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## Structure Reports

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## 3-(2-Bromoacetyl)-6-fluoro-2H-chromen-2-one

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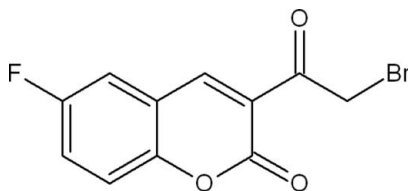
Received 24 July 2011; accepted 1 August 2011

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.029;  $wR$  factor = 0.065; data-to-parameter ratio = 13.8.

The non-H atoms of the title compound,  $\text{C}_{11}\text{H}_6\text{BrFO}_3$ , are essentially coplanar (r.m.s. deviation for all non-H atoms = 0.074 Å). In the crystal, the molecules are linked by  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\text{Br}$  interactions.

## Related literature

For background to coumarins, see: Hooper *et al.*, (1982); Morris *et al.* (1971); Khalfan *et al.* (1987); Domagala *et al.* (1996); Eid *et al.* (1994).



## Experimental

## Crystal data

 $\text{C}_{11}\text{H}_6\text{BrFO}_3$  $M_r = 285.06$ Monoclinic,  $P2_1/n$  $a = 4.0590$  (5) Å $b = 11.7719$  (13) Å $c = 21.608$  (2) Å $\beta = 94.318$  (10)° $V = 1029.6$  (2) Å<sup>3</sup> $Z = 4$ Mo  $K\alpha$  radiation $\mu = 3.99$  mm<sup>-1</sup> $T = 293$  K

0.30 × 0.20 × 0.10 mm

## Data collection

Bruker SMART CCD area-detector

Absorption correction: multi-scan

(SADABS; Sheldrick, 1996)

 $T_{\min} = 0.201$ ,  $T_{\max} = 0.506$ 

10491 measured reflections

2007 independent reflections

1438 reflections with  $I > 2\sigma(I)$  $R_{\text{int}} = 0.036$ 

## Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.029$  $wR(F^2) = 0.065$  $S = 0.95$ 

2007 reflections

145 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.21$  e Å<sup>-3</sup> $\Delta\rho_{\text{min}} = -0.37$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C3}-\text{H3}\cdots\text{O3}^{\text{i}}$	0.93	2.45	3.296 (3)	152
$\text{C7}-\text{H7}\cdots\text{O2}^{\text{ii}}$	0.93	2.52	3.425 (3)	164
$\text{C11}-\text{H11A}\cdots\text{Br1}^{\text{iii}}$	0.97	2.89	3.747 (3)	148

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Window (Farrugia, 1997); software used to prepare material for publication: PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5592).

## References

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## supporting information

*Acta Cryst.* (2011). E67, o2264 [doi:10.1107/S1600536811030960]

**3-(2-Bromoacetyl)-6-fluoro-2H-chromen-2-one**

**H. N. HarishKumar, Sudarshan Mahapatra, K. N. Venugopala and Kittappa Mariswamy Mahadevan**

**S1. Comment**

Coumarine derivatives have potential application in the dye industry (Hooper *et al.*, 1982 & Morris *et al.*, 1971), developing LASER dyes (Khalfan *et al.*, 1987) and pharmaceutical industry for their antiviral activity (Domagala *et al.*, 1996) and antimicrobial activity (Eid *et al.*, 1994). 3-Acetyl coumarins is found to be a major compound in the coumarine series and the title compound is a member of this family. The molecule forms well defined dimer *via* C—H $\cdots$ O intermolecular interaction through the center of inversion. The three dimensional packing motif in the title compound is built up of C—H $\cdots$ O and C—H $\cdots$ Br intermolecular interaction.

