109.5
C7—O8—C9	117.1 (2)	H21A—C21—H21C	109.5
C14—C9—O8	122.5 (2)	H21B—C21—H21C	109.5
C14—C9—C10	125.8 (2)	C11—C22—H22A	109.5

O8—C9—C10	111.7 (2)	C11—C22—H22B	109.5
C9—C10—C11	111.8 (2)	H22A—C22—H22B	109.5
C9—C10—H10A	109.2	C11—C22—H22C	109.5
C11—C10—H10A	109.2	H22A—C22—H22C	109.5
C9—C10—H10B	109.2	H22B—C22—H22C	109.5
C11—C10—H10B	109.2	C11—C23—H23A	109.5
H10A—C10—H10B	107.9	C11—C23—H23B	109.5
C23—C11—C12	110.2 (2)	H23A—C23—H23B	109.5
C23—C11—C22	108.8 (2)	C11—C23—H23C	109.5
C12—C11—C22	109.9 (2)	H23A—C23—H23C	109.5
C23—C11—C10	109.5 (2)	H23B—C23—H23C	109.5
C12—C11—C10	108.1 (2)		

C14—C1—C2—C7	−25.2 (3)	O8—C9—C10—C11	159.3 (2)
C15—C1—C2—C7	95.7 (3)	C9—C10—C11—C23	167.9 (2)
C14—C1—C2—C3	151.0 (2)	C9—C10—C11—C12	47.7 (3)
C15—C1—C2—C3	−88.1 (3)	C9—C10—C11—C22	−72.5 (3)
C7—C2—C3—O19	170.0 (2)	C23—C11—C12—C13	−176.3 (2)
C1—C2—C3—O19	−6.3 (4)	C22—C11—C12—C13	63.7 (3)
C7—C2—C3—C4	−5.4 (4)	C10—C11—C12—C13	−56.7 (3)
C1—C2—C3—C4	178.2 (2)	C11—C12—C13—O24	−144.6 (3)
O19—C3—C4—C5	156.8 (2)	C11—C12—C13—C14	36.9 (3)
C2—C3—C4—C5	−27.7 (3)	O8—C9—C14—C13	178.5 (2)
C3—C4—C5—C21	173.5 (2)	C10—C9—C14—C13	−1.7 (4)
C3—C4—C5—C20	−67.3 (3)	O8—C9—C14—C1	−5.9 (4)
C3—C4—C5—C6	54.3 (3)	C10—C9—C14—C1	173.9 (2)
C4—C5—C6—C7	−49.8 (3)	O24—C13—C14—C9	174.9 (3)
C21—C5—C6—C7	−168.9 (2)	C12—C13—C14—C9	−6.5 (4)
C20—C5—C6—C7	70.8 (3)	O24—C13—C14—C1	−0.8 (4)
C3—C2—C7—O8	−168.6 (2)	C12—C13—C14—C1	177.8 (2)
C1—C2—C7—O8	7.7 (4)	C2—C1—C14—C9	24.4 (3)
C3—C2—C7—C6	9.2 (4)	C15—C1—C14—C9	−96.7 (3)
C1—C2—C7—C6	−174.5 (2)	C2—C1—C14—C13	−160.0 (2)
C5—C6—C7—C2	20.1 (4)	C15—C1—C14—C13	78.9 (3)
C5—C6—C7—O8	−161.9 (2)	C14—C1—C15—O16	7.8 (4)
C2—C7—O8—C9	13.7 (3)	C2—C1—C15—O16	−112.2 (3)
C6—C7—O8—C9	−164.4 (2)	C14—C1—C15—O17	−173.5 (2)
C7—O8—C9—C14	−14.7 (4)	C2—C1—C15—O17	66.4 (3)
C7—O8—C9—C10	165.5 (2)	O16—C15—O17—C18	1.4 (5)
C14—C9—C10—C11	−20.5 (4)	C1—C15—O17—C18	−177.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6*B*···O24^{i}	0.99	2.50	3.324 (4)	140

C18—H18*A*···O17^{ii}	0.98	2.53	3.451 (5)	156
C18—H18*A*···O19^{ii}	0.98	2.41	3.093 (4)	126

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