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3,4-Dihydro-1H-benzo[c]chromene-1,6(2H)-dione

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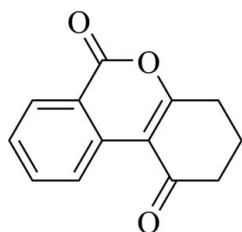
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}—\text{C}) = 0.006$ Å; R factor = 0.077; wR factor = 0.229; data-to-parameter ratio = 12.7.

In the title compound, $\text{C}_{13}\text{H}_{10}\text{O}_3$, the pyranone and benzene rings are almost coplanar, making a dihedral angle of $1.9 (1)^\circ$. The cyclohexenone ring adopts an envelope conformation, with a methylene C atom located at the flap and displaced by $0.639 (3)$ Å from the mean plane of the other five atoms. In the crystal, pairs of weak $\text{C}—\text{H} \cdots \pi$ interactions occur between inversion-related molecules.

Related literature

For applications of benzo[c]chromen-6-ones, see: Schmidt *et al.* (2003); Pandey *et al.* (2004); Matsumoto & Hanawalt (2000); Sun *et al.* (2006). For the synthesis, see: Fan *et al.* (2012).



Experimental

Crystal data

$\text{C}_{13}\text{H}_{10}\text{O}_3$
 $M_r = 214.21$

Monoclinic, $P2_1/c$
 $a = 8.234 (3)$ Å

$b = 10.199 (3)$ Å
 $c = 11.927 (4)$ Å
 $\beta = 97.439 (4)^\circ$
 $V = 993.1 (6)$ Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.10 \text{ mm}^{-1}$
 $T = 296$ K
 $0.47 \times 0.41 \times 0.31$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer
6998 measured reflections

1843 independent reflections
1517 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.077$
 $wR(F^2) = 0.229$
 $S = 1.18$
1843 reflections

145 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.33 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.29 \text{ e } \text{\AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

Cg is the centroid of the C1–C6 benzene ring.

$D—H \cdots A$	$D—H$	$H \cdots A$	$D \cdots A$	$D—H \cdots A$
$\text{C11}—\text{H11B} \cdots \text{Cg}^i$	0.97	2.91	3.723 (5)	142

Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5693).

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supplementary materials

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3,4-Dihydro-1*H*-benzo[*c*]chromene-1,6(2*H*)-dione

Liang-Yan Cui, Yan He and Xue-Sen Fan

Comment

Benzo[*c*]chromen-6-ones constitute one of the major classes of pharmacologically relevant natural products and display a wide range of biological activities (Schmidt *et al.*, 2003; Pandey *et al.*, 2004; Matsumoto *et al.*, 2000; Sun *et al.*, 2006). As part of our research (Fan *et al.* 2012), we have synthesized the title compound (I), and report its crystal structure here.

The title compound, C₁₃H₁₀O₃, consists of three fused six-membered rings, benzene, pyranone and cyclohexenone ring. The pyranone ring is in the middle. All the bond lengths and bond angles are within normal ranges. The pyranone ring and the benzene ring are almost coplanar with a dihedral angle of 1.9 (1) °. The cyclohexenone ring adopts an envelope conformation, a methylene C atom located on the flap and displaced from the mean plane of the other five ring atoms (C8—C11/C13) by 0.639 (3) Å.

In the crystal, weak intermolecular C—H··· π interactions occur between benzene ring and methylene group of adjacent molecules, the separation between the centroid of benzene ring and methylene H atom being 2.91 Å.

Experimental

The title compound was synthesized following the previously reported procedure (Fan *et al.*, 2012). Single crystals, suitable for X-ray diffraction analysis, were obtained by slow evaporation of the solvents from a petroleum ether-ethyl acetate (5:1 *v/v*) solution of the title compound.

Refinement

H atoms were positioned geometrically and refined using riding model with C—H = 0.93–0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Computing details

