Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Bromomethyl-6-methoxy-2*H*-chromen-2-one

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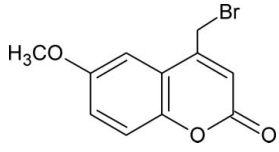
Received 8 September 2010; accepted 16 October 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.044; wR factor = 0.101; data-to-parameter ratio = 15.5.

The structure of the title coumarin derivative, $C_{11}H_9BrO_3$, is stabilized by weak intermolecular C—H···O hydrogen bonds.

Related literature

For the properties of coumarins, see: Kulkarni *et al.* (2006); Fylaktakidou *et al.* (2004); Neyts *et al.* (2009); Kempen *et al.* (2003). For structural analysis of coumarins, see: Gnanaguru *et al.* (1985); Munshi & Guru Row (2005); Gavuzzo *et al.* (1974); Moorthy *et al.* (2003); Katerinopoulos (2004). For Br-containing coumarins, see: Gaultier & Hauw (1965); Kokila *et al.* (1996); Vasudevan *et al.* (1991).



Experimental

Crystal data

$C_{11}H_9BrO_3$
M_r = 269.09
Monoclinic, $P2_1/n$
a = 4.3573 (3) Å
b = 9.2859 (6) Å
c = 25.2677 (17) Å
β = 91.927 (3)°
V = 1021.79 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.01 mm^{-1}
T = 293 K
0.25 × 0.15 × 0.1 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.434, T_{max} = 0.501
9950 measured reflections
2128 independent reflections
1501 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.101
S = 0.96
2128 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.93 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2···O1^{i}	0.93	2.60	3.451 (4)	152
C2—H2···O2^{i}	0.93	2.58	3.446 (5)	155
C10—H10A···O2^{i}	0.97	2.57	3.437 (5)	148
C8—H8···O2^{ii}	0.93	2.56	3.433 (5)	156
C10—H10A···O1^{iii}	0.97	2.98	3.601 (4)	122

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT* (Bruker, 2004); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

RG thanks the MVJ College of Engineering, Bangalore-67 (VTU Research Center) for providing research facilities. The authors also thank the SAIF, IIT-Madras, Channai, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2314).

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supplementary materials

Acta Cryst. (2010). E**66**, o2906 [**doi:10.1107/S1600536810042005**]

4-Bromomethyl-6-methoxy-2*H*-chromen-2-one

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Comment

Coumarins are a class of naturally occurring oxygen heterocycles which have been found to exhibit wide ranging biological activities (Kulkarni *et al.*, 2006; Fylaktakidou *et al.*, 2004; Neyts *et al.*, 2009) through its innumerable derivatives. Structural studies on coumarins have been focused on their solid state photochemical dimerization (Gnanaguru *et al.*, 1985), hydrogen bonding (Munshi *et al.*, 2005), mode of packing (Gavuzzo *et al.*, 1974), molecular self assembling (Moorthy *et al.*, 2003) and photophysical properties (Katerinopoulos *et al.*, 2004). Introduction of bromine has resulted in formation of hydrates, intermolecular hydrogen bonds, and eclipsed conformation, as observed in 3-bromocoumarin (Gaultier *et al.*, 1965), 6-bromo-3-acetylcoumarin (Kokila *et al.*, 1996), and 3-bromoacetylcoumarin (Vasudevan *et al.*, 1991), respectively. 3-Bromophenyl-6-acetoxymethyl-coumarin-3-carboxylates have been found to exhibit potential anticancer and antitumour activity (Kempen *et al.*, 2003).

The title compound is cyclic, planar and aromatic in nature due to the continuous delocalization of electrons over the coumarin rings system. There is a significant deviation from trigonality in bond angle at O1—C1—C2 [117.0 (3)°], due to the electronic repulsion of atom O2 which is bonded to C1. This is also reflected at C9—C4—C5 [117.9 (3)°] and C9—C4—C3 [117.6 (3)°] but these are due to fused benzene and α pyrone rings. Another significant deviation in bond angle is observed at C6—O3—C11 [118.0 (3)°] due to the repulsion between lone pair electrons of atom O3 with valence electrons of C6—O3 and O3—C11 bonds.