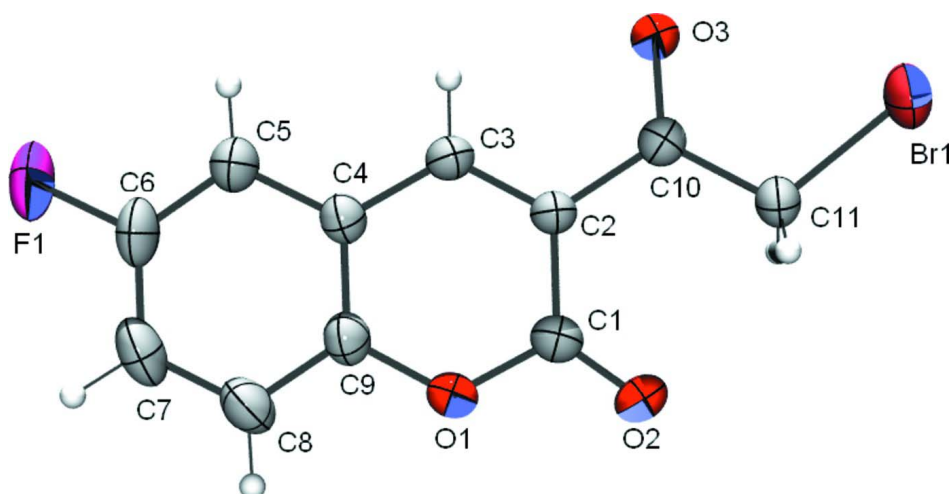
**S2. Experimental**

**Synthesis of 3-Bromoacetyl-6-fluoro-2H-1-benzopyran-2-one:** To a solution of compound 3-acetyl-6-fluoro-2H-1-benzopyran-2-one (206 mg, 1 mmol) in alcohol free chloroform (5 ml), bromine (173.8 mg, 1.1 mmol) in chloroform (2 ml) was added with intermittent shaking and warming. The mixture was heated for fifteen minutes on a water bath, cooled and filtered. The solid was washed with ether and crystallized from glacial acetic acid to yield 3-bromoacetyl-6-fluoro-2H-1-benzopyran-2-one.

**Crystallization:** Needle shape crystals of 3-acetyl-6-fluoro-2H-1-benzopyran-2-one was obtained by dissolving in glacial acetic acid and warmed for few minutes in a 5 ml beaker. Then the total content was covered by paraffin film with few punches and kept for crystallization at room temperature.

**S3. Refinement**

All H atoms were positioned geometrically and refined using a riding model.

**Figure 1**

View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

### 3-(2-Bromoacetyl)-6-fluoro-2H-chromen-2-one

#### Crystal data

$C_{11}H_6BrFO_3$

$M_r = 285.06$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1/n$

$a = 4.0590$  (5) Å

$b = 11.7719$  (13) Å

$c = 21.608$  (2) Å

$\beta = 94.318$  (10)°

$V = 1029.6$  (2) Å<sup>3</sup>

$Z = 4$

$F(000) = 560$

$D_x = 1.839$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 2007 reflections

$\theta = 3.3$ – $26.0$ °

$\mu = 3.99$  mm<sup>-1</sup>

$T = 293$  K

Needle, yellow

$0.30 \times 0.20 \times 0.10$  mm

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: Enhance (Mo) X-ray Source

Graphite monochromator

Detector resolution: 16.0839 pixels mm<sup>-1</sup>

$\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.201$ ,  $T_{\max} = 0.506$

10491 measured reflections

2007 independent reflections

1438 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 26.0$ °,  $\theta_{\min} = 3.3$ °

$h = -5 \rightarrow 4$

$k = -14 \rightarrow 14$

$l = -26 \rightarrow 26$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.065$

$S = 0.95$

2007 reflections

145 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

$$w = 1/[\sigma^2(F_o^2) + (0.0335P)^2]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$$

