Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-[Hydroxy(4-methoxyphenyl)methylidene]indane-1,3-dione

Jing-Chi Gu, Bi-Xue Zhu* and Yun-Qian Zhang

Key Laboratory of Macrocyclic and Supramolecular Chemistry of Guizhou Province, Guizhou University, Guiyang 550025, People's Republic of China.
Correspondence e-mail: gzubxzhu@126.com

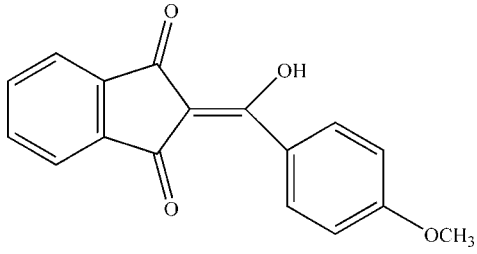
Received 23 April 2012; accepted 14 May 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.127; data-to-parameter ratio = 12.2.

In the title compound, $C_{17}H_{12}O_4$, there is an intramolecular O—H···O hydrogen bond. The dihedral angle between the indane ring system [maximun deviation = 0.023 (2) Å] and the benzene ring is 37.42 (9)°.

Related literature

For general background to the synthesis and pharmacological properties of 1,3-indandione derivatives, see: Cheng *et al.* (2011).



Experimental

Crystal data

$C_{17}H_{12}O_4$
M_r = 280.27
Monoclinic, $P2_1/n$
a = 17.779 (4) Å
b = 3.8405 (9) Å
c = 19.026 (4) Å
β = 92.984 (8)°
V = 1297.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.23 × 0.19 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.977, T_{max} = 0.982
10521 measured reflections
2311 independent reflections
1553 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.127
S = 0.97
2311 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O2	0.82	1.78	2.539 (2)	152

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported financially by the Natural Science Foundation of China (No. 21061003) and the International Cooperation Foundation of Guizhou Province (No. 2009700104).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2275).

References

Bruker (2002). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cheng, C., Jiang, B., Tu, S.-J. & Li, G.-G. (2011). *Green Chem.* **13**, 2107–2115.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2012). E**68**, o1907 [doi:10.1107/S1600536812021824]

2-[Hydroxy(4-methoxyphenyl)methylidene]indane-1,3-dione

Jing-Chi Gu, Bi-Xue Zhu and Yun-Qian Zhang

Comment

In general, 1,3-indandione derivatives demonstrate an anticoagulant properties. The synthesis and pharmacological properties of some chemicals of this category have been reported (Dolmella *et al.*, 1961). The prepare of derivatives containing 2*H*-indene-1,3-dione unit have received very substantial attention (Cheng *et al.*, 2011). In view of this biological importance a part of our ongoing studies of 1,3-indandione derivatives includes the crystal structure determination of the title compound. The molecule of the title compound shows non-coplanar structure (Fig. 1). An intramolecular O—H···O hydrogen bonds is observed (Table 1), which links the hydroxyl oxygen to the nearby keto-oxygen atom of the 2*H*-indene-1,3-dione unit, forming a planar six-membered ring. The dihedral angle between the six-membered ring and the plane of 2*H*-indene-1,3-dione unit is 173.94°, and the dihedral angle between the six-membered ring and the benzene ring is 148.51°.

Experimental

p-methoxy-acetophenone (166 mg, 1.2 mmol) in tetrahydrofuran (15 ml) was added slowly with stirring to dimethyl phthalate(232.8 mg, 1.2 mmol) and NaH (120 mg, 5 mmol) in THF (30 ml) and the mixture was heated at reflux for 12 h. The solution was allowed to cool and the THF was removed partly under reduced pressure. The precipitate was collected by filtration and washed with waterand dried; the residue was crystallized from $CHCl_3$ to afford the title compound as a yellow solid [yield 65%, m.p. 398–400 K]. Single crystal suitable for X-ray diffraction was prepared by slow evaporation of a solution of the title compound in methanol at room temperature.

Refinement

All H atoms were geometrically positioned(C—H = 0.93–0.96 Å) and treated as riding, with U_{iso}(H) = 1.2–1.5 U_{eq}.