Experimental

To a mixture of equimolar quantity of 4-methoxy phenol (0.1 mol) and 4-bromoethylacetoacetate (0.1 mol) was added drop wise sulfuric acid (30 ml) with stirring and maintaining the temperature between 0-5 °C. The reaction mixture was allowed to stand in ice chest overnight and deep red coloured solution was poured into the stream of crushed ice. Solid separated was filtered and washed with water and then with cold ethanol so as to get a colourless compound. Finally, it is recrystallized from acetic acid.

Refinement

All the H atoms were positioned geometrically and refined using a riding model with bond lengths 0.97 (methylene), 0.96 (methyl) or 0.93 Å (aromatic). Isotropic displacement parameters were calculated as *U*īso~(H) = 1.5*U*~eq~(C) for methyl group C11 and *U*īso~(H) = 1.2*U*~eq~(C) for all other H atoms.

Figures

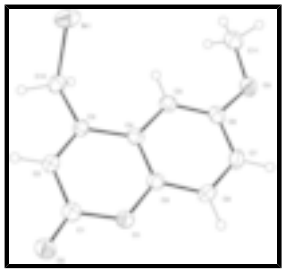

Fig. 1. *ORTEP* diagram of the title molecule with 50% probability displacement ellipsoids for non-H atoms.

4-Bromomethyl-6-methoxy-2*H*-chromen-2-one

Crystal data

$C_{11}H_9BrO_3$	$F(000) = 536$
$M_r = 269.09$	$D_x = 1.749$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3217 reflections
$a = 4.3573$ (3) Å	$\theta = 2.3$–$25.4°$
$b = 9.2859$ (6) Å	$\mu = 4.01$ mm^{-1}
$c = 25.2677$ (17) Å	$T = 293$ K
$\beta = 91.927$ (3)°	Needle, colourless
$V = 1021.79$ (12) Å^3	0.25 × 0.15 × 0.1 mm
$Z = 4$	

Data collection

Bruker Kappa APEXII CCD diffractometer	2128 independent reflections
Radiation source: fine-focus sealed tube	1501 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.037$
ω and φ scans	$\theta_{max} = 26.6°$, $\theta_{min} = 2.3°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	$h = -5 \rightarrow 3$
$T_{min} = 0.434$, $T_{max} = 0.501$	$k = -11 \rightarrow 11$
9950 measured reflections	$l = -31 \rightarrow 31$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.044$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.101$	H-atom parameters constrained
$S = 0.96$	$w = 1/[\sigma^2(F_o^2) + (0.0467P)^2 + 1.203P]$ where $P = (F_o^2 + 2F_c^2)/3$

