

6-Bromo-3-(dibromoacetyl)-2H-chromen-2-one

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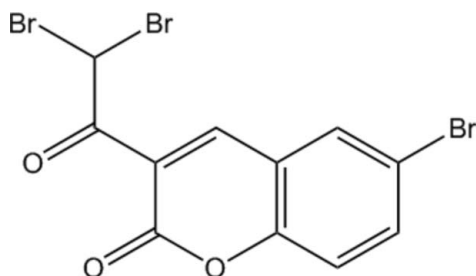
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Key indicators: single-crystal X-ray study; $T = 290$ K; mean $\sigma(\text{C}-\text{C}) = 0.012$ Å; R factor = 0.068; wR factor = 0.173; data-to-parameter ratio = 15.5.

In the title compound, $\text{C}_{11}\text{H}_5\text{Br}_3\text{O}_3$, the coumarin group is planar [maximum deviation from the mean plane is 0.032 (3) Å] and makes a dihedral angle of 19.9 (3)° with the dibromoacetyl group. The crystal structure is stabilized by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For related literature concerning coumarin derivatives, see: Vishnumurthy *et al.* (1996, 1997, 1999, 2001). For synthetic details, see: Venugopala *et al.* (2004).



Experimental

Crystal data

$\text{C}_{11}\text{H}_5\text{Br}_3\text{O}_3$	$V = 1213.4$ (16) Å ³
$M_r = 424.85$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 7.999$ (6) Å	$\mu = 9.96$ mm ⁻¹
$b = 6.794$ (5) Å	$T = 290$ (2) K
$c = 22.442$ (17) Å	$0.23 \times 0.04 \times 0.03$ mm
$\beta = 95.819$ (12)°	

Data collection

Bruker SMART CCD diffractometer	8820 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2443 independent reflections
$T_{\min} = 0.595$, $T_{\max} = 0.742$	1585 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.066$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.068$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.173$	$\Delta\rho_{\text{max}} = 1.20$ e Å ⁻³
$S = 1.05$	$\Delta\rho_{\text{min}} = -1.26$ e Å ⁻³
2443 reflections	
158 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C7}-\text{H7}\cdots\text{O3}^i$	0.93	2.43	3.264 (9)	149
$\text{C1}-\text{H1}\cdots\text{O3}^i$	0.93	2.51	3.317 (10)	145
$\text{C11}-\text{H11}\cdots\text{O1}^{ii}$	1.00 (9)	2.59 (9)	3.519 (11)	154 (1)
$\text{C11}-\text{H11}\cdots\text{O2}^{ii}$	1.00 (9)	2.58 (9)	3.293 (12)	128 (1)

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

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1993); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1999) and CAMERON (Watkin *et al.*, 1993); software used to prepare material for publication: PLATON (Spek, 2003).

We thank the Department of Science and Technology and Professor T. N. Guru Row for the data collection on the CCD facility under the IRHPA-DST scheme. DC thanks the CSIR, India, and the IISc for a Senior Research Fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BI2256).

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supplementary materials

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Comment

Coumarins are an important class of organic compounds having vast structural diversity and useful applications in several areas of synthetic chemistry, medicinal chemistry and photochemistry (Vishnumurthy *et al.*, 1996, 1997, 1999). The formation of [2 + 2] cycloaddition products upon irradiation (Vishnumurthy *et al.*, 2001) of coumarin and its derivatives has demonstrated the importance of preorganization of molecules in the crystalline solid state.

In the title compound (Fig. 1), the coumarin group is planar with a maximum deviation of $-0.032(3)\text{\AA}$ from the weighted least squares plane for atom C8. C—H \cdots O hydrogen bonds involving H1, H7 and O3, are observed between molecules, forming bifurcated $R^2_2(14)$ and $R^2_2(10)$ dimeric units. Furthermore, H11, O1 and O2 are involved in formation of a trimeric motif $R^2_1(4)$, forming chains along the *b* axis (Fig. 2, Table. 1).

Experimental

The title compound was synthesized in accordance with the procedure reported in the literature (Venugopala *et al.*, 2004). Single crystals were grown from a solution in glacial acetic acid.

