organic compounds

3-Benzylidene-6-methoxychroman-4-one

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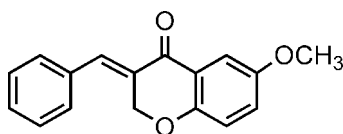
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In the title compound, $C_{17}H_{14}O_3$, the dihedral angle between the phenyl ring and the benzene ring of the chromanone moiety is 67.78 (3)°. The six-membered heterocyclic ring of the chromanone moiety adopts a half-chair conformation. The structure is stabilized by weak intermolecular C—H···O interactions that link the molecules into inversion dimers.

Related literature

For background literature, see: Finch & Tamm (1970); Geen *et al.* (1996); Tietze & Gerlitzer (1997); Cremer & Pople (1975). For a related structure, see: Suresh *et al.* (2007).

Experimental

Crystal data

$C_{17}H_{14}O_3$	$a = 7.2678$ (2) Å
$M_r = 266.28$	$b = 8.3151$ (2) Å
Triclinic, $P\bar{1}$	$c = 11.7999$ (4) Å
$\alpha = 95.964$ (1)°	Mo $K\alpha$ radiation
$\beta = 103.828$ (1)°	$\mu = 0.09$ mm^{-1}
$\gamma = 104.042$ (1)°	$T = 298$ (2) K
$V = 661.74$ (3) Å^3	$0.45 \times 0.42 \times 0.38$ mm
$Z = 2$	

Data collection

Bruker APEXII CCD area-detector diffractometer	8868 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)	3041 independent reflections
$T_{min} = 0.960$, $T_{max} = 0.966$	2404 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.116$	
$S = 1.04$	
3041 reflections	$\Delta\rho_{max} = 0.24$ e Å^{-3}
186 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O1^i	0.93	2.53	3.4293 (18)	163

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* (Bruker, 2004); data reduction: *SAINT-Plus* (Bruker, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2222).

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supporting information

3-Benzylidene-6-methoxychroman-4-one

T. Augustine, Scholastica Mary Vithiya, V. Ramkumar and Charles C. Kanakam

S1. Comment

The chroman-4-one (2,3-dihydro-4-oxo-4*H*-1-benzopyran) ring system occupies an important position among oxygen heterocyclics and features in a wide variety of compounds of biological and medicinal interest (Finch & Tamm, 1970). Many biologically active natural products containing a chroman ring system have been synthesized *via* 2-substituted chroman-4-one intermediates including alpha-tocopherol (vitamin E) (Geen *et al.*, 1996). 3-arylidene-4-chromanones have also been isolated as natural products belonging to the class of compounds called homoisoflavonoids(Tietze & Gerlitzer, 1997).
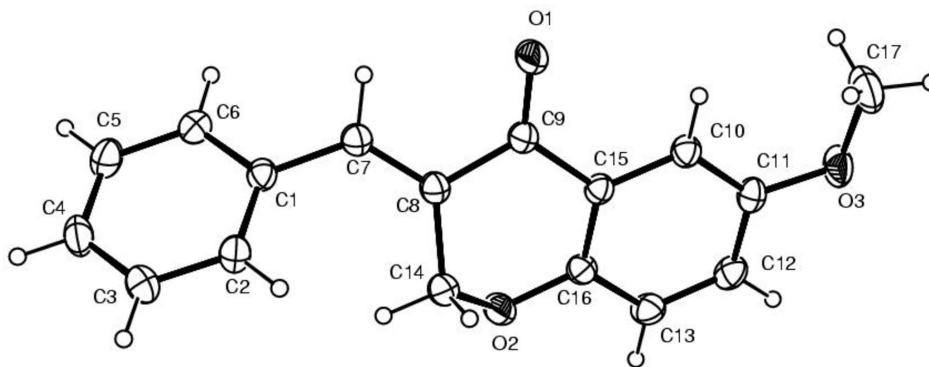
The geometric parameters in the title compound agree with values reported for a similar structure (Suresh *et al.*, 2007). The dihedral angle between the benzene ring of the chromanone moiety and the phenyl ring is 67.78 (3)°. The Chromanone moiety is fused with a six membered heterocyclic ring and the study of torsion angles, asymmetry parameters and least-square plane calculations shows that the chromanone adopts a half chair conformation with a deviation of C14 from the C8/C9/C15/C16/O2 plane by 0.616 (4) Å, Q_2= 0.4053 (14) Å, Q_3= -0.2052 (13)Å, and Q_T=0.4543 (14)Å (Cremer &Pople, (1975). The structure is stabilized by weak intermolecular C—H···O interaction that link the molecules into pairs around a center (Table 1). No other short intermolecular interactions were found.

