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3-Benzylidene-8-methoxy-6-methylchroman-4-one

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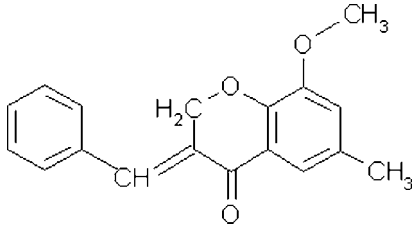
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.099; data-to-parameter ratio = 18.5.

In the title compound, $C_{18}H_{16}O_3$, the phenyl ring makes a dihedral angle of 59.67 (4)° with the benzene ring of the chromanone unit. The molecular structure is stabilized by a weak intramolecular C—H···O interaction and the crystal packing is stabilized by weak intermolecular C—H···O and C—H···π interactions.

Related literature

For related literature, see: Puviarasan *et al.* (1998); Tillekeratne *et al.* (2001); Nissa *et al.* (2001); Kang *et al.* (2004); Wu, Xu, Zhou *et al.* (2005); Wu, Xu, Wan *et al.* (2005); Schollmeyer *et al.* (2005). A similar methoxychroman-4-one compound has been reported recently (Suresh *et al.*, 2007).



Experimental

Crystal data

$C_{18}H_{16}O_3$
M_r = 280.31
Monoclinic, $P2_1/a$
a = 13.0081 (9) Å
b = 8.1407 (5) Å
c = 13.7580 (10) Å
β = 107.522 (5)°
V = 1389.31 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 150 (2) K
0.44 × 0.29 × 0.06 mm

Data collection

Stoe IPDS2 diffractometer
Absorption correction: integration (*X-RED*; Stoe & Cie, 2002)
T_{min} = 0.988, T_{max} = 0.991
15939 measured reflections
3551 independent reflections
2574 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.099
S = 1.03
3551 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the benzene C10/C11/C15–C17/C13 ring and the phenyl C1–C6 ring, respectively.

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O2	0.95	2.43	2.7922 (15)	103
C18—H18*C*···O2^{i}	0.98	2.52	3.3937 (16)	148
C9—H9*B*···*Cg*1^{ii}	0.99	2.99	3.7499 (14)	135
C14—H14*C*···*Cg*2^{iii}	0.98	2.91	3.8592 (15)	162

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2223).

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supplementary materials

Acta Cryst. (2007). E**63**, o4530 [**doi:10.1107/S1600536807053019**]

3-Benzylidene-8-methoxy-6-methylchroman-4-one

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Comment

The Chromanone moiety present in the title compound consisting of the pyrone and benzene rings 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 benzylidine chroman-4-ones, have been identified as a potential source of new anti-fungal agents (Kang *et al.*, 2004).

The geometric parameters in the compound, (I), agree with the reported values of similar structure (Puviarasan *et al.*, 1998; Wu, Xu, Zhou *et al.*, 2005; Wu, Xu, Wan *et al.*, 2005; Schollmeyer *et al.*, 2005; Nissa *et al.*, 2001). The phenyl ring makes a dihedral angle of 59.67 (4)° with the benzene ring of the chromanone unit.

The molecular structure is stabilized by a weak intramolecular C—H···O interaction and the crystal packing is stabilized by a weak intermolecular C—H···O hydrogen bond and C—H···π interactions involving the phenyl C1—C6 ring and the benzene C10/C11/C15—C17/C13 ring.

Experimental

Reaction was carried out in 10 mmol scale of bromomethylpropenoate with 2-methoxy-4-methyl phenol in the presence of potassium carbonate in acetone at reflux temperature for 3 h. Then the pure ester 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 precipitate was purified by recrystalization. Finally the acid was treated with TFAA and the reaction mixture refluxed in dichloromethane for 1 h. It was further purified by column chromatography (silica gel, 3% EtOAc- hexane).

