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6-Chloro-3-(2,2-dibromoacetyl)-2*H*-chromen-2-one

Kaviraj Yarbagi,[a,b] Ramanaiah Chennuru,[c] Sudarshan Mahapatra,[c]* B. Venkateswara Rao[b] and Paul Douglas Sanasi[b]

[a]Dr. Reddys Laboratories Ltd, Custom Pharmaceutical Services, Bollaram Road, Miyapur, Hyderabad 500 049, India, [b]Department of Engineering Chemistry, Andhra University, Visakhapatnam 530 003, India, and [c]Dr. Reddys Laboratories Ltd, IPDO, Bachupally, Hyderabad, 500 090, India. *Correspondence e-mail: smahapatra@drreddys.com

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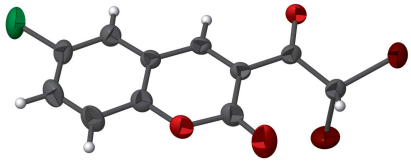
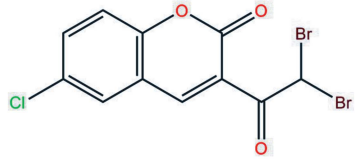
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{11}H_5Br_2ClO_3$, the benzopyran ring system is essentially planar (r.m.s. deviation = 0.023 Å) and one of the bromine atoms is almost coplanar with it [deviation = 0.091 (1) Å]. In the crystal, inversion dimers linked by pairs of double-acceptor $(C-H)_2\cdots O$ hydrogen bonds are seen. Further $C-H\cdots O$ interactions link the dimers into (010) sheets.

3D view



Chemical scheme



Structure description

Two polymorphic forms of 3-acetyl coumarin have been reported in the literature (Munshi *et al.*, 2004; Munshi & Guru Row, 2006). In both cases, weak $C-H\cdots O$ hydrogen bonds are the structure-directing interactions. Halogen-substituted 3-acetyl coumarin derivatives (Chopra *et al.*, 2006, 2007*a*,*b*) have also been described.

In the title compound, the benzopyran ring system is essentially planar (r.m.s. deviation = 0.023 Å) and one of the bromine atoms is almost coplanar with it [deviation = 0.091 (1) Å] (Fig. 1). In the crystal, inversion dimers linked by pairs of double-acceptor $(C-H)_2\cdots O$ hydrogen bonds are seen (Table 1). Further $C-H\cdots O$ interactions link the dimers into (010) sheets.

Synthesis and crystallization

3-Acetyl-6-chloro-2*H*-1-benzopyran-2-one (222 mg, 1 mmol) was dissolved in chloroform (5 ml) and 4.0 ml chloroform containing 347.6 mg bromine was added to it with intermittent shaking and warming. The mixture was heated for 15 min on a water bath, cooled and filtered. The solid was washed with ether and recrystallized from glacial acetic acid solution at room temperature to yield yellow plates of the title compound.

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O3^{i}	0.94 (2)	2.41 (2)	3.238 (3)	147.9 (19)
C5—H5···O3^{i}	0.85 (2)	2.59 (2)	3.299 (3)	142.0 (19)
C11—H11···O2^{ii}	0.94 (2)	2.46 (2)	3.283 (4)	147.0 (17)

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

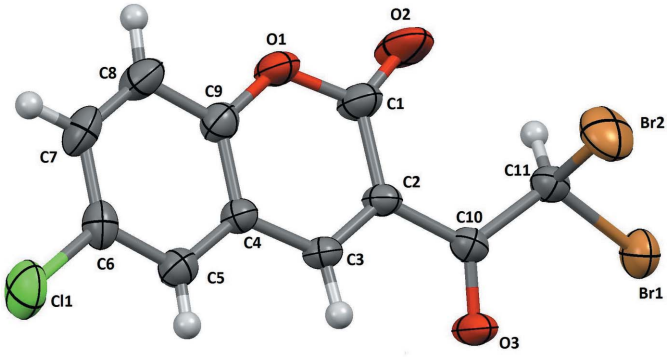


Figure 1
The molecular structure of the title compound, with displacement ellipsoids for non-H atoms drawn at the 50% probability level.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{11}H_5Br_2ClO_3$
M_r	380.42
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (Å)	7.9086 (16), 6.7115 (10), 22.640 (3)
β (°)	96.744 (16)
V (Å^3)	1193.4 (3)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	7.01
Crystal size (mm)	0.51 × 0.15 × 0.09
Data collection	
Diffractometer	Oxford Diffraction Xcalibur, Eos, Nova
Absorption correction	Multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min}, T_{max}	0.124, 0.571
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19365, 4065, 1989
R_{int}	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.765
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.035, 0.074, 0.82
No. of reflections	4065
No. of parameters	174
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.48, −0.86

Computer programs: *CrysAlis PRO* (Oxford Diffraction, 2009), *SHELXL* (Sheldrick, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

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full crystallographic data

IUCrData (2017). **2**, x170356 [https://doi.org/10.1107/S241431461700356X]