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

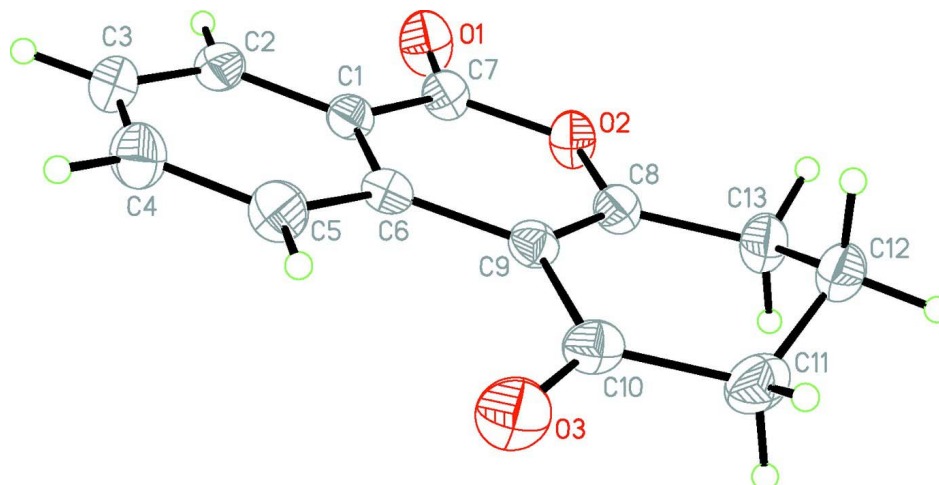


Figure 1

Molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.

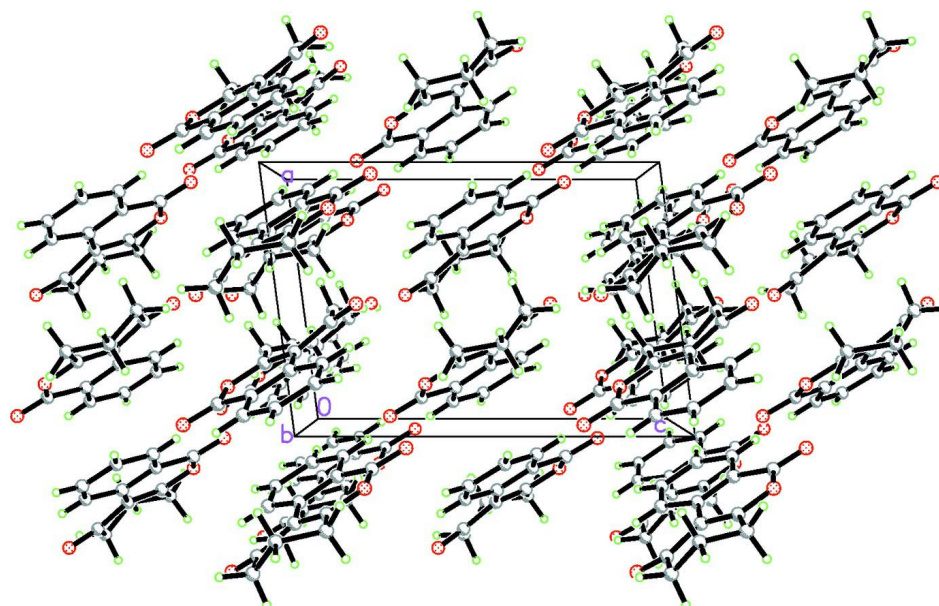


Figure 2

Crystal structure of the title compound with view along the *b* axis.

3,4-Dihydro-1*H*-benzo[*c*]chromene-1,6(2*H*)-dione

Crystal data

$C_{13}H_{10}O_3$

$M_r = 214.21$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1bc$

$a = 8.234\ (3)\ \text{\AA}$

$b = 10.199\ (3)\ \text{\AA}$

$c = 11.927\ (4)\ \text{\AA}$

$\beta = 97.439\ (4)^\circ$

$V = 993.1\ (6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 448$

$D_x = 1.433\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2699 reflections

$\theta = 2.5\text{--}27.2^\circ$

$\mu = 0.10\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Block, colorless

$0.47 \times 0.41 \times 0.31\ \text{mm}$

Data collection

Bruker SMART 1000 CCD area-detector
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 φ and ω scans
 6998 measured reflections
 1843 independent reflections