Refinement

H atoms were positioned geometrically (C—H = 0.95 − 0.99 Å) and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Figures

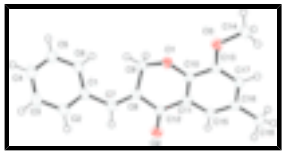


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

3-Benzylidene-8-methoxy-6-methylchroman-4-one

Crystal data

$C_{18}H_{16}O_3$	F_{000} = 592
M_r = 280.31	D_x = 1.340 Mg m^{-3}
Monoclinic, $P2_1/a$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yab	Cell parameters from 20682 reflections
a = 13.0081 (9) Å	θ = 1.6–28.8º
b = 8.1407 (5) Å	μ = 0.09 mm^{-1}
c = 13.7580 (10) Å	T = 150 (2) K
β = 107.522 (5)º	Plate, yellow
V = 1389.31 (16) Å^3	0.44 × 0.29 × 0.06 mm
Z = 4	

Data collection

Stoe IPDS2 diffractometer	3551 independent reflections
Radiation source: fine-focus sealed tube	2574 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.055
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 28.7º
T = 150(2) K	θ_{min} = 2.9º
ω scans	h = −17→17
Absorption correction: integration (*X-RED*; Stoe & Cie, 2002)	k = −10→10
T_{min} = 0.988, T_{max} = 0.991	l = −18→18
15939 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
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H-atom parameters constrained
$wR(F^2)$ = 0.099	$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2 + 0.065P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
3551 reflections	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
192 parameters	$\Delta\rho_{min}$ = −0.15 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	0.78602 (7)	0.02990 (10)	0.02502 (6)	0.0269 (2)
O3	0.75897 (7)	0.05559 (10)	−0.17011 (7)	0.0281 (2)
O2	1.02517 (7)	−0.25015 (11)	0.21383 (7)	0.0316 (2)
C17	0.89940 (10)	−0.14732 (14)	−0.16166 (9)	0.0259 (3)
H17	0.8907	−0.1371	−0.2325	0.031*
C11	0.92597 (9)	−0.17538 (14)	0.04513 (9)	0.0240 (2)
C13	0.83512 (10)	−0.05403 (14)	−0.11889 (9)	0.0243 (2)
C10	0.84867 (9)	−0.06858 (13)	−0.01381 (9)	0.0234 (2)
C8	0.87402 (10)	−0.07504 (14)	0.19619 (9)	0.0249 (2)
C14	0.73210 (12)	0.05403 (17)	−0.27867 (10)	0.0349 (3)
H14A	0.7934	0.0950	−0.2991	0.052*
H14B	0.6693	0.1246	−0.3078	0.052*
H14C	0.7152	−0.0585	−0.3036	0.052*
C16	0.97710 (10)	−0.25671 (14)	−0.10298 (10)	0.0263 (3)
C12	0.94866 (10)	−0.17583 (14)	0.15659 (9)	0.0249 (2)
C1	0.84397 (10)	0.05118 (15)	0.35372 (9)	0.0273 (3)
C7	0.90426 (10)	−0.03749 (15)	0.29547 (10)	0.0271 (3)
H7	0.9744	−0.0731	0.3335	0.033*
C15	0.98897 (10)	−0.27015 (14)	−0.00074 (10)	0.0262 (3)
H15	1.0406	−0.3447	0.0397	0.031*
C6	0.73213 (11)	0.03713 (16)	0.33350 (10)	0.0301 (3)
H6	0.6917	−0.0300	0.2788	0.036*
C2	0.90143 (12)	0.14728 (16)	0.43647 (10)	0.0332 (3)
H2	0.9776	0.1551	0.4527	0.040*
C9	0.77010 (10)	−0.02889 (16)	0.11825 (10)	0.0276 (3)
H9A	0.7220	−0.1259	0.1030	0.033*
H9B	0.7341	0.0576	0.1467	0.033*
C4	0.73819 (13)	0.21907 (18)	0.47312 (11)	0.0389 (3)
H4	0.7020	0.2778	0.5128	0.047*
C5	0.68008 (12)	0.12118 (18)	0.39318 (10)	0.0350 (3)
H5	0.6042	0.1113	0.3790	0.042*
C18	1.04741 (11)	−0.35413 (16)	−0.15150 (11)	0.0329 (3)
H18A	1.1090	−0.3991	−0.0982	0.049*