2128 reflections	$(\Delta/\sigma)_{max} = 0.008$
137 parameters	$\Delta\rho_{max} = 0.93$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.26$ e Å^{-3}
0 constraints	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3658 (8)	0.2636 (4)	0.73840 (14)	0.0406 (9)
C2	0.4641 (8)	0.1339 (4)	0.71330 (13)	0.0375 (8)
H2	0.3944	0.0462	0.7260	0.045*
C3	0.6518 (7)	0.1332 (4)	0.67239 (13)	0.0321 (7)
C4	0.7577 (7)	0.2706 (4)	0.65171 (12)	0.0318 (7)
C5	0.9489 (7)	0.2851 (4)	0.60828 (13)	0.0348 (8)
H5	1.0139	0.2035	0.5905	0.042*
C6	1.0401 (7)	0.4188 (4)	0.59198 (13)	0.0378 (9)
C7	0.9456 (8)	0.5407 (4)	0.61872 (14)	0.0429 (9)
H7	1.0121	0.6311	0.6081	0.051*
C8	0.7556 (8)	0.5293 (4)	0.66048 (14)	0.0401 (9)
H8	0.6889	0.6113	0.6778	0.048*
C9	0.6642 (7)	0.3941 (4)	0.67652 (13)	0.0345 (8)
C10	0.7530 (9)	−0.0074 (4)	0.65024 (14)	0.0421 (9)
H10A	0.7086	−0.0837	0.6751	0.050*
H10B	0.9733	−0.0052	0.6460	0.050*
C11	1.2815 (9)	0.3270 (5)	0.51587 (15)	0.0535 (11)
H11A	1.4005	0.2554	0.5347	0.080*
H11B	1.3939	0.3612	0.4863	0.080*
H11C	1.0908	0.2856	0.5033	0.080*
O1	0.4744 (5)	0.3905 (3)	0.71899 (9)	0.0397 (6)
O2	0.1939 (7)	0.2719 (3)	0.77495 (11)	0.0569 (7)
O3	1.2213 (6)	0.4438 (3)	0.55030 (10)	0.0500 (7)
Br1	0.54925 (10)	−0.04981 (5)	0.582134 (17)	0.0612 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0437 (19)	0.039 (2)	0.039 (2)	−0.0003 (18)	0.0059 (16)	−0.0004 (17)
C2	0.0442 (19)	0.029 (2)	0.0390 (19)	−0.0025 (16)	0.0024 (16)	0.0004 (16)
C3	0.0345 (17)	0.027 (2)	0.0344 (18)	0.0024 (15)	0.0010 (14)	−0.0003 (14)
C4	0.0310 (16)	0.034 (2)	0.0301 (17)	0.0032 (15)	−0.0022 (13)	0.0012 (15)
C5	0.0338 (17)	0.036 (2)	0.0350 (18)	0.0037 (16)	0.0031 (14)	−0.0014 (16)
C6	0.0371 (18)	0.042 (2)	0.0343 (19)	−0.0030 (16)	0.0050 (15)	0.0044 (15)
C7	0.051 (2)	0.030 (2)	0.048 (2)	−0.0055 (18)	0.0027 (17)	0.0076 (17)
C8	0.050 (2)	0.029 (2)	0.042 (2)	0.0018 (17)	0.0008 (16)	−0.0045 (16)
C9	0.0335 (17)	0.037 (2)	0.0333 (18)	−0.0008 (15)	0.0020 (14)	−0.0007 (15)
C10	0.048 (2)	0.032 (2)	0.046 (2)	0.0050 (17)	0.0039 (17)	0.0013 (17)
C11	0.063 (3)	0.053 (3)	0.045 (2)	0.000 (2)	0.0162 (19)	0.002 (2)
O1	0.0483 (14)	0.0339 (15)	0.0376 (13)	0.0013 (12)	0.0117 (11)	−0.0024 (11)

O2	0.0759 (19)	0.0461 (18)	0.0508 (16)	0.0039 (15)	0.0305 (15)	−0.0005 (13)
O3	0.0618 (16)	0.0435 (17)	0.0460 (15)	−0.0072 (13)	0.0208 (13)	0.0030 (13)
Br1	0.0732 (3)	0.0518 (3)	0.0587 (3)	0.0043 (2)	0.0017 (2)	−0.0214 (2)

Geometric parameters (Å, °)