1517 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\text{max}} = 25.5^\circ$, $\theta_{\text{min}} = 2.5^\circ$
 $h = -9 \rightarrow 9$
 $k = -12 \rightarrow 12$
 $l = -14 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.077$
 $wR(F^2) = 0.229$
 $S = 1.18$
 1843 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
 $w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 2.0297P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.33 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.29 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.1515 (4)	0.4254 (4)	0.4108 (3)	0.0372 (8)
C2	0.0900 (5)	0.2983 (4)	0.3970 (4)	0.0462 (10)
H2	0.0294	0.2737	0.3290	0.055*
C3	0.1189 (5)	0.2095 (4)	0.4836 (4)	0.0556 (11)
H3	0.0797	0.1242	0.4741	0.067*
C4	0.2063 (6)	0.2472 (5)	0.5847 (4)	0.0577 (12)
H4	0.2239	0.1871	0.6437	0.069*
C5	0.2681 (5)	0.3725 (4)	0.6002 (3)	0.0494 (10)
H5	0.3266	0.3958	0.6693	0.059*
C6	0.2435 (4)	0.4652 (4)	0.5127 (3)	0.0361 (8)
C7	0.1173 (5)	0.5166 (4)	0.3171 (3)	0.0409 (9)
C8	0.2628 (4)	0.6829 (4)	0.4336 (3)	0.0388 (9)
C9	0.3044 (4)	0.6001 (4)	0.5209 (3)	0.0364 (8)
C10	0.4169 (5)	0.6504 (4)	0.6187 (3)	0.0448 (10)
C11	0.4572 (6)	0.7945 (5)	0.6214 (4)	0.0575 (12)
H11A	0.4649	0.8249	0.6990	0.069*
H11B	0.5640	0.8061	0.5967	0.069*
C12	0.3362 (5)	0.8789 (4)	0.5496 (4)	0.0522 (11)

H12A	0.3787	0.9675	0.5474	0.063*
H12B	0.2345	0.8823	0.5824	0.063*
C13	0.3036 (6)	0.8247 (4)	0.4302 (4)	0.0517 (11)
H13A	0.2133	0.8719	0.3880	0.062*
H13B	0.3998	0.8366	0.3922	0.062*
O1	0.0424 (4)	0.4948 (3)	0.2258 (2)	0.0618 (9)
O2	0.1742 (3)	0.6438 (3)	0.3351 (2)	0.0457 (7)
O3	0.4831 (4)	0.5785 (3)	0.6928 (2)	0.0663 (10)

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

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0344 (18)	0.038 (2)	0.0388 (19)	0.0033 (15)	0.0045 (14)	−0.0020 (15)
C2	0.039 (2)	0.043 (2)	0.055 (2)	−0.0017 (17)	0.0025 (17)	−0.0056 (18)
C3	0.050 (2)	0.040 (2)	0.078 (3)	−0.0044 (19)	0.010 (2)	0.006 (2)
C4	0.063 (3)	0.051 (3)	0.059 (3)	0.002 (2)	0.009 (2)	0.018 (2)
C5	0.053 (2)	0.052 (2)	0.041 (2)	0.001 (2)	0.0006 (18)	0.0082 (18)
C6	0.0314 (18)	0.041 (2)	0.0354 (18)	0.0056 (15)	0.0039 (14)	0.0009 (15)
C7	0.045 (2)	0.041 (2)	0.0347 (19)	0.0033 (16)	−0.0020 (16)	−0.0043 (16)
C8	0.0409 (19)	0.039 (2)	0.0356 (18)	0.0035 (16)	0.0010 (15)	−0.0034 (15)
C9	0.0349 (18)	0.040 (2)	0.0346 (18)	0.0041 (15)	0.0049 (14)	−0.0041 (15)
C10	0.042 (2)	0.055 (2)	0.037 (2)	−0.0001 (18)	0.0021 (16)	−0.0071 (18)
C11	0.052 (2)	0.064 (3)	0.055 (3)	−0.011 (2)	0.000 (2)	−0.016 (2)
C12	0.052 (2)	0.041 (2)	0.064 (3)	−0.0074 (19)	0.009 (2)	−0.012 (2)
C13	0.061 (3)	0.038 (2)	0.055 (2)	−0.0033 (19)	0.004 (2)	0.0006 (19)
O1	0.081 (2)	0.0545 (19)	0.0429 (16)	0.0025 (16)	−0.0182 (15)	−0.0078 (14)
O2	0.0594 (17)	0.0378 (15)	0.0365 (14)	0.0017 (12)	−0.0069 (12)	−0.0001 (11)
O3	0.074 (2)	0.076 (2)	0.0433 (17)	−0.0031 (18)	−0.0168 (15)	0.0024 (16)

Geometric parameters ($\text{\AA}$, $^\circ$)