H18B	1.0734	−0.2823	−0.1962	0.049*
H18C	1.0054	−0.4443	−0.1916	0.049*
C3	0.84876 (13)	0.23136 (18)	0.49522 (11)	0.0393 (3)
H3	0.8888	0.2974	0.5507	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0305 (5)	0.0265 (4)	0.0239 (4)	0.0063 (3)	0.0085 (4)	0.0005 (3)
O3	0.0337 (5)	0.0253 (4)	0.0241 (4)	0.0048 (4)	0.0071 (4)	0.0018 (3)
O2	0.0315 (5)	0.0287 (4)	0.0308 (5)	0.0055 (4)	0.0038 (4)	0.0012 (4)
C17	0.0287 (6)	0.0230 (5)	0.0265 (6)	−0.0041 (5)	0.0090 (5)	−0.0040 (4)
C11	0.0241 (6)	0.0195 (5)	0.0273 (6)	−0.0023 (4)	0.0063 (5)	−0.0013 (4)
C13	0.0261 (6)	0.0189 (5)	0.0267 (6)	−0.0016 (4)	0.0062 (5)	−0.0004 (4)
C10	0.0247 (6)	0.0188 (5)	0.0269 (6)	−0.0017 (4)	0.0080 (5)	−0.0030 (4)
C8	0.0264 (6)	0.0208 (5)	0.0274 (6)	−0.0010 (4)	0.0080 (5)	0.0011 (4)
C14	0.0438 (8)	0.0331 (7)	0.0247 (7)	0.0067 (6)	0.0057 (6)	0.0003 (5)
C16	0.0261 (6)	0.0206 (5)	0.0329 (7)	−0.0036 (5)	0.0098 (5)	−0.0057 (5)
C12	0.0251 (6)	0.0205 (5)	0.0271 (6)	−0.0012 (4)	0.0048 (5)	0.0000 (4)
C1	0.0353 (7)	0.0238 (6)	0.0221 (6)	0.0022 (5)	0.0076 (5)	0.0037 (5)
C7	0.0289 (6)	0.0240 (6)	0.0269 (6)	−0.0001 (5)	0.0061 (5)	0.0017 (5)
C15	0.0240 (6)	0.0201 (5)	0.0329 (7)	−0.0004 (4)	0.0063 (5)	−0.0015 (5)
C6	0.0373 (7)	0.0289 (6)	0.0241 (6)	0.0014 (5)	0.0094 (5)	0.0038 (5)
C2	0.0377 (7)	0.0311 (7)	0.0275 (7)	0.0031 (5)	0.0048 (5)	0.0001 (5)
C9	0.0278 (6)	0.0303 (6)	0.0254 (6)	0.0030 (5)	0.0089 (5)	0.0012 (5)
C4	0.0536 (9)	0.0375 (7)	0.0278 (7)	0.0134 (6)	0.0155 (7)	0.0021 (6)
C5	0.0402 (8)	0.0383 (7)	0.0285 (7)	0.0076 (6)	0.0134 (6)	0.0069 (6)
C18	0.0351 (7)	0.0285 (6)	0.0371 (7)	0.0025 (5)	0.0139 (6)	−0.0052 (5)
C3	0.0529 (9)	0.0349 (7)	0.0256 (7)	0.0066 (6)	0.0052 (6)	−0.0048 (5)

Geometric parameters (Å, °)

O1—C10	1.3617 (14)	C1—C2	1.3981 (18)
O1—C9	1.4412 (15)	C1—C6	1.4012 (18)
O3—C13	1.3615 (14)	C1—C7	1.4685 (17)
O3—C14	1.4280 (15)	C7—H7	0.9500
O2—C12	1.2268 (15)	C15—H15	0.9500
C17—C13	1.3851 (16)	C6—C5	1.3913 (18)
C17—C16	1.4054 (18)	C6—H6	0.9500
C17—H17	0.9500	C2—C3	1.3873 (19)
C11—C10	1.3906 (16)	C2—H2	0.9500
C11—C15	1.4058 (16)	C9—H9A	0.9900
C11—C12	1.4718 (17)	C9—H9B	0.9900
C13—C10	1.4076 (17)	C4—C3	1.381 (2)
C8—C7	1.3381 (18)	C4—C5	1.384 (2)
C8—C12	1.4937 (16)	C4—H4	0.9500
C8—C9	1.4985 (18)	C5—H5	0.9500
C14—H14A	0.9800	C18—H18A	0.9800
C14—H14B	0.9800	C18—H18B	0.9800

