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4-Bromomethyl-7,8-dimethylcoumarin

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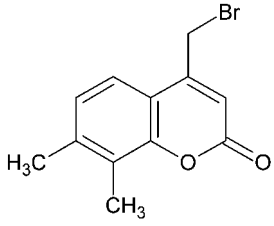
Key indicators: single-crystal X-ray study; T = 292 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.116; data-to-parameter ratio = 26.2.

In the title molecule, $C_{12}H_{11}BrO_2$, all non-H atoms with the exception of the Br atom are essentially coplanar (r.m.s. deviation = 0.018 Å). The C—Br bond is inclined by 80.17 (12)° to this plane. The crystal structure is stabilized by weak C—H···O hydrogen bonds.

Related literature

For potential synthetic applications of the title compound, see: Cui *et al.* (2007); Zhao *et al.* (2008). For related structures, see: Gowda *et al.* (2009, 2010).



Experimental

Crystal data

$C_{12}H_{11}BrO_2$
M_r = 267.12
Monoclinic, $C2/c$
a = 18.5025 (14) Å
b = 9.8785 (7) Å
c = 13.1639 (10) Å
β = 118.908 (2)°
V = 2106.3 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.88 mm^{-1}
T = 292 K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker 2004)
T_{min} = 0.432, T_{max} = 0.571
14710 measured reflections
3610 independent reflections
2516 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.116
S = 1.05
3610 reflections
138 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.73 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12B···O2^{i}	0.97	2.40	3.342 (3)	163

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2* and *SAINT* (Bruker, 2004); data reduction: *SAINT*; 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 *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5165).

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supporting information

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4-Bromomethyl-7,8-dimethylcoumarin

Ramakrishna Gowda, K.V. Arjuna Gowda, Mahantesha Basanagouda and Manohar V. Kulkarni

S1. Comment

The title compound has potential use in Heck and Suzuki cross coupling reactions (Cui *et al.*, 2007) and Negishi coupling reactions (Zhao *et al.*, 2008). In continuation of our work on the crystal structures of halogenated coumarin derivatives (Gowda *et al.*, 2009; 2010) herein we report crystal structure of title compound.

The molecular structure of the title compound is shown in Fig. 1. In the molecule, with the exception of the Br atom, all non-hydrogen atoms [C1-C12/O1/O2] are essentially planar [r.m.s. = 0.018Å]]. The C-Br bond is inclined [defined by the C7/C12/Br1 plane] by 80.17 (12)° to this plane. The crystal structure is stabilized by weak C-H···O hydrogen bonds (Fig. 2).

S2. Experimental

To a mixture of equimolar quantities of 2,3-dimethylphenol (0.1 mol) and 4-bromoethylacetoacetate (0.1 mol), sulfuric acid (30 ml) was added dropwise with stirring while maintaining the temperature between 273-278K. The reaction mixture was allowed to stand in ice chest overnight and the deep red coloured solution was poured into a stream of crushed ice. The solid which separated was filtered and washed with water and then with cold ethanol to yield a colourless compound which was recrystallized from acetic acid.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model with bond lengths 0.96 (methyl) or 0.93 Å (aromatic) and U_{iso}(H) = 1.5U_{eq}(C) for methyl groups and U_{iso}(H) = 1.2U_{eq}(C) for all other H atoms.