Computing details

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

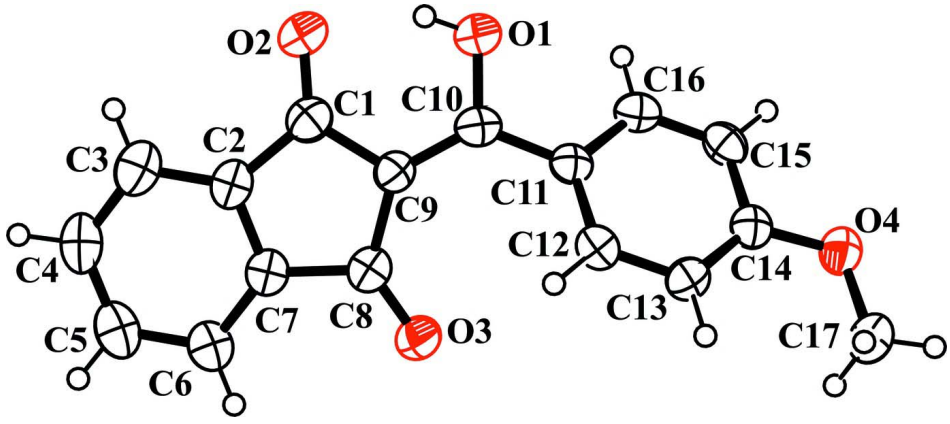


Figure 1

The molecular structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2-[Hydroxy(4-methoxyphenyl)methylidene]indane-1,3-dione

Crystal data

$C_{17}H_{12}O_4$
$M_r = 280.27$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 17.779$ (4) Å
$b = 3.8405$ (9) Å
$c = 19.026$ (4) Å
$\beta = 92.984$ (8)°
$V = 1297.4$ (5) Å^3
$Z = 4$
$F(000) = 584$
$D_x = 1.435$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2311 reflections
$\theta = 1.5$–25.1°
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, colorless
0.23 × 0.19 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min} = 0.977$, $T_{max} = 0.982$
10521 measured reflections
2311 independent reflections
1553 reflections with $I > 2\sigma(I)$
$R_{int} = 0.077$
$\theta_{max} = 25.1°$, $\theta_{min} = 1.5°$
$h = -21 \rightarrow 19$
$k = -4 \rightarrow 4$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.127$
$S = 0.97$
2311 reflections
191 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
$w = 1/[\sigma^2(F_o^2) + (0.0678P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ 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}
C1	0.51739 (13)	0.1113 (6)	0.36369 (11)	0.0431 (6)
C2	0.44114 (13)	0.2488 (6)	0.37373 (11)	0.0407 (6)
C3	0.40844 (16)	0.3680 (6)	0.43398 (12)	0.0551 (7)
H3	0.4347	0.3647	0.4775	0.066*
C4	0.33527 (16)	0.4919 (7)	0.42678 (14)	0.0561 (7)
H4	0.3119	0.5734	0.4662	0.067*
C5	0.29666 (15)	0.4961 (6)	0.36209 (13)	0.0513 (7)
H5	0.2477	0.5817	0.3587	0.062*
C6	0.32898 (13)	0.3762 (6)	0.30215 (12)	0.0448 (6)
H6	0.3026	0.3810	0.2586	0.054*
C7	0.40147 (12)	0.2492 (5)	0.30887 (10)	0.0370 (5)
C8	0.44965 (13)	0.1018 (5)	0.25401 (11)	0.0366 (5)
C9	0.52302 (12)	0.0199 (5)	0.28999 (10)	0.0353 (5)
C10	0.59134 (12)	−0.0856 (5)	0.26557 (11)	0.0374 (6)
C11	0.61151 (12)	−0.1687 (5)	0.19378 (10)	0.0338 (5)
C12	0.56158 (12)	−0.3235 (5)	0.14478 (11)	0.0375 (5)
H12	0.5125	−0.3693	0.1568	0.045*
C13	0.58354 (13)	−0.4110 (5)	0.07820 (11)	0.0378 (6)
H13	0.5495	−0.5174	0.0462	0.045*
C14	0.65592 (12)	−0.3404 (5)	0.05943 (10)	0.0363 (5)
C15	0.70668 (12)	−0.1831 (6)	0.10769 (11)	0.0401 (6)
H15	0.7552	−0.1315	0.0949	0.048*
C16	0.68528 (12)	−0.1036 (5)	0.17427 (11)	0.0391 (6)
H16	0.7201	−0.0057	0.2067	0.047*
C17	0.63265 (14)	−0.5648 (7)	−0.05674 (12)	0.0503 (7)
H17A	0.6587	−0.6058	−0.0989	0.075*
H17B	0.5915	−0.4073	−0.0667	0.075*
H17C	0.6136	−0.7813	−0.0398	0.075*
O1	0.65026 (9)	−0.1081 (5)	0.31159 (8)	0.0537 (5)
H1	0.6368	−0.0636	0.3512	0.081*
O2	0.56922 (10)	0.0915 (5)	0.40994 (8)	0.0609 (5)
O3	0.42812 (9)	0.0644 (4)	0.19222 (8)	0.0477 (5)
O4	0.68340 (9)	−0.4164 (4)	−0.00444 (7)	0.0470 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0489 (15)	0.0397 (14)	0.0407 (13)	−0.0010 (11)	0.0010 (12)	0.0020 (10)
C2	0.0521 (15)	0.0316 (13)	0.0389 (12)	−0.0033 (11)	0.0086 (11)	0.0000 (10)
C3	0.0693 (19)	0.0524 (16)	0.0443 (14)	−0.0007 (14)	0.0100 (13)	0.0003 (12)
C4	0.0665 (19)	0.0485 (16)	0.0553 (16)	0.0035 (14)	0.0233 (14)	−0.0022 (12)
C5	0.0532 (16)	0.0361 (14)	0.0661 (18)	0.0039 (12)	0.0179 (14)	0.0051 (12)
C6	0.0484 (15)	0.0346 (14)	0.0520 (14)	0.0016 (11)	0.0074 (12)	0.0033 (10)
C7	0.0459 (15)	0.0246 (12)	0.0407 (12)	−0.0029 (10)	0.0057 (11)	0.0026 (9)
C8	0.0479 (14)	0.0245 (12)	0.0376 (13)	−0.0026 (10)	0.0037 (11)	0.0050 (9)
C9	0.0403 (14)	0.0312 (12)	0.0344 (12)	0.0012 (10)	0.0010 (10)	−0.0003 (9)
C10	0.0414 (14)	0.0291 (12)	0.0410 (12)	−0.0007 (10)	−0.0053 (11)	0.0015 (9)
C11	0.0369 (13)	0.0255 (11)	0.0389 (12)	0.0034 (10)	−0.0010 (10)	0.0034 (9)
C12	0.0366 (13)	0.0303 (12)	0.0459 (13)	−0.0029 (10)	0.0048 (11)	0.0022 (10)
C13	0.0431 (14)	0.0307 (12)	0.0393 (12)	−0.0037 (10)	0.0000 (11)	−0.0017 (9)
C14	0.0418 (14)	0.0281 (12)	0.0390 (12)	0.0042 (10)	0.0035 (11)	0.0043 (9)
C15	0.0336 (13)	0.0383 (13)	0.0488 (13)	−0.0013 (10)	0.0052 (11)	0.0060 (10)
C16	0.0379 (13)	0.0350 (13)	0.0438 (13)	−0.0011 (10)	−0.0045 (11)	0.0022 (10)
C17	0.0592 (16)	0.0494 (16)	0.0423 (13)	−0.0001 (12)	0.0042 (12)	−0.0057 (11)
O1	0.0471 (10)	0.0711 (13)	0.0420 (9)	0.0095 (9)	−0.0056 (8)	−0.0058 (8)
O2	0.0582 (12)	0.0835 (14)	0.0400 (9)	0.0096 (10)	−0.0059 (9)	−0.0056 (8)
O3	0.0501 (10)	0.0543 (11)	0.0384 (9)	0.0066 (8)	−0.0017 (8)	−0.0009 (7)
O4	0.0474 (10)	0.0535 (11)	0.0406 (9)	0.0002 (8)	0.0073 (8)	−0.0030 (7)

