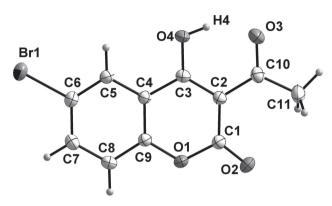
9

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# Crystal structure of 3-acetyl-6-bromo-4-hydroxy-2*H*-chromen-2-one, C<sub>11</sub>H<sub>7</sub>BrO<sub>4</sub>



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#### **Abstract**

 $C_{11}H_7BrO_4$ , triclinic,  $P\bar{1}$  (no. 2),  $\alpha=4.3164(5)$  Å, b=10.7922(12) Å, c=11.1521(11) Å,  $\alpha=98.075(4)^\circ$ ,  $\beta=100.741(4)^\circ$ ,  $\gamma=95.325(5)^\circ$ , V=501.55(9) Å<sup>3</sup>, Z=2,  $R_{\rm gt}(F)=0.0203$ ,  $wR_{\rm ref}(F^2)=0.0544$ , T=200(2) K.

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The crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

## Source of materials

All commercially available starting materials were used without further purification. A mixture of bromosalicylaldehyde (1 mol) and ethylacetoacetate (1 mol) were stirred in 30 mL of ethanol. Then the reaction mixture was cooled and ice-cold water was added. To this mixture 15 g of piperidine was added, in portions, with constant stirring. The mixture was then maintained at 0-5 °C for 3 hours and a white

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Table 1: Data collection and handling.

Crystal:	Rod, colorless	
Size:	$0.52 \times 0.10 \times 0.06~\text{mm}$	
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)	
μ:	$4.09 \; \text{mm}^{-1}$	
Diffractometer, scan mode:	Bruker APEX-II, $oldsymbol{arphi}$ and $\omega$ -scans	
$ heta_{max}$ , completeness:	28.3°, >99%	
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}, R_{\text{int}}$ :	22323, 2483, 0.022	
Criterion for $I_{obs}$ , $N(hkl)_{gt}$ :	$I_{\rm obs} > 2 \ \sigma(I_{\rm obs}), 2286$	
$N(param)_{refined}$ :	147	
Programs:	Bruker programs [1, 2], SHELX	
	[3, 4], PLATON [5], Mercury [6]	

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\mathring{A}^2$ ).

Atom	x	у	Z	$m{U}_{iso}$ * $/m{U}_{eq}$
Br1	0.13721(4)	0.17993(2)	0.01884(2)	0.03753(7)
01	0.7786(3)	0.13535(10)	0.53551(10)	0.0288(2)
02	0.9251(3)	0.20150(12)	0.73466(11)	0.0371(3)
03	0.3131(3)	0.48279(11)	0.70865(11)	0.0357(3)
04	0.1861(3)	0.40602(11)	0.48776(11)	0.0319(2)
H4	0.187571	0.452297	0.555097	0.048*
C1	0.7601(4)	0.21857(15)	0.64011(14)	0.0268(3)
C2	0.5496(3)	0.31471(14)	0.62429(13)	0.0246(3)
C3	0.3821(3)	0.32300(13)	0.50633(14)	0.0242(3)
C4	0.4234(3)	0.23887(14)	0.40000(13)	0.0233(3)
C5	0.2745(4)	0.24839(15)	0.27927(14)	0.0266(3)
H5	0.140138	0.311862	0.264499	0.032*
C6	0.3262(4)	0.16441(15)	0.18292(14)	0.0274(3)
C7	0.5157(4)	0.06842(15)	0.20280(15)	0.0298(3)
H7	0.543009	0.009749	0.134778	0.036*
C8	0.6637(4)	0.05881(15)	0.32171(15)	0.0289(3)
Н8	0.793860	-0.006008	0.336341	0.035*
C9	0.6186(3)	0.14544(14)	0.41908(14)	0.0240(3)
C10	0.5063(4)	0.40395(16)	0.72819(15)	0.0291(3)
C11	0.6796(5)	0.4088(2)	0.85630(16)	0.0421(4)
H11A	0.623638	0.329581	0.885067	0.063*
H11B	0.621464	0.479164	0.909931	0.063*
H11C	0.908764	0.421006	0.859012	0.063*

precipitate separated out which was filtered, washed with ethanol. The solid was recrystallized from water/hexane to obtain colourless rod crystals.

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### **Experimental details**

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation, with U(H) set to 1.2  $U_{eq}(C)$ . The H atoms of the methyl group were allowed to rotate with a fixed angle around the C-C bond to best fit the experimental electron density (HFIX 137 [2]), with  $U_{\rm iso}({\rm H})$  set to 1.5  $U_{\rm eq}({\rm C})$ .

## Comment

Coumarin derivatives constitute the core structure of various natural products and are often the vital pharmacophore that originate in numerous medicinal agents such as antimicrobial, antifungal and antioxidant agents [7, 8]. Special properties of coumarin derivatives are of interest as targets to organic chemists and serve as intermediates in the synthesis of novel biological active compounds. In addition, certain derivatives of coumarins are known to induce apoptosis by cytochrome C release and caspase activation [9]. Recent reports describe some coumarin derivatives such as 7-hydroxy-coumarin [10], 7,8-diacetoxy-4-methylcoumarin and 7,8-diacetoxy-4-methylcoumarin [11, 12] with selective cytotoxicity to cancer cells, which inhibit the growth of lung cancer cells without damaging the growth of normal peripheral blood mononuclear cells.

The title compound exhibits strong intramolecular hydrogen bonding of the  $O-H\cdots O$  type between the hydroxy group and the ketonic O atom [0-0=2.428(2) Å; see the]figure]. The packing of crystal structure is dominated by weak intermolecular interactions. Additional  $\pi$ - $\pi$  stacking interactions between adjacent rings (centroid distance is 3.331(2) Å) further stabilize the crystal.

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