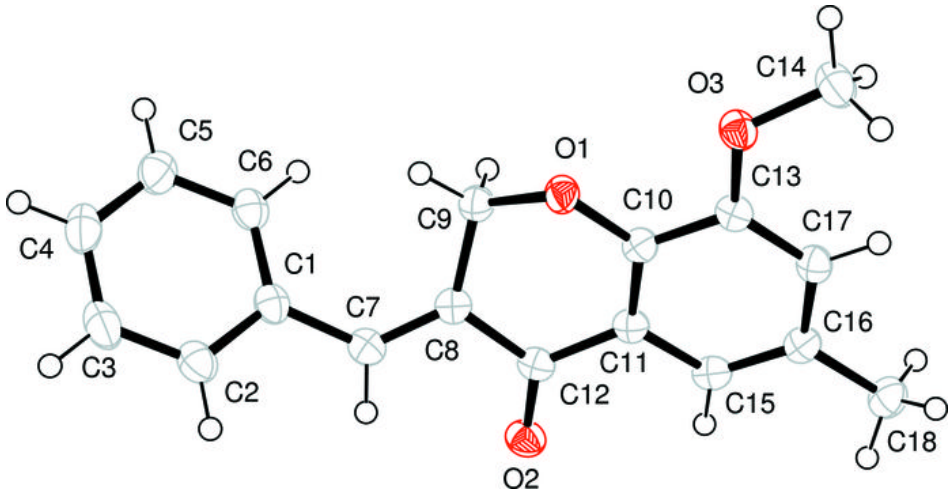
C14—H14C	0.9800	C18—H18C	0.9800
C16—C15	1.3724 (18)	C3—H3	0.9500
C16—C18	1.5095 (17)		
C10—O1—C9	114.64 (9)	C8—C7—H7	115.5
C13—O3—C14	116.51 (9)	C1—C7—H7	115.5
C13—C17—C16	121.68 (11)	C16—C15—C11	121.03 (11)
C13—C17—H17	119.2	C16—C15—H15	119.5
C16—C17—H17	119.2	C11—C15—H15	119.5
C10—C11—C15	119.89 (11)	C5—C6—C1	120.22 (13)
C10—C11—C12	119.48 (10)	C5—C6—H6	119.9
C15—C11—C12	120.35 (11)	C1—C6—H6	119.9
O3—C13—C17	125.28 (11)	C3—C2—C1	120.92 (13)
O3—C13—C10	115.68 (10)	C3—C2—H2	119.5
C17—C13—C10	119.03 (11)	C1—C2—H2	119.5
O1—C10—C11	123.53 (11)	O1—C9—C8	112.32 (10)
O1—C10—C13	116.66 (10)	O1—C9—H9A	109.1
C11—C10—C13	119.76 (10)	C8—C9—H9A	109.1
C7—C8—C12	118.31 (11)	O1—C9—H9B	109.1
C7—C8—C9	126.72 (11)	C8—C9—H9B	109.1
C12—C8—C9	114.97 (10)	H9A—C9—H9B	107.9
O3—C14—H14A	109.5	C3—C4—C5	120.00 (13)
O3—C14—H14B	109.5	C3—C4—H4	120.0
H14A—C14—H14B	109.5	C5—C4—H4	120.0
O3—C14—H14C	109.5	C4—C5—C6	120.39 (14)
H14A—C14—H14C	109.5	C4—C5—H5	119.8
H14B—C14—H14C	109.5	C6—C5—H5	119.8
C15—C16—C17	118.60 (11)	C16—C18—H18A	109.5
C15—C16—C18	121.01 (12)	C16—C18—H18B	109.5
C17—C16—C18	120.37 (11)	H18A—C18—H18B	109.5
O2—C12—C11	122.65 (11)	C16—C18—H18C	109.5
O2—C12—C8	121.84 (11)	H18A—C18—H18C	109.5
C11—C12—C8	115.48 (10)	H18B—C18—H18C	109.5
C2—C1—C6	118.42 (12)	C4—C3—C2	120.01 (13)
C2—C1—C7	118.49 (12)	C4—C3—H3	120.0
C6—C1—C7	123.00 (12)	C2—C3—H3	120.0
C8—C7—C1	128.91 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O2	0.95	2.43	2.7922 (15)	103
C18—H18C···O2^{i}	0.98	2.52	3.3937 (16)	148
C9—H9B···Cg1^{ii}	0.99	2.99	3.7499 (14)	135
C14—H14C···Cg2^{iii}	0.98	2.91	3.8592 (15)	162

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

Fig. 1


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