Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-Benzylidene-8-methoxy-6-(prop-1-enyl)chroman-4-one

R. Suresh,[a] Charles C. Kanagam,[b] P. R. Umarani,[c] V. Manivannan[c]* and Orhan Büyükgüngör[d]

[a]Department of Chemistry, Presidency College, Chennai 600 005, India, [b]Department of Chemistry, Valliammai Engineering College, Kattankulathur, Chennai, India, [c]Department of Physics, Presidency College, Chennai 600 005, India, and [d]Department of Physics, Ondokuz Mayis University, TR-55139 Samsun, Turkey
Correspondence e-mail: manivan_1999@yahoo.com

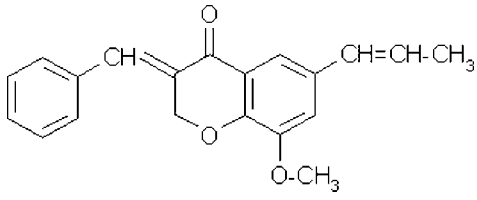
Received 16 October 2007; accepted 17 October 2007

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.005 Å; R factor = 0.068; wR factor = 0.183; data-to-parameter ratio = 15.1.

In the title compound, $C_{20}H_{18}O_3$, the phenyl ring makes a dihedral angle of 50.41 (10)° with the benzene ring of the chromanone unit. The molecular structure is stabilized by weak intramolecular C—H···O interactions and the crystal packing is stabilized by weak intermolecular C—H···O interactions.

Related literature

For related literature, see: Puviarasan *et al.* (1998); Tillekeratne *et al.* (2001); Nissa *et al.* (2001); Jae Gon Kang *et al.* (2004); Wu *et al.* (2005*a*,*b*); Schollmeyer *et al.* (2005).



Experimental

Crystal data

$C_{20}H_{18}O_3$
M_r = 306.34
Monoclinic, $P2_1/c$
a = 10.0763 (8) Å
b = 6.9018 (7) Å
c = 22.1841 (18) Å
β = 98.690 (7)°
V = 1525.1 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 150 (2) K
0.18 × 0.11 × 0.06 mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.984, T_{max} = 0.995
11793 measured reflections
3177 independent reflections
1494 reflections with $I > 2\sigma(I)$
R_{int} = 0.133

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.183
S = 0.92
3177 reflections
210 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O2	0.95	2.46	2.817 (4)	102
C9—H9B···O2^{i}	0.99	2.57	3.345 (4)	135
C20—H20A···O1^{ii}	0.98	2.51	3.418 (4)	154

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayis University, Turkey, for the use of the Stoe IPDSII diffractometer (purchased under grant F.279 of the University Research Fund).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2548).

References

Kang, J. G., Shin, S. Y., Kim, M. J., Bajpai, V., Maheshwari, D. K. & Kang, S. C. (2004). *J. Antibiot.* **57**, 726–731.
Nissa, M. N., Rajakannan, V., Kim, M.-J. & Velmurugan, D. (2001). *Acta Cryst.* E**57**, o1230–o1232.
Puviarasan, K., Govindasamy, L., Velmurugan, D., Shanmuga Sundara Raj, S., Shanmuga Sundaram, M., Raghunathan, R. & Fun, H.-K. (1998). *Acta Cryst.* C**54**, 961–963.
Schollmeyer, D., Kammerer, B., Peifer, C. & Laufer, S. (2005). *Acta Cryst.* E**61**, o868–o869.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.
Tillekeratne, L. M., Sherette, A., Grossman, P., Hupe, L., Hupe, D. & Hudson, R. A. (2001). *Bioorg. Med. Chem. Lett.* **11**, 2763–2764.
Wu, H., Xu, Z., Wan, Y., Liang, Y.-M. & Yu, K.-B. (2005*a*). *Acta Cryst.* E**61**, o1692–o1693.
Wu, H., Xu, Z., Zhou, J. & Liang, Y.-M. (2005*b*). *Acta Cryst.* E**61**, o1095–o1096.

 doi:10.1107/S1600536807051161

supplementary materials

Acta Cryst. (2007). E**63**, o4387 [**doi:10.1107/S1600536807051161**]

3-Benzylidene-8-methoxy-6-(prop-1-enyl)chroman-4-one

R. Suresh, C. C. Kanagam, P. R. Umarani, V. Manivannan and O. Büyükgüngör

Comment

The chromanone moiety present in the title compound consisting of the pyrone ring and benzene ring plays an important role in many areas of medicines such as inhibition of HIV replication (Tillekeratne *et al.*, 2001). The naturally occurring classes of compounds to which they belong, the benzylidene chroman-4-ones have been identified as a potential source of new anti-fungal agents (Jae Gon Kang *et al.*, 2004).