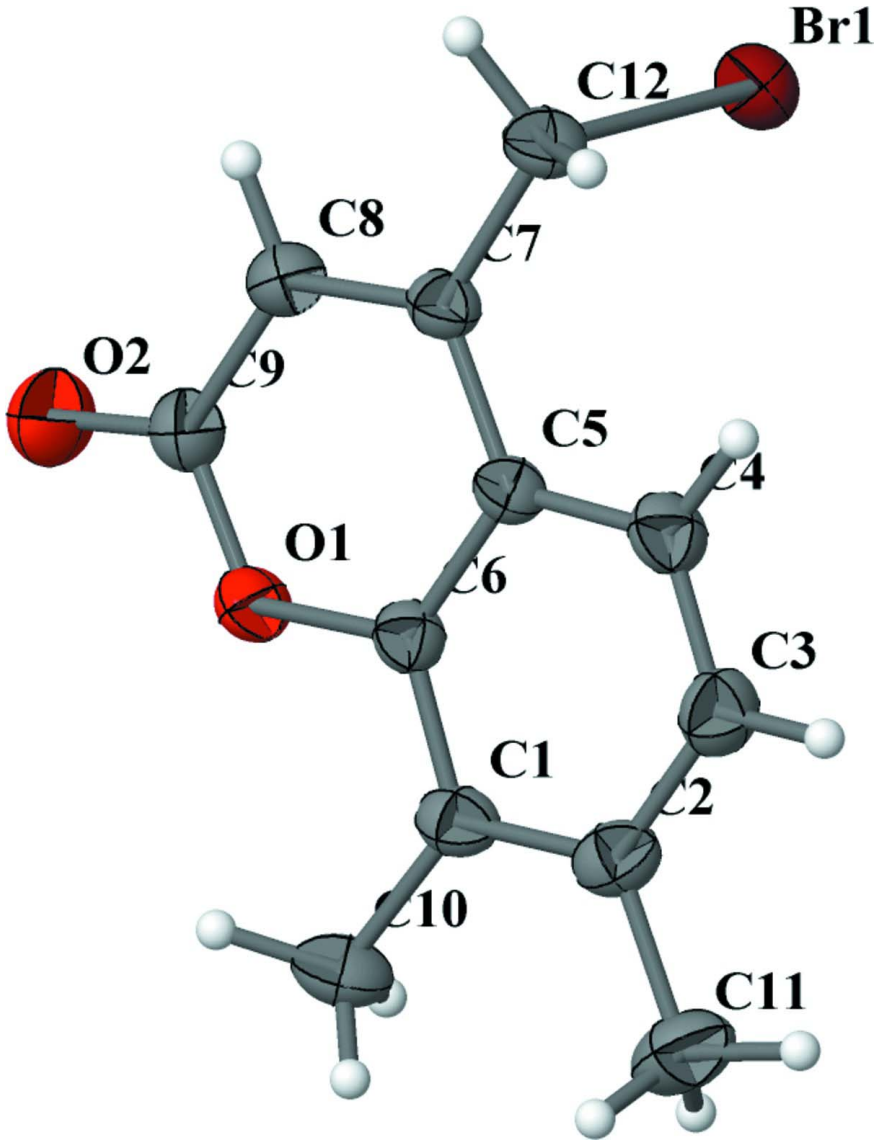

Figure 1

The molecular structure of the title compound shown with 50% probability displacement ellipsoids.

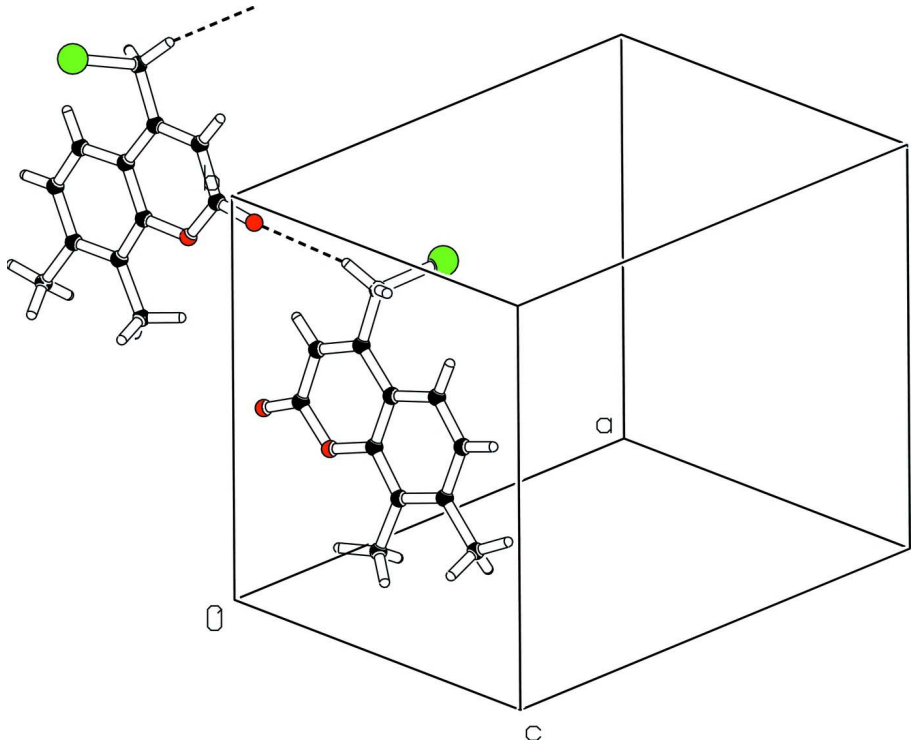

Figure 2

Part of the crystal structure showing weak C-H···O hydrogen bonds as dashed lines.

