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3-Benzylidene-8-methoxy-6-(prop-2-enyl)chroman-4-one

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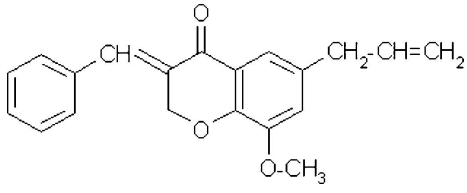
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.129; data-to-parameter ratio = 21.4.

In the title compound, $C_{20}H_{18}O_3$, the phenyl ring makes a dihedral angle of 39.97 (4)° with the benzene ring of the chromanone unit. The molecular structure and the crystal packing are stabilized by weak intra- and intermolecular C—H···O interactions.

Related literature

For related literature see: Tillekeratne *et al.* (2001); Nissa *et al.* (2001); Kang *et al.* (2004); Wu, Xu, Zhou & Liang (2005); Wu, Xu, Wan *et al.* (2005); Schollmeyer *et al.* (2005).



Experimental

Crystal data

$C_{20}H_{18}O_3$
M_r = 306.34
Monoclinic, $P2_1/n$
a = 8.9229 (5) Å
b = 14.0910 (6) Å
c = 12.3493 (6) Å
β = 92.930 (4)°
V = 1550.68 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 150 (2) K
0.50 × 0.42 × 0.36 mm

Data collection

Stoe IPDS2 diffractometer
Absorption correction: integration (*X-RED*; Stoe & Cie, 2002)
T_{min} = 0.958, T_{max} = 0.969
27302 measured reflections
4481 independent reflections
3495 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.129
S = 1.06
4481 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C4—H4···O2^{i}	0.93	2.58	3.4364 (16)	153
C7—H7···O2	0.93	2.40	2.7899 (14)	105

Symmetry code: (i) $x-\frac{1}{2}, -y-\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (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: IS2214).

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supplementary materials

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3-Benzylidene-8-methoxy-6-(prop-2-enyl)chroman-4-one

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Comment

The chromanone moiety present in the title compound consisting of the pyrone ring and benzene ring which 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 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 (Wu, Xu, Zhou & Liang, 2005; Wu, Xu, Wan *et al.*, 2005; Schollmeyer *et al.*, 2005; Nissa *et al.*, 2001). The phenyl ring makes a dihedral angle of 39.97 (4)° with the benzene ring of 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 interaction.

Experimental

Methyl-2-bromomethyl-3-phenyl-propenoate (10 mmol) was treated with eugenol (10 mmol) in the presence of potassium carbonate in acetone at reflux temperature for 3 h. The pure ester of methyl-3-phenyl-2-(2-methoxy-4-prop-2-enyl)phenoxy methyl-prop-2-noate was obtained after silica gel column chromatography (3% EtOAc–hexane). Hydrolysis of this ester was carried out with KOH in aqueous 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 dichloromethane for 1 h. 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 = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic C—H, C—H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for CH_2, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3.

Figures

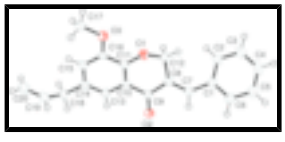


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

3-Benzylidene-8-methoxy-6-(prop-2-enyl)chroman-4-one

Crystal data

$C_{20}H_{18}O_3$ F_{000} = 648

M_r = 306.34	D_x = 1.312 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 31265 reflections
a = 8.9229 (5) Å	θ = 2.2–30.0º
b = 14.0910 (6) Å	μ = 0.09 mm^{-1}
c = 12.3493 (6) Å	T = 150 (2) K
β = 92.930 (4)º	Prism, yellow
V = 1550.68 (13) Å^3	0.50 × 0.42 × 0.36 mm
Z = 4	