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Crystal data

$C_{11}H_5Br_2ClO_3$
M_r = 380.42
Monoclinic, $P2_1/n$
a = 7.9086 (16) Å
b = 6.7115 (10) Å
c = 22.640 (3) Å
β = 96.744 (16)°
V = 1193.4 (3) Å^3
Z = 4
$F(000)$ = 728
D_x = 2.117 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2350 reflections
θ = 3.2–26.0°
μ = 7.01 mm^{-1}
T = 293 K
Plate, metallic light yellow
0.51 × 0.15 × 0.09 mm

Data collection

Oxford Diffraction Xcalibur, Eos, Nova diffractometer
Radiation source: sealed tube, Incoatec Iμs
Graphite monochromator
Detector resolution: 8 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (CrysAlis PRO; Oxford Diffraction, 2009)
T_{min} = 0.124, T_{max} = 0.571
19365 measured reflections
4065 independent reflections
1989 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 33.0°, θ_{min} = 3.2°
h = −11→11
k = −10→10
l = −33→33

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.074
S = 0.82
4065 reflections
174 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.48 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 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 $F^2 > 2\text{sigma}(F^2)$ 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}
Br1	0.40623 (4)	−0.46632 (4)	0.16129 (2)	0.04633 (10)
Br2	0.32549 (5)	−0.02781 (5)	0.20266 (2)	0.06416 (12)
Cl1	0.82997 (11)	0.81238 (11)	−0.03885 (4)	0.0587 (2)
O1	0.8448 (2)	0.3098 (3)	0.17211 (7)	0.0434 (5)
O2	0.7652 (3)	0.0538 (3)	0.22160 (9)	0.0742 (8)
O3	0.4892 (3)	−0.1777 (2)	0.06963 (7)	0.0450 (5)
C1	0.7507 (4)	0.1378 (4)	0.17503 (12)	0.0415 (7)
C2	0.6475 (3)	0.0758 (3)	0.12046 (10)	0.0300 (6)
C3	0.6492 (3)	0.1857 (3)	0.07073 (11)	0.0312 (6)
C4	0.7432 (3)	0.3664 (3)	0.06950 (10)	0.0304 (6)
C5	0.7427 (3)	0.4869 (4)	0.01960 (12)	0.0370 (6)
C6	0.8356 (3)	0.6603 (4)	0.02339 (12)	0.0393 (7)
C7	0.9292 (4)	0.7169 (4)	0.07625 (13)	0.0470 (8)
C8	0.9309 (4)	0.5995 (4)	0.12582 (14)	0.0460 (7)
C9	0.8380 (3)	0.4255 (4)	0.12192 (11)	0.0364 (6)
C10	0.5405 (3)	−0.1082 (4)	0.11729 (11)	0.0321 (6)
C11	0.4948 (4)	−0.2029 (4)	0.17427 (11)	0.0350 (6)
H3	0.578 (3)	0.149 (3)	0.0364 (11)	0.039 (7)*
H5	0.685 (3)	0.460 (3)	−0.0136 (11)	0.037 (8)*
H7	0.992 (3)	0.830 (4)	0.0772 (10)	0.038 (7)*
H8	0.988 (3)	0.629 (3)	0.1616 (11)	0.034 (7)*
H11	0.585 (3)	−0.220 (3)	0.2045 (11)	0.044 (8)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.05146 (19)	0.04012 (15)	0.04839 (17)	−0.00928 (13)	0.00999 (14)	0.00593 (12)
Br2	0.0737 (3)	0.0622 (2)	0.0621 (2)	0.00668 (17)	0.03098 (18)	−0.01290 (16)
Cl1	0.0587 (5)	0.0499 (4)	0.0673 (5)	−0.0148 (4)	0.0059 (4)	0.0200 (4)
O1	0.0493 (13)	0.0465 (10)	0.0314 (10)	−0.0138 (9)	−0.0086 (9)	−0.0023 (8)
O2	0.102 (2)	0.0750 (15)	0.0362 (12)	−0.0414 (14)	−0.0306 (13)	0.0174 (11)
O3	0.0661 (14)	0.0427 (10)	0.0245 (10)	−0.0197 (9)	−0.0024 (9)	−0.0030 (8)
C1	0.0472 (18)	0.0416 (15)	0.0326 (15)	−0.0095 (13)	−0.0077 (13)	−0.0001 (12)
C2	0.0347 (15)	0.0317 (13)	0.0226 (12)	−0.0016 (11)	−0.0011 (11)	−0.0021 (10)
C3	0.0323 (15)	0.0357 (13)	0.0247 (13)	−0.0040 (11)	−0.0005 (11)	−0.0051 (11)
C4	0.0294 (14)	0.0338 (13)	0.0275 (13)	−0.0029 (11)	0.0010 (11)	−0.0032 (10)
C5	0.0346 (15)	0.0396 (15)	0.0359 (15)	−0.0046 (12)	0.0007 (12)	0.0000 (12)
C6	0.0355 (17)	0.0361 (14)	0.0482 (17)	−0.0040 (12)	0.0122 (13)	0.0047 (12)
C7	0.0395 (18)	0.0386 (16)	0.062 (2)	−0.0150 (13)	0.0029 (15)	−0.0036 (14)
C8	0.0425 (19)	0.0425 (15)	0.0498 (19)	−0.0116 (13)	−0.0076 (15)	−0.0057 (14)