4-Bromomethyl-7,8-dimethylcoumarin

Crystal data

$C_{12}H_{11}BrO_2$
$M_r = 267.12$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 18.5025\ (14)$ Å
$b = 9.8785\ (7)$ Å
$c = 13.1639\ (10)$ Å
$\beta = 118.908\ (2)°$
$V = 2106.3\ (3)$ Å^3
$Z = 8$

$F(000) = 1072$
$D_x = 1.685$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5018 reflections
$\theta = 2.4–28.6°$
$\mu = 3.88$ mm^{-1}
$T = 292$ K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Bruker 2004)
$T_{min} = 0.432$, $T_{max} = 0.571$

14710 measured reflections
3610 independent reflections
2516 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 31.9°$, $\theta_{min} = 2.4°$
$h = -27\rightarrow27$
$k = -14\rightarrow14$
$l = -19\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.116$
$S = 1.05$
3610 reflections
138 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 1.9612P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.78$ e Å^{-3}
$\Delta\rho_{min} = -0.73$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.36207 (15)	0.1296 (2)	0.0849 (2)	0.0332 (5)
C2	0.39174 (15)	0.1512 (2)	0.2032 (2)	0.0363 (5)
C3	0.40658 (16)	0.2836 (3)	0.24674 (19)	0.0398 (5)
H3	0.4263	0.2974	0.3257	0.048*
C4	0.39291 (16)	0.3930 (3)	0.17644 (19)	0.0366 (5)
H4	0.4033	0.4797	0.2079	0.044*
C5	0.36354 (14)	0.3756 (2)	0.05795 (17)	0.0289 (4)
C6	0.34902 (13)	0.2433 (2)	0.01547 (17)	0.0292 (4)
C7	0.34654 (14)	0.4850 (2)	−0.02371 (18)	0.0303 (4)
C8	0.31617 (15)	0.4562 (2)	−0.13705 (19)	0.0354 (5)
H8	0.3025	0.5272	−0.1896	0.043*
C9	0.30411 (16)	0.3200 (2)	−0.1796 (2)	0.0371 (5)
C10	0.34498 (19)	−0.0090 (2)	0.0328 (3)	0.0460 (6)
H10A	0.3014	−0.0043	−0.0461	0.069*
H10B	0.3287	−0.0674	0.0764	0.069*
H10C	0.3939	−0.0443	0.0346	0.069*
C11	0.4099 (2)	0.0349 (3)	0.2855 (3)	0.0530 (7)
H11A	0.3594	−0.0110	0.2678	0.079*
H11B	0.4350	0.0684	0.3637	0.079*
H11C	0.4469	−0.0271	0.2778	0.079*
C12	0.36044 (16)	0.6286 (2)	0.0163 (2)	0.0383 (5)
H12A	0.3423	0.6421	0.0734	0.046*
H12B	0.3287	0.6883	−0.0489	0.046*
O1	0.32072 (11)	0.21805 (17)	−0.10098 (13)	0.0360 (4)
O2	0.28162 (15)	0.2864 (2)	−0.27881 (15)	0.0554 (5)

Br1	0.478174 (17)	0.67133 (3)	0.08473 (2)	0.04795 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0327 (11)	0.0270 (10)	0.0415 (11)	0.0027 (9)	0.0190 (9)	0.0012 (9)
C2	0.0344 (12)	0.0387 (13)	0.0398 (11)	0.0035 (10)	0.0211 (10)	0.0077 (9)
C3	0.0438 (13)	0.0458 (13)	0.0298 (10)	−0.0008 (11)	0.0178 (9)	−0.0007 (9)
C4	0.0432 (13)	0.0338 (12)	0.0336 (10)	−0.0029 (10)	0.0192 (9)	−0.0068 (9)
C5	0.0311 (11)	0.0248 (9)	0.0302 (9)	0.0002 (8)	0.0143 (8)	−0.0018 (7)
C6	0.0300 (10)	0.0285 (10)	0.0293 (9)	0.0007 (8)	0.0144 (8)	−0.0022 (7)
C7	0.0296 (10)	0.0252 (10)	0.0348 (9)	0.0003 (8)	0.0145 (8)	−0.0006 (8)
C8	0.0386 (12)	0.0317 (11)	0.0333 (9)	−0.0003 (9)	0.0153 (9)	0.0036 (8)
C9	0.0383 (12)	0.0369 (12)	0.0323 (9)	−0.0018 (10)	0.0140 (9)	−0.0019 (9)
C10	0.0564 (16)	0.0264 (11)	0.0576 (14)	0.0007 (11)	0.0295 (13)	−0.0007 (10)
C11	0.0552 (17)	0.0542 (17)	0.0511 (14)	0.0062 (14)	0.0269 (13)	0.0211 (13)
C12	0.0402 (13)	0.0256 (10)	0.0434 (12)	0.0017 (10)	0.0158 (10)	−0.0003 (9)
O1	0.0464 (10)	0.0278 (8)	0.0312 (7)	−0.0013 (7)	0.0169 (7)	−0.0053 (6)
O2	0.0764 (15)	0.0524 (11)	0.0310 (8)	−0.0053 (11)	0.0208 (9)	−0.0057 (8)
Br1	0.04529 (18)	0.04007 (16)	0.04762 (16)	−0.00806 (11)	0.01383 (12)	0.00024 (10)