The geometric parameters in the title compound agree with the reported values of similar structures (Puviarasan *et al.*, 1998; Wu *et al.*, 2005*a*,b; Schollmeyer *et al.*, 2005; Nissa *et al.*, 2001). The chromanone moiety consists of one benzene ring fused with a six membered heterocyclic ring, which adopts a half-chair conformation. The phenyl ring makes a dihedral angle of 50.41 (10) ° with the benzene ring of chromanone unit.

The molecular structure is stabilized by weak intramolecular C—H···O interactions and the crystal packing is stabilized by weak intermolecular C—H···O interactions.

Experimental

10 mmol of Methyl-2-bromomethyl-3-phenyl-propenoate was treated with 10 mmol of eugenol in the presence of potassium carbonate in acetone at reflux temperature for 3 hrs. The pure ester of methyl- 3-phenyl-2-(2-methoxy-4- prop-2-enyl)phenoxy methyl-prop- 2enoate was obtained after silica gel column chromatography (3% EtOAc- hexane). Hydrolysis of this ester was carried out with KOH in aquous 1,4 –Dioxane at room temperature. The reaction mixture was acidified and the precipitated acid was purified by recrystalization. Finally the acid was treated with TFAA and the reaction mixture refluxed in dichloro- methane for 1 hr. It was further purified by column chromatography (silica gel-3% EtOAc- hexane).

Refinement

H atoms were positioned geometrically and refined using riding model with C—H = 0.95Å and U_{iso}(H) = 1.2Ueq(C) for aromatic C—H, C—H = 0.99Å and U_{iso}(H) = 1.2Ueq(C) for CH_2, C—H = 0.98Å and U_{iso}(H) = 1.5Ueq(C) for CH_3.

Figures

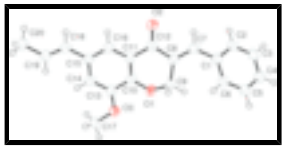


Fig. 1. The molecular structure of the title compound with atom labels and 50% probability displacement ellipsoids for non-H atoms.

3-Benzylidene-8-methoxy-6-(prop-1-enyl)chroman-4-one

Crystal data

$C_{20}H_{18}O_3$	F_{000} = 648
M_r = 306.34	D_x = 1.334 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 10.0763 (8) Å	Cell parameters from 9395 reflections
b = 6.9018 (7) Å	θ = 1.9–27.2º
c = 22.1841 (18) Å	μ = 0.09 mm^{-1}
β = 98.690 (7)º	T = 150 (2) K
V = 1525.1 (2) Å^3	Prism, yellow
Z = 4	0.18 × 0.11 × 0.06 mm

Data collection

Stoe IPDS2 diffractometer	3177 independent reflections
Radiation source: fine-focus sealed tube	1494 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.133
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 27.2º
T = 150(2) K	θ_{min} = 1.9º
rotation method scans	h = −12→12
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	k = −8→8
T_{min} = 0.984, T_{max} = 0.995	l = −28→28
11793 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.068	H-atom parameters constrained
$wR(F^2)$ = 0.183	$w = 1/[\sigma^2(F_o^2) + (0.0793P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.92	$(\Delta/\sigma)_{max}$ = 0.002
3177 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
210 parameters	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.1367 (2)	0.6980 (3)	0.13018 (11)	0.0438 (6)
O2	−0.0608 (2)	0.6743 (4)	0.27802 (11)	0.0512 (7)
C10	0.0003 (3)	0.7017 (5)	0.12271 (16)	0.0412 (8)
C11	−0.0732 (3)	0.6822 (4)	0.17019 (15)	0.0394 (8)
O3	0.0178 (2)	0.7463 (4)	0.01969 (11)	0.0485 (6)
C8	0.1466 (3)	0.6369 (5)	0.23959 (16)	0.0409 (8)
C15	−0.2804 (3)	0.7204 (5)	0.09918 (16)	0.0406 (8)
C1	0.3676 (3)	0.6343 (5)	0.31085 (16)	0.0405 (8)
C16	−0.2138 (3)	0.6905 (5)	0.15790 (17)	0.0430 (8)
H16	−0.2644	0.6754	0.1904	0.052*
C18	−0.4281 (3)	0.7281 (5)	0.08759 (17)	0.0450 (8)
H18	−0.4728	0.7173	0.1222	0.054*
C6	0.4472 (3)	0.5143 (5)	0.28020 (16)	0.0437 (8)
H6	0.4056	0.4332	0.2482	0.052*
C12	−0.0010 (3)	0.6639 (5)	0.23362 (16)	0.0415 (8)
C5	0.5854 (4)	0.5116 (5)	0.29567 (16)	0.0442 (8)
H5	0.6376	0.4298	0.2740	0.053*
C14	−0.2031 (3)	0.7411 (5)	0.05181 (16)	0.0429 (8)
H14	−0.2471	0.7620	0.0114	0.052*
C13	−0.0650 (3)	0.7318 (5)	0.06266 (16)	0.0426 (8)
C7	0.2216 (3)	0.6554 (4)	0.29504 (17)	0.0420 (8)
H7	0.1743	0.6858	0.3278	0.050*
C9	0.1985 (3)	0.5868 (5)	0.18191 (16)	0.0422 (8)
H9A	0.2966	0.6088	0.1877	0.051*
H9B	0.1825	0.4473	0.1732	0.051*
C19	−0.5049 (4)	0.7483 (5)	0.03451 (17)	0.0494 (9)
H19	−0.4607	0.7596	−0.0002	0.059*
C4	0.6485 (4)	0.6273 (5)	0.34257 (16)	0.0452 (8)
H4	0.7434	0.6254	0.3530	0.054*
C2	0.4336 (4)	0.7471 (5)	0.35922 (16)	0.0443 (8)
H2	0.3823	0.8258	0.3822	0.053*
C3	0.5720 (4)	0.7448 (5)	0.37377 (16)	0.0471 (8)
H3	0.6147	0.8253	0.4057	0.057*