*Special details*

**Geometry.** Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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 ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.29450 (7)	0.10699 (2)	-0.07092 (1)	0.0553 (1)
F1	0.1896 (5)	0.78595 (14)	0.21546 (8)	0.0728 (7)
O1	-0.1428 (4)	0.34429 (15)	0.16976 (8)	0.0450 (6)
O2	-0.1904 (5)	0.19171 (16)	0.11181 (9)	0.0630 (8)
O3	0.3915 (5)	0.34027 (16)	-0.01840 (9)	0.0606 (8)
C1	-0.0789 (7)	0.2860 (2)	0.11695 (12)	0.0405 (9)
C2	0.1133 (6)	0.3458 (2)	0.07253 (11)	0.0320 (8)
C3	0.1926 (6)	0.4562 (2)	0.08252 (11)	0.0351 (9)
C4	0.1121 (6)	0.5154 (2)	0.13648 (11)	0.0338 (8)
C5	0.1933 (7)	0.6296 (2)	0.14854 (13)	0.0428 (10)
C6	0.1139 (7)	0.6754 (2)	0.20335 (14)	0.0478 (10)
C7	-0.0399 (7)	0.6145 (3)	0.24778 (13)	0.0531 (11)
C8	-0.1208 (7)	0.5031 (3)	0.23639 (12)	0.0487 (10)
C9	-0.0485 (6)	0.4551 (2)	0.18082 (11)	0.0372 (9)
C10	0.2260 (6)	0.2882 (2)	0.01622 (12)	0.0362 (9)
C11	0.1383 (7)	0.1654 (2)	0.00482 (12)	0.0417 (9)
H3	0.30428	0.49474	0.05289	0.0421*
H5	0.29859	0.67285	0.11987	0.0514*
H7	-0.08758	0.64873	0.28485	0.0637*
H8	-0.22291	0.46050	0.26572	0.0585*
H11A	-0.10000	0.15723	0.00318	0.0500*
H11B	0.23180	0.12039	0.03940	0.0500*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Br1	0.0651 (2)	0.0464 (2)	0.0556 (2)	-0.0025 (2)	0.0125 (2)	-0.0186 (2)
F1	0.1021 (14)	0.0435 (11)	0.0734 (12)	-0.0040 (10)	0.0097 (10)	-0.0273 (9)
O1	0.0634 (12)	0.0387 (11)	0.0351 (10)	-0.0073 (10)	0.0176 (9)	-0.0014 (9)
O2	0.0971 (16)	0.0386 (13)	0.0576 (13)	-0.0262 (12)	0.0342 (12)	-0.0060 (10)
O3	0.0977 (16)	0.0382 (11)	0.0506 (12)	-0.0227 (12)	0.0377 (12)	-0.0104 (10)
C1	0.0487 (17)	0.0389 (17)	0.0346 (15)	-0.0033 (14)	0.0086 (13)	0.0010 (13)
C2	0.0372 (15)	0.0297 (14)	0.0297 (14)	-0.0041 (12)	0.0070 (12)	0.0004 (11)

C3	0.0406 (15)	0.0320 (16)	0.0331 (15)	-0.0054 (12)	0.0065 (12)	0.0017 (11)
C4	0.0361 (14)	0.0329 (15)	0.0324 (14)	0.0025 (12)	0.0027 (12)	-0.0025 (12)
C5	0.0499 (17)	0.0359 (17)	0.0433 (16)	-0.0014 (13)	0.0075 (13)	-0.0040 (12)
C6	0.0550 (18)	0.0336 (17)	0.0540 (19)	0.0068 (15)	-0.0013 (16)	-0.0144 (14)
C7	0.063 (2)	0.055 (2)	0.0413 (17)	0.0138 (17)	0.0044 (15)	-0.0154 (15)
C8	0.0570 (19)	0.0529 (19)	0.0376 (16)	0.0047 (16)	0.0123 (14)	-0.0051 (14)
C9	0.0416 (16)	0.0338 (16)	0.0363 (15)	0.0039 (13)	0.0037 (13)	-0.0037 (12)
C10	0.0407 (15)	0.0321 (16)	0.0361 (15)	-0.0031 (12)	0.0053 (12)	-0.0013 (12)
C11	0.0477 (16)	0.0343 (16)	0.0446 (16)	-0.0053 (14)	0.0142 (13)	-0.0095 (12)