Data collection

Stoe IPDS2 diffractometer	4481 independent reflections
Radiation source: fine-focus sealed tube	3495 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.057
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 30.0º
T = 150(2) K	θ_{min} = 2.2º
ω scans	h = −12→12
Absorption correction: integration (X-RED; Stoe & Cie, 2002)	k = −19→19
T_{min} = 0.958, T_{max} = 0.969	l = −17→17
27302 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
$R[F^2 > 2\sigma(F^2)]$ = 0.045	H-atom parameters constrained
$wR(F^2)$ = 0.129	$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2 + 0.1756P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
4481 reflections	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
209 parameters	$\Delta\rho_{min}$ = −0.27 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 calculat-

ing *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	−0.00323 (10)	0.10788 (6)	0.71245 (6)	0.0365 (2)
O2	0.19644 (10)	0.00693 (6)	0.99286 (7)	0.0379 (2)
O3	−0.01473 (11)	0.28696 (6)	0.66460 (7)	0.0391 (2)
C1	0.01490 (13)	−0.20158 (8)	0.77596 (9)	0.0320 (2)
C2	−0.11928 (14)	−0.19972 (9)	0.71231 (10)	0.0367 (2)
H2	−0.1786	−0.1454	0.7111	0.044*
C3	−0.16463 (15)	−0.27785 (10)	0.65112 (11)	0.0421 (3)
H3	−0.2531	−0.2751	0.6080	0.051*
C4	−0.07937 (16)	−0.36015 (9)	0.65349 (11)	0.0428 (3)
H4	−0.1094	−0.4120	0.6112	0.051*
C5	0.05080 (15)	−0.36449 (9)	0.71928 (12)	0.0424 (3)
H5	0.1074	−0.4199	0.7226	0.051*
C6	0.09676 (14)	−0.28618 (8)	0.78026 (10)	0.0364 (2)
H6	0.1837	−0.2900	0.8249	0.044*
C7	0.07444 (13)	−0.12215 (8)	0.84140 (9)	0.0319 (2)
H7	0.1408	−0.1392	0.8986	0.038*
C8	0.04848 (12)	−0.02850 (8)	0.83192 (9)	0.0306 (2)
C9	0.12098 (12)	0.03627 (8)	0.91420 (9)	0.0302 (2)
C10	0.09940 (12)	0.13854 (8)	0.89403 (9)	0.0293 (2)
C11	0.04207 (12)	0.16868 (8)	0.79283 (8)	0.0303 (2)
C12	−0.05548 (14)	0.01735 (8)	0.74838 (9)	0.0341 (2)
H12A	−0.0681	−0.0246	0.6863	0.041*
H12B	−0.1530	0.0256	0.7784	0.041*
C13	0.14881 (13)	0.20629 (8)	0.97125 (9)	0.0315 (2)
H13	0.1874	0.1863	1.0388	0.038*
C14	0.14090 (13)	0.30157 (8)	0.94844 (9)	0.0333 (2)
C15	0.08513 (13)	0.33094 (8)	0.84531 (9)	0.0334 (2)
H15	0.0805	0.3954	0.8292	0.040*
C16	0.03716 (13)	0.26584 (8)	0.76749 (9)	0.0317 (2)
C17	−0.01812 (15)	0.38441 (9)	0.63519 (10)	0.0392 (3)
H17A	−0.0826	0.4183	0.6816	0.059*
H17B	−0.0555	0.3906	0.5612	0.059*
H17C	0.0814	0.4102	0.6428	0.059*
C18	0.19189 (16)	0.37561 (9)	1.03162 (10)	0.0399 (3)
H18A	0.1044	0.4058	1.0600	0.048*
H18B	0.2466	0.3445	1.0915	0.048*
C19	0.28911 (15)	0.44939 (10)	0.98556 (11)	0.0423 (3)
H19	0.3776	0.4293	0.9561	0.051*
C20	0.25975 (18)	0.54132 (10)	0.98312 (12)	0.0499 (3)
H20A	0.1724	0.5642	1.0117	0.060*
H20B	0.3265	0.5831	0.9528	0.060*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0521 (5)	0.0291 (4)	0.0270 (4)	−0.0020 (3)	−0.0094 (3)	−0.0009 (3)
O2	0.0440 (5)	0.0327 (4)	0.0355 (4)	0.0021 (3)	−0.0134 (3)	0.0024 (3)
O3	0.0548 (5)	0.0308 (4)	0.0300 (4)	−0.0029 (3)	−0.0132 (4)	0.0046 (3)
C1	0.0350 (5)	0.0296 (5)	0.0315 (5)	−0.0022 (4)	0.0018 (4)	−0.0003 (4)
C2	0.0376 (6)	0.0329 (5)	0.0393 (6)	−0.0017 (4)	−0.0030 (5)	−0.0009 (4)
C3	0.0447 (7)	0.0422 (6)	0.0387 (6)	−0.0107 (5)	−0.0037 (5)	−0.0025 (5)
C4	0.0521 (7)	0.0367 (6)	0.0402 (6)	−0.0114 (5)	0.0080 (5)	−0.0089 (5)
C5	0.0449 (6)	0.0305 (6)	0.0526 (7)	−0.0021 (5)	0.0100 (6)	−0.0058 (5)
C6	0.0360 (6)	0.0305 (5)	0.0429 (6)	−0.0007 (4)	0.0028 (5)	−0.0007 (4)
C7	0.0326 (5)	0.0308 (5)	0.0316 (5)	−0.0003 (4)	−0.0030 (4)	−0.0004 (4)
C8	0.0328 (5)	0.0302 (5)	0.0283 (5)	0.0004 (4)	−0.0030 (4)	−0.0012 (4)
C9	0.0318 (5)	0.0300 (5)	0.0282 (5)	0.0009 (4)	−0.0038 (4)	0.0004 (4)
C10	0.0315 (5)	0.0291 (5)	0.0269 (5)	0.0004 (4)	−0.0032 (4)	−0.0003 (4)
C11	0.0332 (5)	0.0305 (5)	0.0264 (5)	0.0000 (4)	−0.0042 (4)	−0.0019 (4)
C12	0.0412 (6)	0.0284 (5)	0.0316 (5)	−0.0010 (4)	−0.0081 (4)	−0.0004 (4)
C13	0.0363 (5)	0.0314 (5)	0.0262 (5)	−0.0004 (4)	−0.0051 (4)	0.0002 (4)
C14	0.0382 (6)	0.0320 (5)	0.0292 (5)	−0.0022 (4)	−0.0045 (4)	−0.0019 (4)
C15	0.0391 (6)	0.0288 (5)	0.0319 (5)	−0.0007 (4)	−0.0043 (4)	0.0004 (4)
C16	0.0344 (5)	0.0327 (5)	0.0273 (5)	0.0005 (4)	−0.0051 (4)	0.0021 (4)
C17	0.0461 (6)	0.0330 (6)	0.0373 (6)	−0.0018 (5)	−0.0088 (5)	0.0083 (4)
C18	0.0530 (7)	0.0343 (6)	0.0316 (6)	−0.0043 (5)	−0.0068 (5)	−0.0029 (4)
C19	0.0424 (6)	0.0452 (7)	0.0384 (6)	−0.0069 (5)	−0.0058 (5)	−0.0043 (5)
C20	0.0605 (8)	0.0417 (7)	0.0456 (7)	−0.0116 (6)	−0.0149 (6)	0.0064 (6)