Geometric parameters (Å, º)

C1—C6	1.393 (3)	C8—C9	1.433 (3)
C1—C2	1.394 (3)	C8—H8	0.9300
C1—C10	1.495 (3)	C9—O2	1.210 (3)
C2—C3	1.401 (4)	C9—O1	1.368 (3)
C2—C11	1.502 (3)	C10—H10A	0.9600
C3—C4	1.364 (4)	C10—H10B	0.9600
C3—H3	0.9300	C10—H10C	0.9600
C4—C5	1.392 (3)	C11—H11A	0.9600
C4—H4	0.9300	C11—H11B	0.9600
C5—C6	1.396 (3)	C11—H11C	0.9600
C5—C7	1.447 (3)	C12—Br1	1.959 (3)
C6—O1	1.382 (2)	C12—H12A	0.9700
C7—C8	1.346 (3)	C12—H12B	0.9700
C7—C12	1.492 (3)		
C6—C1—C2	117.3 (2)	C9—C8—H8	118.9
C6—C1—C10	120.4 (2)	O2—C9—O1	116.7 (2)
C2—C1—C10	122.3 (2)	O2—C9—C8	125.9 (2)
C1—C2—C3	119.6 (2)	O1—C9—C8	117.38 (19)
C1—C2—C11	121.2 (2)	C1—C10—H10A	109.5
C3—C2—C11	119.2 (2)	C1—C10—H10B	109.5
C4—C3—C2	121.7 (2)	H10A—C10—H10B	109.5
C4—C3—H3	119.1	C1—C10—H10C	109.5
C2—C3—H3	119.1	H10A—C10—H10C	109.5
C3—C4—C5	120.4 (2)	H10B—C10—H10C	109.5

C3—C4—H4	119.8	C2—C11—H11A	109.5
C5—C4—H4	119.8	C2—C11—H11B	109.5
C4—C5—C6	117.4 (2)	H11A—C11—H11B	109.5
C4—C5—C7	124.5 (2)	C2—C11—H11C	109.5
C6—C5—C7	118.09 (18)	H11A—C11—H11C	109.5
O1—C6—C1	115.77 (19)	H11B—C11—H11C	109.5
O1—C6—C5	120.65 (19)	C7—C12—Br1	109.33 (17)
C1—C6—C5	123.6 (2)	C7—C12—H12A	109.8
C8—C7—C5	119.4 (2)	Br1—C12—H12A	109.8
C8—C7—C12	120.0 (2)	C7—C12—H12B	109.8
C5—C7—C12	120.64 (19)	Br1—C12—H12B	109.8
C7—C8—C9	122.3 (2)	H12A—C12—H12B	108.3
C7—C8—H8	118.9	C9—O1—C6	122.16 (18)

C6—C1—C2—C3	0.3 (4)	C7—C5—C6—C1	−179.3 (2)
C10—C1—C2—C3	180.0 (2)	C4—C5—C7—C8	−178.3 (2)
C6—C1—C2—C11	−178.3 (2)	C6—C5—C7—C8	1.2 (3)
C10—C1—C2—C11	1.3 (4)	C4—C5—C7—C12	0.3 (4)
C1—C2—C3—C4	−0.1 (4)	C6—C5—C7—C12	179.7 (2)
C11—C2—C3—C4	178.6 (3)	C5—C7—C8—C9	−3.5 (4)
C2—C3—C4—C5	−0.1 (4)	C12—C7—C8—C9	178.0 (2)
C3—C4—C5—C6	0.0 (4)	C7—C8—C9—O2	−175.6 (3)
C3—C4—C5—C7	179.5 (2)	C7—C8—C9—O1	3.3 (4)
C2—C1—C6—O1	179.1 (2)	C8—C7—C12—Br1	−101.7 (2)
C10—C1—C6—O1	−0.5 (3)	C5—C7—C12—Br1	79.8 (2)
C2—C1—C6—C5	−0.4 (4)	O2—C9—O1—C6	178.2 (2)
C10—C1—C6—C5	180.0 (2)	C8—C9—O1—C6	−0.8 (4)
C4—C5—C6—O1	−179.3 (2)	C1—C6—O1—C9	179.1 (2)
C7—C5—C6—O1	1.2 (3)	C5—C6—O1—C9	−1.4 (3)
C4—C5—C6—C1	0.2 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12*B*···O2^{i}	0.97	2.40	3.342 (3)	163

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