*Geometric parameters (Å, °)*

Br1—C11	1.926 (3)	C5—C6	1.362 (4)
F1—C6	1.358 (3)	C6—C7	1.384 (4)
O1—C1	1.373 (3)	C7—C8	1.370 (5)
O1—C9	1.375 (3)	C8—C9	1.379 (4)
O2—C1	1.201 (3)	C10—C11	1.505 (3)
O3—C10	1.209 (3)	C3—H3	0.9300
C1—C2	1.462 (4)	C5—H5	0.9300
C2—C3	1.352 (3)	C7—H7	0.9300
C2—C10	1.494 (3)	C8—H8	0.9300
C3—C4	1.418 (3)	C11—H11A	0.9700
C4—C5	1.404 (3)	C11—H11B	0.9700
C4—C9	1.393 (3)		
Br1...O3	2.9861 (19)	C5...O3 <sup>vi</sup>	3.404 (3)
Br1...C11 <sup>i</sup>	3.747 (3)	C6...C7 <sup>i</sup>	3.569 (4)
Br1...H11A <sup>i</sup>	2.8900	C7...C6 <sup>v</sup>	3.569 (4)
F1...O1 <sup>ii</sup>	3.053 (3)	C8...C5 <sup>v</sup>	3.574 (4)
F1...C8 <sup>ii</sup>	3.226 (4)	C8...F1 <sup>iv</sup>	3.226 (4)
F1...C9 <sup>ii</sup>	3.259 (3)	C9...C4 <sup>v</sup>	3.541 (3)
F1...H8 <sup>iii</sup>	2.8400	C9...F1 <sup>iv</sup>	3.259 (3)
O1...F1 <sup>iv</sup>	3.053 (3)	C10...C1 <sup>i</sup>	3.431 (4)
O2...C11	2.772 (3)	C10...O2 <sup>i</sup>	3.230 (3)
O2...C2 <sup>v</sup>	3.411 (3)	C11...Br1 <sup>v</sup>	3.747 (3)
O2...C10 <sup>v</sup>	3.230 (3)	C11...O2	2.772 (3)
O3...C2 <sup>i</sup>	3.403 (3)	C1...H7 <sup>vii</sup>	3.0600
O3...C3 <sup>vi</sup>	3.296 (3)	C1...H11A	2.8800
O3...C5 <sup>vi</sup>	3.404 (3)	C1...H11B	2.9200
O3...Br1	2.9861 (19)	C11...H11A <sup>i</sup>	3.1000
O1...H7 <sup>vii</sup>	2.7600	H3...O3	2.4300
O2...H7 <sup>vii</sup>	2.5200	H3...H5	2.5500
O2...H11B	2.5500	H3...O3 <sup>vi</sup>	2.4500
O2...H11A	2.4400	H5...H3	2.5500
O2...H11B <sup>v</sup>	2.8500	H5...O3 <sup>vi</sup>	2.6100
O3...H3	2.4300	H7...O1 <sup>iii</sup>	2.7600
O3...H3 <sup>vi</sup>	2.4500	H7...O2 <sup>iii</sup>	2.5200
O3...H5 <sup>vi</sup>	2.6100	H7...C1 <sup>iii</sup>	3.0600