Refinement

The methine H atom was located in a difference Fourier map and refined isotropically, while the remaining H atoms were placed in calculated positions with C—H = 0.93\AA and refined as riding with $U_{\text{eq}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

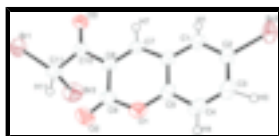


Fig. 1. Molecular structure of the title compound drawn with displacement ellipsoids at 50% probability for non-H atoms.

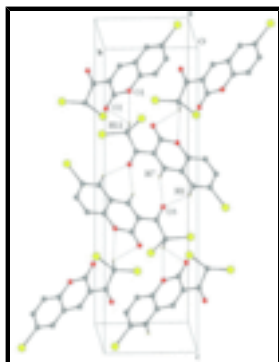


Fig. 2. Packing diagram highlighting C—H \cdots O hydrogen bonds.

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Crystal data

$C_{11}H_5Br_3O_3$	$F_{000} = 800$
$M_r = 424.85$	$D_x = 2.326 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
Hall symbol: -P 2yn	$\lambda = 0.71073 \text{ \AA}$
$a = 7.999 (6) \text{ \AA}$	Cell parameters from 765 reflections
$b = 6.794 (5) \text{ \AA}$	$\theta = 1.4\text{--}25.8^\circ$
$c = 22.442 (17) \text{ \AA}$	$\mu = 9.96 \text{ mm}^{-1}$
$\beta = 95.819 (12)^\circ$	$T = 290 (2) \text{ K}$
$V = 1213.4 (16) \text{ \AA}^3$	Needle, yellow
$Z = 4$	$0.23 \times 0.04 \times 0.03 \text{ mm}$

Data collection

Bruker SMART CCD diffractometer	2443 independent reflections
Radiation source: fine-focus sealed tube	1585 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.066$
$T = 290(2) \text{ K}$	$\theta_{\text{max}} = 26.4^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.8^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{\text{min}} = 0.595$, $T_{\text{max}} = 0.742$	$k = -8 \rightarrow 8$
8820 measured reflections	$l = -27 \rightarrow 27$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.068$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.173$	$w = 1/[\sigma^2(F_o^2) + (0.0977P)^2]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2443 reflections	$(\Delta/\sigma)_{\text{max}} = <0.000$
158 parameters	$\Delta\rho_{\text{max}} = 1.20 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -1.26 \text{ e \AA}^{-3}$
	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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	-0.09220 (13)	1.46570 (14)	0.16134 (5)	0.0483 (3)
Br2	0.32441 (12)	0.18266 (14)	-0.04363 (5)	0.0513 (4)
Br3	-0.16865 (16)	1.03307 (18)	0.20405 (5)	0.0654 (4)
O1	0.3421 (8)	0.7006 (9)	0.1727 (3)	0.0430 (15)
O2	0.2656 (11)	0.9552 (11)	0.2223 (3)	0.075 (3)
O3	-0.0060 (8)	1.1803 (9)	0.0700 (2)	0.0438 (16)
C1	0.2423 (10)	0.5207 (12)	0.0200 (4)	0.0328 (19)
C2	0.3320 (11)	0.3498 (12)	0.0239 (4)	0.037 (2)
C3	0.4266 (11)	0.2942 (14)	0.0771 (4)	0.046 (2)
C4	0.4265 (11)	0.4105 (13)	0.1262 (4)	0.045 (2)
C5	0.3374 (11)	0.5832 (12)	0.1224 (4)	0.037 (2)
C6	0.2424 (10)	0.6429 (11)	0.0695 (4)	0.0293 (18)
C7	0.1510 (10)	0.8219 (12)	0.0713 (3)	0.0295 (18)
C8	0.1489 (10)	0.9319 (12)	0.1208 (4)	0.0304 (18)
C9	0.2510 (13)	0.8716 (13)	0.1758 (4)	0.042 (2)
C10	0.0453 (10)	1.1136 (12)	0.1172 (4)	0.0308 (18)
C11	-0.0027 (11)	1.2063 (13)	0.1754 (4)	0.037 (2)
H1	0.1811	0.5554	-0.0159	0.039*
H3	0.4889	0.1785	0.0788	0.055*
H7	0.0896	0.8649	0.0363	0.035*
H11	0.075 (12)	1.188 (13)	0.213 (4)	0.05 (3)*
H4	0.4865	0.3735	0.1621	0.054*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0425 (6)	0.0451 (6)	0.0587 (6)	0.0118 (4)	0.0125 (5)	-0.0063 (4)
Br2	0.0372 (6)	0.0460 (6)	0.0712 (7)	0.0105 (4)	0.0073 (5)	-0.0152 (5)
Br3	0.0631 (8)	0.0721 (8)	0.0660 (7)	-0.0104 (6)	0.0312 (6)	0.0105 (5)
O1	0.034 (4)	0.052 (4)	0.041 (3)	0.015 (3)	-0.007 (3)	0.003 (3)
O2	0.096 (7)	0.077 (5)	0.045 (4)	0.042 (5)	-0.030 (4)	-0.017 (4)
O3	0.055 (4)	0.047 (4)	0.030 (3)	0.015 (3)	0.005 (3)	0.001 (3)