Geometric parameters (Å, °)

O1—C11	1.3570 (13)	C18—C14	1.5176 (16)
O1—C12	1.4362 (14)	C18—H18A	0.9700
O3—C16	1.3628 (13)	C18—H18B	0.9700
O3—C17	1.4204 (14)	C6—C5	1.3860 (17)
O2—C9	1.2252 (13)	C6—H6	0.9300
C10—C11	1.3923 (15)	C17—H17A	0.9600
C10—C13	1.4044 (15)	C17—H17B	0.9600
C10—C9	1.4734 (15)	C17—H17C	0.9600
C9—C8	1.4889 (15)	C12—H12A	0.9700
C8—C7	1.3439 (15)	C12—H12B	0.9700
C8—C12	1.4981 (15)	C19—C20	1.322 (2)
C13—C14	1.3728 (16)	C19—H19	0.9300
C13—H13	0.9300	C2—C3	1.3839 (17)
C15—C16	1.3806 (15)	C2—H2	0.9300
C15—C14	1.4057 (15)	C3—C4	1.386 (2)
C15—H15	0.9300	C3—H3	0.9300
C1—C6	1.3977 (16)	C5—C4	1.384 (2)
C1—C2	1.3985 (17)	C5—H5	0.9300
C1—C7	1.4643 (15)	C4—H4	0.9300