S2. Experimental

Methyl-(2*Z*)-2-bromo methyl-3-aryl prop-2-enoate (0.006 mole, 1.53 g) was treated with 4-methoxy phenol (0.006 mole, 0.9 ml) in the presence of potassium carbonate in acetone at reflux temperature for 3 hrs. The pure ester, 3-aryl-2-(4-methoxy)-phenoxymethylprop-2-enoate was obtained after purifying it using silica gel and column chromatography (3% ethyl acetate - 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 triflouroacetic anhydride and the reaction mixture was refluxed in dichloro- methane for 1 hr. It was further purified by column chromatography (silica gel-3% ethyl acetate - hexane) and the crystals used for data collection were obtained by slow evaporation from methanol.

S3. 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 CH2, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{iso}(C) for CH3.

Figure 1

ORTEP of the molecule with atoms represented as 30% probability ellipsoids.

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Crystal data

$C_{17}H_{14}O_3$	$Z = 2$
$M_r = 266.28$	$F(000) = 280$
Triclinic, $P\overline{1}$	$D_x = 1.336$ Mg m^{-3}
Hall symbol: -p 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.2678$ (2) Å	Cell parameters from 4851 reflections
$b = 8.3151$ (2) Å	$\theta = 2.6$–$28.3°$
$c = 11.7999$ (4) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 95.964$ (1)°	$T = 298$ K
$\beta = 103.828$ (1)°	Block, colourless
$\gamma = 104.042$ (1)°	$0.45 \times 0.42 \times 0.38$ mm
$V = 661.74$ (3) Å^3	

Data collection

Bruker APEXII CCD area-detector diffractometer	8868 measured reflections
	3041 independent reflections
Radiation source: fine-focus sealed tube	2404 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.018$
φ and ω scans	$\theta_{max} = 28.3°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)	$h = -8 \rightarrow 9$
	$k = -10 \rightarrow 11$
$T_{min} = 0.960$, $T_{max} = 0.966$	$l = -15 \rightarrow 15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.039$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.116$	
$S = 1.04$	H atoms treated by a mixture of independent and constrained refinement
3041 reflections	
186 parameters	$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.1403P]$
0 restraints	where $P = (F_o^2 + 2F_c^2)/3$
Primary atom site location: structure-invariant direct methods	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.25$ e Å^{-3}
	$\Delta\rho_{min} = -0.23$ e Å^{-3}

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}
C1	−0.38800 (18)	0.42099 (15)	0.12980 (11)	0.0357 (3)
C2	−0.49675 (19)	0.45875 (16)	0.20631 (12)	0.0413 (3)
H2	−0.4480	0.5584	0.2610	0.050*
C3	−0.6764 (2)	0.34969 (18)	0.20189 (13)	0.0469 (3)
H3	−0.7484	0.3771	0.2527	0.056*
C4	−0.7491 (2)	0.20056 (18)	0.12250 (14)	0.0499 (3)
H4	−0.8689	0.1265	0.1205	0.060*
C5	−0.6433 (2)	0.16181 (18)	0.04615 (13)	0.0526 (4)
H5	−0.6916	0.0606	−0.0069	0.063*
C6	−0.4668 (2)	0.27146 (17)	0.04756 (12)	0.0448 (3)
H6	−0.3995	0.2458	−0.0066	0.054*
C7	−0.19833 (18)	0.53387 (16)	0.12988 (12)	0.0384 (3)
C8	−0.04981 (18)	0.62292 (15)	0.22311 (11)	0.0366 (3)
C9	0.12813 (18)	0.73207 (16)	0.20106 (11)	0.0375 (3)
C10	0.47891 (18)	0.89529 (15)	0.29540 (11)	0.0370 (3)
H10	0.4781	0.9476	0.2294	0.044*
C11	0.64996 (18)	0.92856 (15)	0.38626 (11)	0.0390 (3)
C12	0.65136 (19)	0.84577 (17)	0.48305 (12)	0.0439 (3)
H12	0.7679	0.8659	0.5429	0.053*
C13	0.4833 (2)	0.73495 (18)	0.49135 (12)	0.0439 (3)
H13	0.4862	0.6800	0.5562	0.053*
C14	−0.03927 (19)	0.62083 (18)	0.35174 (11)	0.0427 (3)
H14A	−0.0509	0.7271	0.3874	0.051*
H14B	−0.1486	0.5319	0.3584	0.051*
C15	0.30664 (17)	0.78222 (14)	0.30322 (10)	0.0336 (3)
C16	0.30814 (18)	0.70497 (15)	0.40210 (11)	0.0362 (3)
C17	0.8268 (3)	1.1358 (3)	0.29888 (18)	0.0764 (6)
H17A	0.8124	1.0644	0.2261	0.115*
H17B	0.9493	1.2225	0.3186	0.115*
H17C	0.7195	1.1861	0.2896	0.115*
O1	0.13097 (14)	0.77364 (14)	0.10531 (9)	0.0566 (3)