C1—C2	1.393 (5)	C8—C9	1.349 (5)
C1—C6	1.406 (5)	C8—O2	1.360 (4)
C1—C7	1.454 (5)	C8—C13	1.486 (5)
C2—C3	1.372 (6)	C9—C10	1.484 (5)
C2—H2	0.9300	C10—O3	1.222 (5)
C3—C4	1.376 (6)	C10—C11	1.506 (6)
C3—H3	0.9300	C11—C12	1.499 (6)
C4—C5	1.379 (6)	C11—H11A	0.9700
C4—H4	0.9300	C11—H11B	0.9700
C5—C6	1.403 (5)	C12—C13	1.519 (6)
C5—H5	0.9300	C12—H12A	0.9700
C6—C9	1.463 (5)	C12—H12B	0.9700
C7—O1	1.201 (4)	C13—H13A	0.9700
C7—O2	1.387 (5)	C13—H13B	0.9700
C2—C1—C6	121.2 (3)	C8—C9—C10	117.4 (3)
C2—C1—C7	118.3 (3)	C6—C9—C10	123.5 (3)
C6—C1—C7	120.6 (3)	O3—C10—C9	122.4 (4)
C3—C2—C1	120.1 (4)	O3—C10—C11	119.6 (4)

C3—C2—H2	120.0	C9—C10—C11	117.9 (4)
C1—C2—H2	120.0	C12—C11—C10	114.8 (3)
C2—C3—C4	119.7 (4)	C12—C11—H11A	108.6
C2—C3—H3	120.2	C10—C11—H11A	108.6
C4—C3—H3	120.2	C12—C11—H11B	108.6
C3—C4—C5	121.2 (4)	C10—C11—H11B	108.6
C3—C4—H4	119.4	H11A—C11—H11B	107.5
C5—C4—H4	119.4	C11—C12—C13	110.5 (4)
C4—C5—C6	120.6 (4)	C11—C12—H12A	109.6
C4—C5—H5	119.7	C13—C12—H12A	109.6
C6—C5—H5	119.7	C11—C12—H12B	109.6
C5—C6—C1	117.2 (4)	C13—C12—H12B	109.6
C5—C6—C9	124.6 (3)	H12A—C12—H12B	108.1
C1—C6—C9	118.1 (3)	C8—C13—C12	110.0 (3)
O1—C7—O2	115.9 (3)	C8—C13—H13A	109.7
O1—C7—C1	127.2 (4)	C12—C13—H13A	109.7
O2—C7—C1	116.9 (3)	C8—C13—H13B	109.7
C9—C8—O2	122.5 (3)	C12—C13—H13B	109.7
C9—C8—C13	126.5 (3)	H13A—C13—H13B	108.2
O2—C8—C13	111.0 (3)	C8—O2—C7	122.7 (3)
C8—C9—C6	119.1 (3)		
C6—C1—C2—C3	0.0 (6)	C5—C6—C9—C8	−175.2 (4)
C7—C1—C2—C3	−179.7 (4)	C1—C6—C9—C8	3.7 (5)
C1—C2—C3—C4	1.1 (6)	C5—C6—C9—C10	7.6 (6)
C2—C3—C4—C5	−1.1 (7)	C1—C6—C9—C10	−173.5 (3)
C3—C4—C5—C6	−0.1 (7)	C8—C9—C10—O3	−167.7 (4)
C4—C5—C6—C1	1.2 (6)	C6—C9—C10—O3	9.5 (6)
C4—C5—C6—C9	−179.8 (4)	C8—C9—C10—C11	8.5 (5)
C2—C1—C6—C5	−1.1 (5)	C6—C9—C10—C11	−174.2 (3)
C7—C1—C6—C5	178.5 (3)	O3—C10—C11—C12	−162.4 (4)
C2—C1—C6—C9	179.8 (3)	C9—C10—C11—C12	21.2 (6)
C7—C1—C6—C9	−0.5 (5)	C10—C11—C12—C13	−51.1 (5)
C2—C1—C7—O1	−1.2 (6)	C9—C8—C13—C12	−23.3 (6)
C6—C1—C7—O1	179.1 (4)	O2—C8—C13—C12	156.4 (3)
C2—C1—C7—O2	177.4 (3)	C11—C12—C13—C8	50.7 (5)
C6—C1—C7—O2	−2.3 (5)	C9—C8—O2—C7	1.1 (6)
O2—C8—C9—C6	−4.1 (5)	C13—C8—O2—C7	−178.6 (3)
C13—C8—C9—C6	175.5 (4)	O1—C7—O2—C8	−179.1 (4)
O2—C8—C9—C10	173.2 (3)	C1—C7—O2—C8	2.2 (5)
C13—C8—C9—C10	−7.1 (6)		

Hydrogen-bond geometry ($\text{\AA}$, $^\circ$)

Cg is the centroid of the C1–C6 benzene ring.

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
C11—H11B $\cdots$ Cg ⁱ	0.97	2.91	3.723 (5)	142

Symmetry code: (i) $-x+1, -y+1, -z+1$.