C20	−0.6550 (3)	0.7552 (6)	0.02331 (18)	0.0536 (10)
H20A	−0.6891	0.7538	0.0624	0.080*
H20B	−0.6897	0.6422	−0.0009	0.080*
H20C	−0.6846	0.8739	0.0010	0.080*
C17	−0.0458 (4)	0.7642 (6)	−0.04259 (16)	0.0532 (9)
H17A	−0.1030	0.6511	−0.0537	0.080*
H17B	0.0231	0.7718	−0.0694	0.080*
H17C	−0.1007	0.8820	−0.0472	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0293 (12)	0.0611 (15)	0.0418 (14)	0.0009 (10)	0.0082 (11)	0.0041 (10)
O2	0.0401 (15)	0.0729 (17)	0.0420 (15)	0.0049 (11)	0.0110 (13)	−0.0008 (12)
C10	0.0328 (18)	0.0476 (18)	0.044 (2)	0.0005 (13)	0.0081 (17)	−0.0013 (14)
C11	0.0348 (19)	0.0438 (18)	0.040 (2)	0.0007 (13)	0.0063 (16)	−0.0003 (14)
O3	0.0370 (14)	0.0714 (16)	0.0381 (13)	−0.0002 (11)	0.0089 (12)	0.0027 (11)
C8	0.0345 (19)	0.0481 (19)	0.040 (2)	0.0010 (13)	0.0075 (18)	0.0003 (15)
C15	0.0338 (18)	0.0448 (18)	0.044 (2)	0.0004 (13)	0.0101 (17)	−0.0032 (14)
C1	0.0374 (19)	0.0442 (18)	0.040 (2)	0.0014 (13)	0.0058 (17)	0.0038 (14)
C16	0.0355 (19)	0.0499 (19)	0.045 (2)	0.0003 (14)	0.0096 (17)	0.0009 (15)
C18	0.0358 (19)	0.053 (2)	0.048 (2)	0.0018 (15)	0.0113 (18)	−0.0032 (16)
C6	0.040 (2)	0.0484 (19)	0.042 (2)	0.0030 (14)	0.0048 (18)	−0.0024 (15)
C12	0.039 (2)	0.0463 (18)	0.040 (2)	0.0016 (13)	0.0070 (17)	−0.0002 (14)
C5	0.043 (2)	0.0467 (19)	0.044 (2)	0.0053 (14)	0.0091 (18)	0.0028 (15)
C14	0.0374 (19)	0.0474 (18)	0.043 (2)	−0.0006 (14)	0.0037 (16)	−0.0028 (15)
C13	0.037 (2)	0.0496 (19)	0.042 (2)	−0.0019 (14)	0.0106 (17)	−0.0006 (15)
C7	0.038 (2)	0.0455 (19)	0.044 (2)	0.0007 (13)	0.0110 (18)	0.0000 (15)
C9	0.0341 (19)	0.0524 (19)	0.039 (2)	0.0045 (14)	0.0031 (17)	0.0002 (15)
C19	0.041 (2)	0.063 (2)	0.045 (2)	0.0059 (16)	0.0093 (19)	0.0038 (17)
C4	0.038 (2)	0.052 (2)	0.045 (2)	0.0002 (14)	0.0044 (18)	0.0045 (16)
C2	0.041 (2)	0.053 (2)	0.0392 (19)	0.0012 (15)	0.0072 (17)	−0.0018 (15)
C3	0.045 (2)	0.053 (2)	0.043 (2)	−0.0042 (15)	0.0046 (18)	−0.0019 (16)
C20	0.034 (2)	0.070 (2)	0.057 (2)	0.0005 (16)	0.0072 (19)	0.0014 (19)
C17	0.046 (2)	0.077 (3)	0.037 (2)	−0.0006 (18)	0.0081 (18)	0.0043 (18)