C1...C2 <sup>v</sup>	3.420 (4)	H8...F1 <sup>vii</sup>	2.8400
C1...C10 <sup>v</sup>	3.431 (4)	H11A...Br1 <sup>v</sup>	2.8900
C2...O2 <sup>i</sup>	3.411 (3)	H11A...O2	2.4400
C2...O3 <sup>v</sup>	3.403 (3)	H11A...C1	2.8800
C2...C1 <sup>i</sup>	3.420 (4)	H11A...C11 <sup>v</sup>	3.1000
C3...O3 <sup>vi</sup>	3.296 (3)	H11B...O2	2.5500
C4...C9 <sup>i</sup>	3.541 (3)	H11B...O2 <sup>i</sup>	2.8500
C5...C8 <sup>i</sup>	3.574 (4)	H11B...C1	2.9200
C1—O1—C9	123.43 (19)	C4—C9—C8	122.1 (2)
O1—C1—O2	116.5 (2)	O3—C10—C2	119.5 (2)
O1—C1—C2	116.7 (2)	O3—C10—C11	121.4 (2)
O2—C1—C2	126.9 (2)	C2—C10—C11	119.1 (2)
C1—C2—C3	119.4 (2)	Br1—C11—C10	113.15 (18)
C1—C2—C10	121.8 (2)	C2—C3—H3	119.00
C3—C2—C10	118.8 (2)	C4—C3—H3	119.00
C2—C3—C4	122.5 (2)	C4—C5—H5	121.00
C3—C4—C5	123.9 (2)	C6—C5—H5	121.00
C3—C4—C9	117.6 (2)	C6—C7—H7	120.00
C5—C4—C9	118.4 (2)	C8—C7—H7	120.00
C4—C5—C6	118.2 (2)	C7—C8—H8	120.00
F1—C6—C5	118.8 (2)	C9—C8—H8	121.00
F1—C6—C7	118.0 (3)	Br1—C11—H11A	109.00
C5—C6—C7	123.2 (2)	Br1—C11—H11B	109.00
C6—C7—C8	119.1 (3)	C10—C11—H11A	109.00
C7—C8—C9	119.0 (3)	C10—C11—H11B	109.00
O1—C9—C4	120.2 (2)	H11A—C11—H11B	108.00
O1—C9—C8	117.7 (2)		
C9—O1—C1—O2	-176.2 (2)	C3—C4—C5—C6	-177.3 (3)
C9—O1—C1—C2	2.9 (3)	C9—C4—C5—C6	0.7 (4)
C1—O1—C9—C4	2.0 (3)	C3—C4—C9—O1	-4.2 (3)
C1—O1—C9—C8	-178.5 (2)	C3—C4—C9—C8	176.3 (2)
O1—C1—C2—C3	-5.6 (4)	C5—C4—C9—O1	177.7 (2)
O1—C1—C2—C10	174.2 (2)	C5—C4—C9—C8	-1.8 (4)
O2—C1—C2—C3	173.4 (3)	C4—C5—C6—F1	-179.7 (2)
O2—C1—C2—C10	-6.8 (4)	C4—C5—C6—C7	0.6 (4)
C1—C2—C3—C4	3.6 (4)	F1—C6—C7—C8	179.5 (3)
C10—C2—C3—C4	-176.3 (2)	C5—C6—C7—C8	-0.8 (4)
C1—C2—C10—O3	-178.2 (2)	C6—C7—C8—C9	-0.4 (4)
C1—C2—C10—C11	0.2 (4)	C7—C8—C9—O1	-177.8 (2)
C3—C2—C10—O3	1.7 (4)	C7—C8—C9—C4	1.7 (4)
C3—C2—C10—C11	-179.9 (2)	O3—C10—C11—Br1	-3.2 (3)
C2—C3—C4—C5	179.4 (3)	C2—C10—C11—Br1	178.48 (18)
C2—C3—C4—C9	1.4 (4)		

Symmetry codes: (i)  $x+1, y, z$ ; (ii)  $-x+1/2, y+1/2, -z+1/2$ ; (iii)  $-x-1/2, y+1/2, -z+1/2$ ; (iv)  $-x+1/2, y-1/2, -z+1/2$ ; (v)  $x-1, y, z$ ; (vi)  $-x+1, -y+1, -z$ ; (vii)  $-x-1/2, y-1/2, -z+1/2$ .

*Hydrogen-bond geometry (Å, °)*

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C3—H3···O3 <sup>vi</sup>	0.93	2.45	3.296 (3)	152
C7—H7···O2 <sup>iii</sup>	0.93	2.52	3.425 (3)	164
C11—H11 <i>A</i> ···Br1 <sup>v</sup>	0.97	2.89	3.747 (3)	148

Symmetry codes: (iii)  $-x-1/2, y+1/2, -z+1/2$ ; (v)  $x-1, y, z$ ; (vi)  $-x+1, -y+1, -z$ .