supplementary materials

C1	0.018 (4)	0.039 (5)	0.040 (5)	0.004 (3)	0.001 (4)	-0.001 (4)
C2	0.022 (5)	0.039 (5)	0.050 (5)	0.003 (4)	0.008 (4)	-0.005 (4)
C3	0.028 (5)	0.043 (6)	0.065 (6)	0.010 (4)	0.001 (5)	-0.004 (5)
C4	0.027 (5)	0.047 (5)	0.057 (6)	0.012 (4)	-0.009 (4)	0.008 (5)
C5	0.030 (5)	0.032 (5)	0.048 (5)	0.003 (4)	0.003 (4)	0.004 (4)
C6	0.018 (4)	0.032 (5)	0.039 (5)	0.001 (3)	0.005 (4)	0.003 (3)
C7	0.019 (4)	0.040 (5)	0.028 (4)	0.005 (3)	0.000 (3)	0.004 (3)
C8	0.022 (5)	0.035 (5)	0.033 (4)	0.002 (3)	0.001 (4)	0.005 (3)
C9	0.043 (6)	0.044 (6)	0.038 (5)	0.007 (4)	-0.005 (4)	-0.003 (4)
C10	0.024 (5)	0.035 (4)	0.033 (5)	-0.004 (3)	0.003 (4)	-0.002 (4)
C11	0.030 (5)	0.040 (5)	0.041 (5)	0.006 (4)	0.005 (4)	0.003 (4)

Geometric parameters (\AA , $^\circ$)

Br1—C11	1.917 (9)	C7—C8	1.341 (11)
Br2—C2	1.890 (9)	C7—H7	0.930
Br3—C11	1.932 (9)	C3—C4	1.355 (13)
O3—C10	1.187 (10)	C3—C2	1.399 (13)
O1—C9	1.377 (10)	C3—H3	0.930
O1—C5	1.378 (10)	C8—C9	1.467 (12)
C6—C1	1.387 (11)	C8—C10	1.484 (11)
C6—C5	1.403 (12)	O2—C9	1.183 (10)
C6—C7	1.422 (11)	C10—C11	1.534 (11)
C1—C2	1.363 (11)	C11—H11	1.00 (9)
C1—H1	0.930	C4—H4	0.930
C5—C4	1.371 (12)		
C9—O1—C5	124.0 (7)	O2—C9—O1	116.4 (8)
C1—C6—C5	117.5 (7)	O2—C9—C8	127.1 (9)
C1—C6—C7	125.2 (8)	O1—C9—C8	116.4 (7)
C5—C6—C7	117.4 (7)	O3—C10—C8	120.4 (7)
C2—C1—C6	120.1 (8)	O3—C10—C11	120.7 (8)
C2—C1—H1	120.0	C8—C10—C11	118.8 (7)
C6—C1—H1	120.0	C1—C2—C3	121.3 (8)
C4—C5—O1	118.3 (8)	C1—C2—Br2	119.1 (7)
C4—C5—C6	122.2 (8)	C3—C2—Br2	119.6 (6)
O1—C5—C6	119.5 (7)	C10—C11—Br1	111.0 (6)
C8—C7—C6	123.4 (7)	C10—C11—Br3	105.8 (6)
C8—C7—H7	118.3	Br1—C11—Br3	110.9 (4)
C6—C7—H7	118.3	C10—C11—H11	118 (5)
C4—C3—C2	119.4 (8)	Br1—C11—H11	116 (5)
C4—C3—H3	120.3	Br3—C11—H11	92 (5)
C2—C3—H3	120.3	C3—C4—C5	119.5 (9)
C7—C8—C9	119.2 (7)	C3—C4—H4	120.2
C7—C8—C10	118.2 (7)	C5—C4—H4	120.2
C9—C8—C10	122.6 (7)		
C5—C6—C1—C2	0.1 (12)	C10—C8—C9—O1	-179.4 (8)
C7—C6—C1—C2	-178.5 (8)	C7—C8—C10—O3	-15.0 (12)
C9—O1—C5—C4	-176.2 (8)	C9—C8—C10—O3	163.7 (8)
C9—O1—C5—C6	3.8 (13)	C7—C8—C10—C11	161.5 (8)