Geometric parameters (Å, °)

O1—C10	1.360 (4)	C6—H6	0.9500
O1—C9	1.441 (4)	C5—C4	1.387 (5)
O2—C12	1.232 (4)	C5—H5	0.9500
C10—C11	1.383 (4)	C14—C13	1.377 (5)
C10—C13	1.409 (5)	C14—H14	0.9500
C11—C16	1.403 (5)	C7—H7	0.9500
C11—C12	1.489 (5)	C9—H9A	0.9900
O3—C13	1.362 (4)	C9—H9B	0.9900
O3—C17	1.438 (4)	C19—C20	1.496 (5)
C8—C7	1.349 (5)	C19—H19	0.9500
C8—C12	1.485 (5)	C4—C3	1.375 (5)

C8—C9	1.494 (4)	C4—H4	0.9500
C15—C16	1.387 (5)	C2—C3	1.383 (5)
C15—C14	1.407 (4)	C2—H2	0.9500
C15—C18	1.473 (5)	C3—H3	0.9500
C1—C6	1.399 (4)	C20—H20A	0.9800
C1—C2	1.408 (5)	C20—H20B	0.9800
C1—C7	1.467 (5)	C20—H20C	0.9800
C16—H16	0.9500	C17—H17A	0.9800
C18—C19	1.315 (5)	C17—H17B	0.9800
C18—H18	0.9500	C17—H17C	0.9800
C6—C5	1.383 (5)		
C10—O1—C9	114.4 (2)	O3—C13—C10	115.1 (3)
O1—C10—C11	123.6 (3)	C14—C13—C10	119.2 (3)
O1—C10—C13	115.9 (3)	C8—C7—C1	127.8 (3)
C11—C10—C13	120.5 (3)	C8—C7—H7	116.1
C10—C11—C16	119.3 (3)	C1—C7—H7	116.1
C10—C11—C12	119.1 (3)	O1—C9—C8	113.2 (3)
C16—C11—C12	121.5 (3)	O1—C9—H9A	108.9
C13—O3—C17	116.6 (3)	C8—C9—H9A	108.9
C7—C8—C12	119.0 (3)	O1—C9—H9B	108.9
C7—C8—C9	125.6 (3)	C8—C9—H9B	108.9
C12—C8—C9	115.3 (3)	H9A—C9—H9B	107.8
C16—C15—C14	118.2 (3)	C18—C19—C20	126.6 (3)
C16—C15—C18	120.1 (3)	C18—C19—H19	116.7
C14—C15—C18	121.7 (3)	C20—C19—H19	116.7
C6—C1—C2	117.3 (3)	C3—C4—C5	119.3 (3)
C6—C1—C7	124.8 (3)	C3—C4—H4	120.4
C2—C1—C7	117.9 (3)	C5—C4—H4	120.4
C15—C16—C11	121.3 (3)	C3—C2—C1	121.0 (3)
C15—C16—H16	119.4	C3—C2—H2	119.5
C11—C16—H16	119.4	C1—C2—H2	119.5
C19—C18—C15	127.1 (3)	C4—C3—C2	120.7 (3)
C19—C18—H18	116.5	C4—C3—H3	119.6
C15—C18—H18	116.5	C2—C3—H3	119.6
C5—C6—C1	121.1 (3)	C19—C20—H20A	109.5
C5—C6—H6	119.4	C19—C20—H20B	109.5
C1—C6—H6	119.4	H20A—C20—H20B	109.5
O2—C12—C8	122.7 (3)	C19—C20—H20C	109.5
O2—C12—C11	121.5 (3)	H20A—C20—H20C	109.5
C8—C12—C11	115.8 (3)	H20B—C20—H20C	109.5
C6—C5—C4	120.5 (3)	O3—C17—H17A	109.5
C6—C5—H5	119.7	O3—C17—H17B	109.5
C4—C5—H5	119.7	H17A—C17—H17B	109.5
C13—C14—C15	121.6 (3)	O3—C17—H17C	109.5
C13—C14—H14	119.2	H17A—C17—H17C	109.5
C15—C14—H14	119.2	H17B—C17—H17C	109.5
O3—C13—C14	125.7 (3)		
C9—O1—C10—C11	27.8 (4)	C16—C15—C14—C13	0.2 (5)