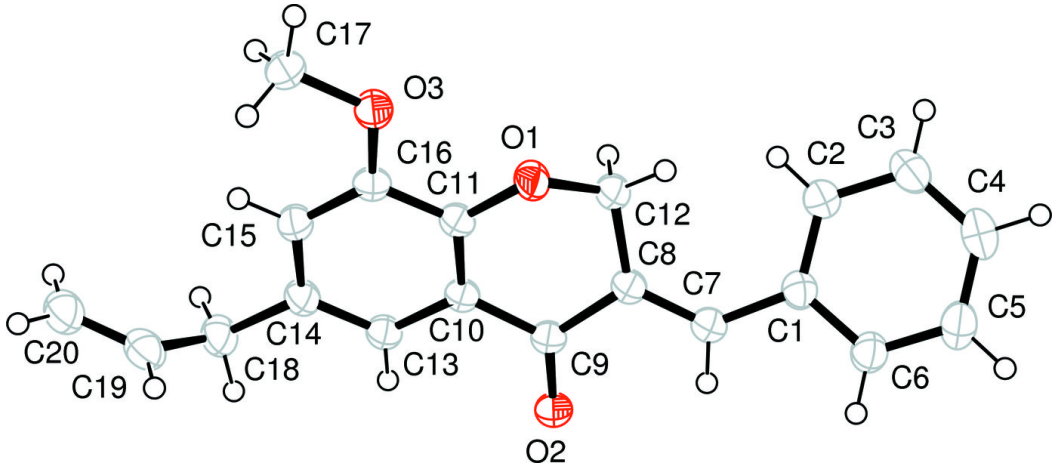
C11—C16	1.4047 (15)	C20—H20A	0.9300
C7—H7	0.9300	C20—H20B	0.9300
C18—C19	1.4858 (19)		
C11—O1—C12	115.08 (8)	C5—C6—H6	119.4
C16—O3—C17	116.83 (9)	C1—C6—H6	119.4
C11—C10—C13	119.36 (10)	O3—C17—H17A	109.5
C11—C10—C9	119.27 (10)	O3—C17—H17B	109.5
C13—C10—C9	121.08 (10)	H17A—C17—H17B	109.5
O2—C9—C10	121.73 (10)	O3—C17—H17C	109.5
O2—C9—C8	122.42 (10)	H17A—C17—H17C	109.5
C10—C9—C8	115.83 (9)	H17B—C17—H17C	109.5
C7—C8—C9	118.40 (10)	O1—C12—C8	113.36 (9)
C7—C8—C12	125.55 (10)	O1—C12—H12A	108.9
C9—C8—C12	115.95 (9)	C8—C12—H12A	108.9
C14—C13—C10	120.96 (10)	O1—C12—H12B	108.9
C14—C13—H13	119.5	C8—C12—H12B	108.9
C10—C13—H13	119.5	H12A—C12—H12B	107.7
C16—C15—C14	121.19 (10)	C13—C14—C15	119.07 (10)
C16—C15—H15	119.4	C13—C14—C18	121.53 (10)
C14—C15—H15	119.4	C15—C14—C18	119.40 (10)
C6—C1—C2	117.91 (11)	C20—C19—C18	125.15 (14)
C6—C1—C7	117.18 (10)	C20—C19—H19	117.4
C2—C1—C7	124.89 (11)	C18—C19—H19	117.4
O1—C11—C10	123.10 (10)	C3—C2—C1	120.66 (12)
O1—C11—C16	116.60 (9)	C3—C2—H2	119.7
C10—C11—C16	120.20 (10)	C1—C2—H2	119.7
O3—C16—C15	125.62 (10)	C2—C3—C4	120.60 (12)
O3—C16—C11	115.20 (10)	C2—C3—H3	119.7
C15—C16—C11	119.18 (10)	C4—C3—H3	119.7
C8—C7—C1	130.27 (11)	C4—C5—C6	119.98 (12)
C8—C7—H7	114.9	C4—C5—H5	120.0
C1—C7—H7	114.9	C6—C5—H5	120.0
C19—C18—C14	112.49 (11)	C5—C4—C3	119.50 (12)
C19—C18—H18A	109.1	C5—C4—H4	120.3
C14—C18—H18A	109.1	C3—C4—H4	120.3
C19—C18—H18B	109.1	C19—C20—H20A	120.0
C14—C18—H18B	109.1	C19—C20—H20B	120.0
H18A—C18—H18B	107.8	H20A—C20—H20B	120.0
C5—C6—C1	121.25 (12)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C4—H4···O2^{i}	0.93	2.58	3.4364 (16)	153
C7—H7···O2	0.93	2.40	2.7899 (14)	105

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

Fig. 1


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