C1—C6—C5—C4	-0.5 (13)	C9—C8—C10—C11	-19.8 (12)
C7—C6—C5—C4	178.2 (8)	C6—C1—C2—C3	-0.5 (13)
C1—C6—C5—O1	179.5 (7)	C6—C1—C2—Br2	178.2 (6)
C7—C6—C5—O1	-1.7 (12)	C4—C3—C2—C1	1.3 (14)
C1—C6—C7—C8	177.2 (8)	C4—C3—C2—Br2	-177.4 (7)
C5—C6—C7—C8	-1.5 (12)	O3—C10—C11—Br1	-16.2 (10)
C6—C7—C8—C9	2.7 (13)	C8—C10—C11—Br1	167.3 (6)
C6—C7—C8—C10	-178.6 (7)	O3—C10—C11—Br3	104.3 (8)
C5—O1—C9—O2	178.7 (9)	C8—C10—C11—Br3	-72.2 (8)
C5—O1—C9—C8	-2.5 (13)	C2—C3—C4—C5	-1.7 (15)
C7—C8—C9—O2	177.9 (11)	O1—C5—C4—C3	-178.7 (8)
C10—C8—C9—O2	-0.7 (16)	C6—C5—C4—C3	1.3 (15)
C7—C8—C9—O1	-0.8 (13)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C7—H7 \cdots O3 ⁱ	0.93	2.43	3.264 (9)	149
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Fig. 1

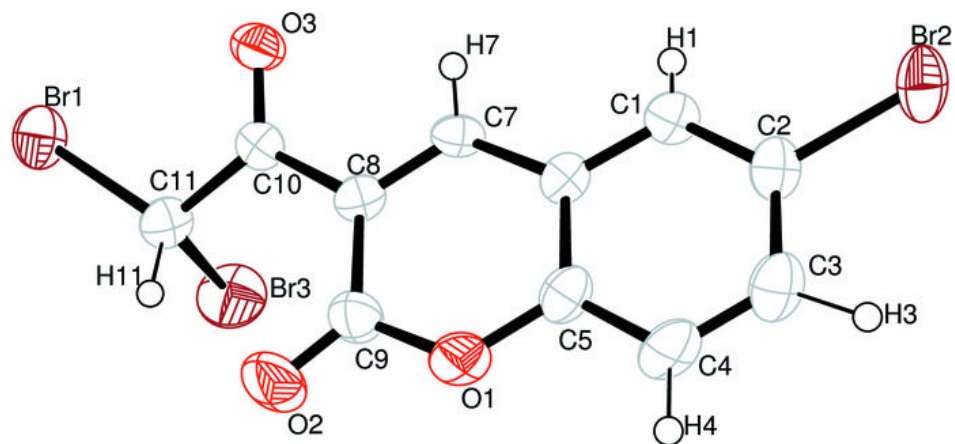


Fig. 2