C9	0.0321 (15)	0.0381 (14)	0.0384 (15)	−0.0038 (11)	0.0020 (12)	−0.0024 (12)
C10	0.0348 (15)	0.0354 (13)	0.0258 (13)	−0.0017 (11)	0.0018 (11)	0.0013 (11)
C11	0.0386 (17)	0.0380 (14)	0.0278 (13)	−0.0045 (12)	0.0007 (12)	0.0022 (11)

Geometric parameters (Å, º)

Br1—C11	1.912 (2)	C4—C5	1.389 (3)
Br2—C11	1.947 (3)	C4—C9	1.385 (3)
Cl1—C6	1.736 (3)	C5—C6	1.374 (3)
O1—C1	1.379 (3)	C5—H5	0.85 (2)
O1—C9	1.372 (3)	C6—C7	1.385 (4)
O2—C1	1.189 (3)	C7—C8	1.370 (4)
O3—C10	1.202 (3)	C7—H7	0.90 (2)
C1—C2	1.459 (3)	C8—C9	1.377 (4)
C2—C3	1.347 (3)	C8—H8	0.90 (2)
C2—C10	1.494 (3)	C10—C11	1.519 (3)
C3—C4	1.424 (3)	C11—H11	0.93 (3)
C3—H3	0.94 (2)		
C9—O1—C1	122.99 (19)	C6—C7—H7	119.0 (15)
O1—C1—C2	116.7 (2)	C8—C7—C6	120.2 (3)
O2—C1—O1	116.3 (2)	C8—C7—H7	120.8 (15)
O2—C1—C2	127.0 (2)	C7—C8—C9	118.8 (3)
C1—C2—C10	122.3 (2)	C7—C8—H8	124.3 (15)
C3—C2—C1	119.5 (2)	C9—C8—H8	116.9 (15)
C3—C2—C10	118.3 (2)	O1—C9—C4	120.8 (2)
C2—C3—C4	122.6 (2)	O1—C9—C8	117.3 (2)
C2—C3—H3	119.0 (15)	C8—C9—C4	121.9 (3)
C4—C3—H3	118.1 (15)	O3—C10—C2	119.6 (2)
C5—C4—C3	124.1 (2)	O3—C10—C11	120.8 (2)
C9—C4—C3	117.3 (2)	C2—C10—C11	119.7 (2)
C9—C4—C5	118.7 (2)	Br1—C11—Br2	110.81 (14)
C4—C5—H5	122.8 (17)	Br1—C11—H11	103.7 (14)
C6—C5—C4	119.5 (2)	Br2—C11—H11	109.0 (15)
C6—C5—H5	117.7 (17)	C10—C11—Br1	112.07 (17)
C5—C6—Cl1	119.0 (2)	C10—C11—Br2	105.61 (16)
C5—C6—C7	120.9 (2)	C10—C11—H11	115.8 (16)
C7—C6—Cl1	120.0 (2)		
Cl1—C6—C7—C8	178.6 (2)	C3—C2—C10—C11	−162.8 (2)
O1—C1—C2—C3	0.4 (4)	C3—C4—C5—C6	178.8 (3)
O1—C1—C2—C10	179.6 (2)	C3—C4—C9—O1	2.2 (4)
O2—C1—C2—C3	−178.0 (3)	C3—C4—C9—C8	−178.8 (3)
O2—C1—C2—C10	1.2 (5)	C4—C5—C6—Cl1	−178.5 (2)
O3—C10—C11—Br1	15.3 (3)	C4—C5—C6—C7	−0.1 (4)
O3—C10—C11—Br2	−105.4 (2)	C5—C4—C9—O1	−178.9 (2)
C1—O1—C9—C4	−4.5 (4)	C5—C4—C9—C8	0.1 (4)
C1—O1—C9—C8	176.4 (3)	C5—C6—C7—C8	0.2 (4)

C1—C2—C3—C4	−2.6 (4)	C6—C7—C8—C9	−0.1 (4)
C1—C2—C10—O3	−163.2 (3)	C7—C8—C9—O1	179.0 (3)
C1—C2—C10—C11	18.0 (4)	C7—C8—C9—C4	−0.1 (4)
C2—C3—C4—C5	−177.5 (3)	C9—O1—C1—O2	−178.3 (3)
C2—C3—C4—C9	1.4 (4)	C9—O1—C1—C2	3.2 (4)
C2—C10—C11—Br1	−165.94 (19)	C9—C4—C5—C6	0.0 (4)
C2—C10—C11—Br2	73.3 (3)	C10—C2—C3—C4	178.2 (2)
C3—C2—C10—O3	16.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O3^{i}	0.94 (2)	2.41 (2)	3.238 (3)	147.9 (19)
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Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) −*x*+3/2, *y*−1/2, −*z*+1/2.