C1—O2	1.211 (4)	C7—C8	1.367 (5)
C1—O1	1.367 (4)	C7—H7	0.9300
C1—C2	1.434 (5)	C8—C9	1.382 (5)
C2—C3	1.340 (5)	C8—H8	0.9300
C2—H2	0.9300	C9—O1	1.377 (4)
C3—C4	1.459 (5)	C10—Br1	1.950 (4)
C3—C10	1.493 (5)	C10—H10A	0.9700
C4—C9	1.375 (5)	C10—H10B	0.9700
C4—C5	1.406 (5)	C11—O3	1.420 (5)
C5—C6	1.371 (5)	C11—H11A	0.9600
C5—H5	0.9300	C11—H11B	0.9600
C6—O3	1.357 (4)	C11—H11C	0.9600
C6—C7	1.388 (5)		
O2—C1—O1	116.7 (3)	C7—C8—C9	119.0 (3)
O2—C1—C2	126.3 (4)	C7—C8—H8	120.5
O1—C1—C2	117.0 (3)	C9—C8—H8	120.5
C3—C2—C1	123.0 (3)	C4—C9—C8	122.1 (3)
C3—C2—H2	118.5	C4—C9—O1	122.0 (3)
C1—C2—H2	118.5	C8—C9—O1	115.9 (3)
C2—C3—C4	118.7 (3)	C3—C10—Br1	112.1 (2)
C2—C3—C10	119.3 (3)	C3—C10—H10A	109.2
C4—C3—C10	122.0 (3)	Br1—C10—H10A	109.2
C9—C4—C5	117.9 (3)	C3—C10—H10B	109.2
C9—C4—C3	117.6 (3)	Br1—C10—H10B	109.2
C5—C4—C3	124.5 (3)	H10A—C10—H10B	107.9
C6—C5—C4	120.4 (3)	O3—C11—H11A	109.5
C6—C5—H5	119.8	O3—C11—H11B	109.5
C4—C5—H5	119.8	H11A—C11—H11B	109.5
O3—C6—C5	124.7 (3)	O3—C11—H11C	109.5
O3—C6—C7	115.4 (3)	H11A—C11—H11C	109.5
C5—C6—C7	119.9 (3)	H11B—C11—H11C	109.5
C8—C7—C6	120.7 (3)	C1—O1—C9	121.6 (3)
C8—C7—H7	119.7	C6—O3—C11	118.0 (3)
C6—C7—H7	119.7		
O2—C1—C2—C3	179.0 (3)	C5—C4—C9—C8	−0.9 (5)
O1—C1—C2—C3	−0.5 (5)	C3—C4—C9—C8	179.0 (3)
C1—C2—C3—C4	−1.0 (5)	C5—C4—C9—O1	179.3 (3)
C1—C2—C3—C10	177.4 (3)	C3—C4—C9—O1	−0.8 (4)
C2—C3—C4—C9	1.6 (4)	C7—C8—C9—C4	−0.2 (5)
C10—C3—C4—C9	−176.7 (3)	C7—C8—C9—O1	179.6 (3)
C2—C3—C4—C5	−178.5 (3)	C2—C3—C10—Br1	106.3 (3)
C10—C3—C4—C5	3.2 (5)	C4—C3—C10—Br1	−75.4 (3)
C9—C4—C5—C6	0.7 (5)	O2—C1—O1—C9	−178.1 (3)

C3—C4—C5—C6	−179.2 (3)	C2—C1—O1—C9	1.4 (5)
C4—C5—C6—O3	−179.3 (3)	C4—C9—O1—C1	−0.8 (5)
C4—C5—C6—C7	0.5 (5)	C8—C9—O1—C1	179.4 (3)
O3—C6—C7—C8	178.2 (3)	C5—C6—O3—C11	10.6 (5)
C5—C6—C7—C8	−1.6 (5)	C7—C6—O3—C11	−169.2 (3)
C6—C7—C8—C9	1.4 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···O1^{i}	0.93	2.60	3.451 (4)	152
C2—H2···O2^{i}	0.93	2.58	3.446 (5)	155
C10—H10A···O2^{i}	0.97	2.57	3.437 (5)	148
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Symmetry codes: (i) −*x*+1/2, *y*−1/2, −*z*+3/2; (ii) −*x*+1/2, *y*+1/2, −*z*+3/2; (iii) −*x*+3/2, *y*−1/2, −*z*+3/2.

Fig. 1