O2	0.14409 (13)	0.59397 (12)	0.41471 (8)	0.0454 (2)
O3	0.82540 (14)	1.03993 (13)	0.38987 (9)	0.0540 (3)
H7	−0.175 (2)	0.5435 (19)	0.0546 (15)	0.051 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0311 (6)	0.0373 (6)	0.0357 (6)	0.0073 (5)	0.0046 (5)	0.0092 (5)
C2	0.0344 (6)	0.0382 (6)	0.0475 (7)	0.0079 (5)	0.0088 (5)	0.0016 (5)
C3	0.0365 (7)	0.0516 (8)	0.0538 (8)	0.0108 (6)	0.0158 (6)	0.0100 (6)
C4	0.0349 (7)	0.0470 (7)	0.0587 (9)	−0.0018 (6)	0.0082 (6)	0.0122 (6)
C5	0.0497 (8)	0.0431 (7)	0.0504 (8)	−0.0009 (6)	0.0048 (6)	−0.0030 (6)
C6	0.0426 (7)	0.0480 (7)	0.0384 (7)	0.0072 (6)	0.0091 (5)	0.0016 (5)
C7	0.0339 (6)	0.0423 (6)	0.0378 (6)	0.0081 (5)	0.0092 (5)	0.0087 (5)
C8	0.0310 (6)	0.0403 (6)	0.0380 (6)	0.0084 (5)	0.0097 (5)	0.0074 (5)
C9	0.0321 (6)	0.0423 (6)	0.0353 (6)	0.0069 (5)	0.0071 (5)	0.0079 (5)
C10	0.0331 (6)	0.0381 (6)	0.0375 (6)	0.0086 (5)	0.0064 (5)	0.0066 (5)
C11	0.0297 (6)	0.0386 (6)	0.0444 (7)	0.0081 (5)	0.0059 (5)	0.0025 (5)
C12	0.0347 (7)	0.0539 (8)	0.0385 (7)	0.0150 (6)	0.0006 (5)	0.0039 (6)
C13	0.0413 (7)	0.0551 (8)	0.0352 (6)	0.0154 (6)	0.0067 (5)	0.0111 (5)
C14	0.0321 (6)	0.0548 (8)	0.0392 (7)	0.0076 (6)	0.0114 (5)	0.0059 (6)
C15	0.0298 (6)	0.0357 (6)	0.0334 (6)	0.0087 (5)	0.0067 (5)	0.0032 (4)
C16	0.0334 (6)	0.0401 (6)	0.0349 (6)	0.0101 (5)	0.0100 (5)	0.0047 (5)
C17	0.0437 (9)	0.0871 (13)	0.0821 (13)	−0.0091 (9)	0.0049 (8)	0.0372 (10)
O1	0.0392 (5)	0.0764 (7)	0.0416 (5)	−0.0049 (5)	0.0035 (4)	0.0223 (5)
O2	0.0369 (5)	0.0580 (6)	0.0403 (5)	0.0077 (4)	0.0104 (4)	0.0173 (4)
O3	0.0305 (5)	0.0574 (6)	0.0614 (6)	−0.0006 (4)	−0.0003 (4)	0.0152 (5)

Geometric parameters (Å, º)

C1—C2	1.3932 (18)	C10—C11	1.3805 (17)
C1—C6	1.3978 (17)	C10—C15	1.4007 (17)
C1—C7	1.4671 (17)	C10—H10	0.9300
C2—C3	1.3830 (19)	C11—O3	1.3704 (15)
C2—H2	0.9300	C11—C12	1.3927 (19)
C3—C4	1.378 (2)	C12—C13	1.371 (2)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.377 (2)	C13—C16	1.3938 (17)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.377 (2)	C14—O2	1.4434 (16)
C5—H5	0.9300	C14—H14A	0.9700
C6—H6	0.9300	C14—H14B	0.9700
C7—C8	1.3412 (17)	C15—C16	1.3883 (17)
C7—H7	0.950 (16)	C16—O2	1.3693 (15)
C8—C9	1.4846 (18)	C17—O3	1.403 (2)
C8—C14	1.5036 (18)	C17—H17A	0.9600
C9—O1	1.2187 (15)	C17—H17B	0.9600
C9—C15	1.4818 (16)	C17—H17C	0.9600

