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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***

School of Chemistry and Chemical Engineering, Henan Normal University, Henan 453007, People's Republic of China
Correspondence e-mail: xuesen.fan@htu.cn

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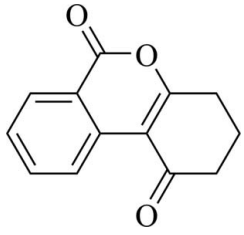
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.077; $wR$ factor = 0.229; data-to-parameter ratio = 12.7.

In the title compound, $C_{13}H_{10}O_3$, the pyranone and benzene rings are almost coplanar, making a dihedral angle of 1.9 (1)°. 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 C—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

$C_{13}H_{10}O_3$
$M_r$ = 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)°
$V$ = 993.1 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 296 K
0.47 × 0.41 × 0.31 mm

### Data collection

Bruker SMART 1000 CCD area-detector diffractometer
6998 measured reflections
1843 independent reflections
1517 reflections with $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.29 e Å$^{-3}$

**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$\cdots A$ |
|---|---|---|---|---|
| C11—H11$B\cdots Cg^{i}$ | 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).

## References

Bruker (2007). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fan, X.-S., He, Y., Cui, L.-Y., Guo, S.-H., Wang, J.-J. & Zhang, X.-Y. (2012). *Eur. J. Org. Chem.* pp. 673–677.
Matsumoto, A. & Hanawalt, P. C. (2000). *Cancer Res.* **60**, 3921–3926.
Pandey, J., Jha, A. K. & Hajela, K. (2004). *Bioorg. Med. Chem.* **12**, 2239–2249.
Schmidt, J. M., Tremblay, G. B., Pagé, M., Mercure, J., Feher, M., Dunn-Dufult, R., Peter, M. G. & Redden, P. R. (2003). *J. Med. Chem.* **46**, 1289–1292.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sun, W., Cama, L. D., Birzin, E. T., Warrier, S., Locco, L., Mosley, R., Hammond, M. L. & Rohrer, S. P. (2006). *Bioorg. Med. Chem. Lett.* **16**, 1468–1472.

# supporting information

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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**

### S1. 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, $C_{13}H_{10}O_3$, 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···$\pi$ 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.

### S2. 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.

### S3. Refinement

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

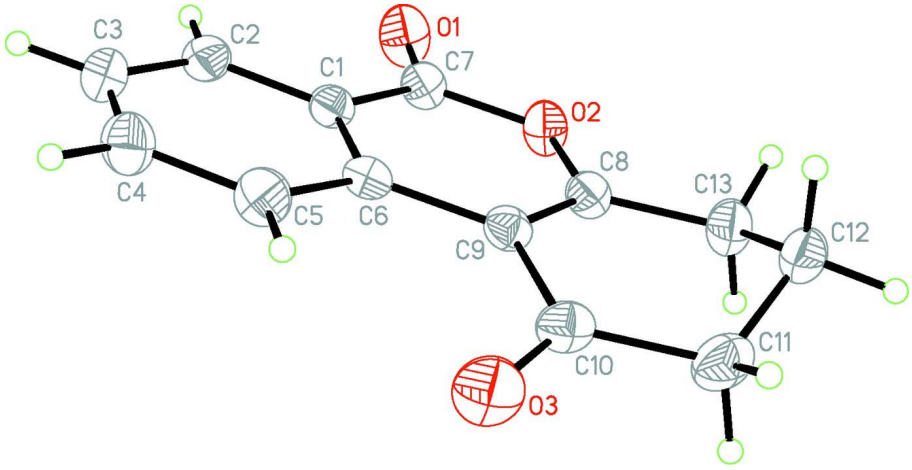


**Figure 1**

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

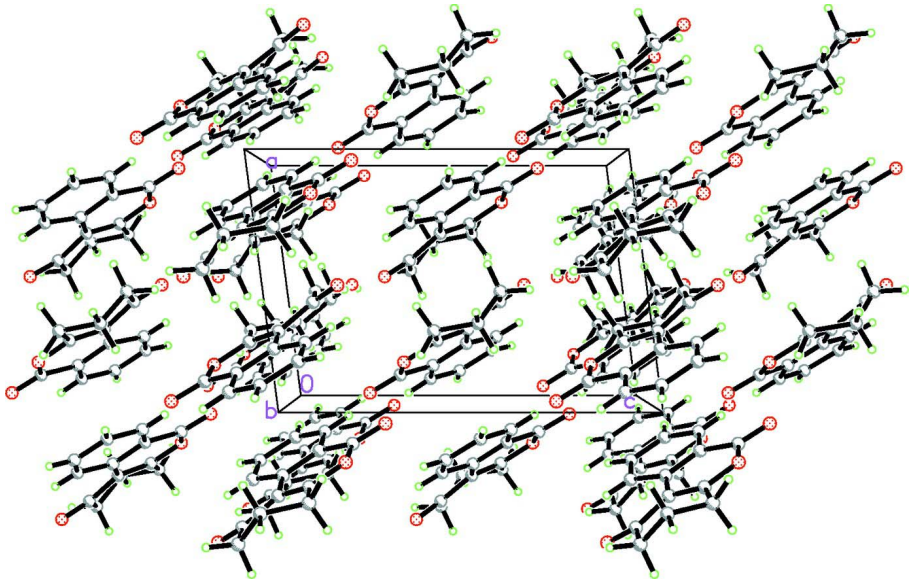


**Figure 2**

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 2ybc
$a = 8.234\ (3)$ Å
$b = 10.199\ (3)$ Å
$c = 11.927\ (4)$ Å
$\beta = 97.439\ (4)°$
$V = 993.1\ (6)$ Å$^3$
$Z = 4$

$F(000) = 448$
$D_x = 1.433$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2699 reflections
$\theta = 2.5–27.2°$
$\mu = 0.10$ mm$^{-1}$
$T = 296$ K
Block, colorless
0.47 × 0.41 × 0.31 mm

*Data collection*

Bruker SMART 1000 CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
6998 measured reflections
1843 independent reflections
1517 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.5°$
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

*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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å$^{-3}$
$\Delta\rho_{min} = -0.29$ e Å$^{-3}$

*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 (Å²)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 (Å, º)*

| | | | |
|---|---|---|---|
| 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 (Å, º)*

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C11—H11*B*···*Cg*$^{i}$ | 0.97 | 2.91 | 3.723 (5) | 142 |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.