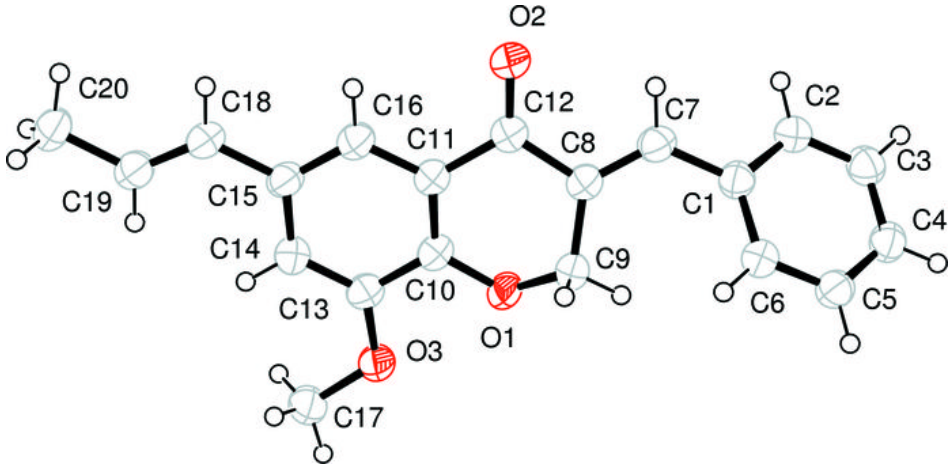
C9—O1—C10—C13	−153.7 (3)	C18—C15—C14—C13	−179.3 (3)
O1—C10—C11—C16	179.2 (3)	C17—O3—C13—C14	−2.9 (5)
C13—C10—C11—C16	0.7 (5)	C17—O3—C13—C10	176.0 (3)
O1—C10—C11—C12	2.6 (5)	C15—C14—C13—O3	178.6 (3)
C13—C10—C11—C12	−175.9 (3)	C15—C14—C13—C10	−0.3 (5)
C14—C15—C16—C11	0.4 (5)	O1—C10—C13—O3	2.3 (4)
C18—C15—C16—C11	179.9 (3)	C11—C10—C13—O3	−179.2 (3)
C10—C11—C16—C15	−0.8 (5)	O1—C10—C13—C14	−178.7 (3)
C12—C11—C16—C15	175.7 (3)	C11—C10—C13—C14	−0.2 (5)
C16—C15—C18—C19	−177.4 (3)	C12—C8—C7—C1	179.8 (3)
C14—C15—C18—C19	2.1 (5)	C9—C8—C7—C1	1.1 (5)
C2—C1—C6—C5	−2.0 (5)	C6—C1—C7—C8	−29.5 (5)
C7—C1—C6—C5	176.0 (3)	C2—C1—C7—C8	148.4 (3)
C7—C8—C12—O2	−11.0 (5)	C10—O1—C9—C8	−49.7 (4)
C9—C8—C12—O2	167.9 (3)	C7—C8—C9—O1	−138.7 (3)
C7—C8—C12—C11	167.7 (3)	C12—C8—C9—O1	42.5 (4)
C9—C8—C12—C11	−13.4 (4)	C15—C18—C19—C20	179.8 (4)
C10—C11—C12—O2	169.4 (3)	C6—C5—C4—C3	0.2 (5)
C16—C11—C12—O2	−7.1 (5)	C6—C1—C2—C3	2.8 (5)
C10—C11—C12—C8	−9.4 (4)	C7—C1—C2—C3	−175.4 (3)
C16—C11—C12—C8	174.1 (3)	C5—C4—C3—C2	0.6 (5)
C1—C6—C5—C4	0.6 (5)	C1—C2—C3—C4	−2.1 (5)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O2	0.95	2.46	2.817 (4)	102
C9—H9B···O2^{i}	0.99	2.57	3.345 (4)	135
C20—H20A···O1^{ii}	0.98	2.51	3.418 (4)	154

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

Fig. 1


O2
C20
C18
C16
C12
C7
C2
C11
C8
C3
C19
C15
C1
C4
C14
C9
C10
C13
C6
C5
O1
O3
C17