C2—C1—C6	118.20 (12)	O3—C11—C10	124.75 (12)
C2—C1—C7	122.70 (11)	O3—C11—C12	115.45 (11)
C6—C1—C7	119.07 (12)	C10—C11—C12	119.80 (12)
C3—C2—C1	120.72 (12)	C13—C12—C11	120.93 (12)
C3—C2—H2	119.6	C13—C12—H12	119.5
C1—C2—H2	119.6	C11—C12—H12	119.5
C4—C3—C2	120.26 (13)	C12—C13—C16	119.70 (12)
C4—C3—H3	119.9	C12—C13—H13	120.1
C2—C3—H3	119.9	C16—C13—H13	120.1
C5—C4—C3	119.63 (13)	O2—C14—C8	111.15 (10)
C5—C4—H4	120.2	O2—C14—H14A	109.4
C3—C4—H4	120.2	C8—C14—H14A	109.4
C6—C5—C4	120.64 (13)	O2—C14—H14B	109.4
C6—C5—H5	119.7	C8—C14—H14B	109.4
C4—C5—H5	119.7	H14A—C14—H14B	108.0
C5—C6—C1	120.49 (13)	C16—C15—C10	120.07 (11)
C5—C6—H6	119.8	C16—C15—C9	119.69 (11)
C1—C6—H6	119.8	C10—C15—C9	119.96 (11)
C8—C7—C1	128.33 (12)	O2—C16—C15	122.60 (11)
C8—C7—H7	115.3 (9)	O2—C16—C13	117.53 (11)
C1—C7—H7	116.4 (9)	C15—C16—C13	119.83 (12)
C7—C8—C9	118.66 (11)	O3—C17—H17A	109.5
C7—C8—C14	126.65 (12)	O3—C17—H17B	109.5
C9—C8—C14	114.65 (10)	H17A—C17—H17B	109.5
O1—C9—C15	121.81 (11)	O3—C17—H17C	109.5
O1—C9—C8	123.09 (11)	H17A—C17—H17C	109.5
C15—C9—C8	115.06 (11)	H17B—C17—H17C	109.5
C11—C10—C15	119.58 (12)	C16—O2—C14	113.85 (10)
C11—C10—H10	120.2	C11—O3—C17	117.35 (11)
C15—C10—H10	120.2		
C6—C1—C2—C3	0.93 (19)	C11—C12—C13—C16	−0.3 (2)
C7—C1—C2—C3	179.01 (12)	C7—C8—C14—O2	129.37 (13)
C1—C2—C3—C4	0.9 (2)	C9—C8—C14—O2	−48.50 (15)
C2—C3—C4—C5	−1.0 (2)	C11—C10—C15—C16	0.33 (18)
C3—C4—C5—C6	−0.7 (2)	C11—C10—C15—C9	−173.63 (11)
C4—C5—C6—C1	2.6 (2)	O1—C9—C15—C16	−166.84 (12)
C2—C1—C6—C5	−2.7 (2)	C8—C9—C15—C16	10.83 (17)
C7—C1—C6—C5	179.18 (12)	O1—C9—C15—C10	7.14 (19)
C2—C1—C7—C8	41.2 (2)	C8—C9—C15—C10	−175.19 (10)
C6—C1—C7—C8	−140.74 (14)	C10—C15—C16—O2	179.75 (11)
C1—C7—C8—C9	−178.86 (12)	C9—C15—C16—O2	−6.28 (18)
C1—C7—C8—C14	3.3 (2)	C10—C15—C16—C13	−2.59 (18)
C7—C8—C9—O1	16.3 (2)	C9—C15—C16—C13	171.39 (11)
C14—C8—C9—O1	−165.66 (13)	C12—C13—C16—O2	−179.65 (11)
C7—C8—C9—C15	−161.35 (11)	C12—C13—C16—C15	2.57 (19)
C14—C8—C9—C15	16.71 (16)	C15—C16—O2—C14	−27.22 (16)

C15—C10—C11—O3	−178.13 (11)	C13—C16—O2—C14	155.06 (11)
C15—C10—C11—C12	1.94 (18)	C8—C14—O2—C16	53.73 (14)
O3—C11—C12—C13	178.09 (12)	C10—C11—O3—C17	5.0 (2)
C10—C11—C12—C13	−2.0 (2)	C12—C11—O3—C17	−175.11 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O1[i]	0.93	2.53	3.4293 (18)	163

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