Geometric parameters (Å, º)

C1—O2	1.243 (3)	C10—C11	1.465 (3)
C1—C9	1.454 (3)	C11—C12	1.387 (3)
C1—C2	1.476 (3)	C11—C16	1.404 (3)
C2—C7	1.389 (3)	C12—C13	1.386 (3)
C2—C3	1.390 (3)	C12—H12	0.9300
C3—C4	1.385 (4)	C13—C14	1.380 (3)
C3—H3	0.9300	C13—H13	0.9300
C4—C5	1.378 (3)	C14—O4	1.365 (2)
C4—H4	0.9300	C14—C15	1.392 (3)
C5—C6	1.382 (3)	C15—C16	1.376 (3)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.378 (3)	C16—H16	0.9300
C6—H6	0.9300	C17—O4	1.427 (3)
C7—C8	1.496 (3)	C17—H17A	0.9600
C8—O3	1.226 (2)	C17—H17B	0.9600
C8—C9	1.475 (3)	C17—H17C	0.9600
C9—C10	1.384 (3)	O1—H1	0.8200
C10—O1	1.333 (2)		
O2—C1—C9	125.6 (2)	O1—C10—C11	112.11 (19)
O2—C1—C2	125.6 (2)	C9—C10—C11	129.68 (19)
C9—C1—C2	108.71 (19)	C12—C11—C16	118.2 (2)
C7—C2—C3	121.2 (2)	C12—C11—C10	122.7 (2)

C7—C2—C1	108.22 (19)	C16—C11—C10	119.01 (19)
C3—C2—C1	130.6 (2)	C13—C12—C11	121.1 (2)
C4—C3—C2	117.6 (2)	C13—C12—H12	119.4
C4—C3—H3	121.2	C11—C12—H12	119.4
C2—C3—H3	121.2	C14—C13—C12	120.0 (2)
C5—C4—C3	120.9 (2)	C14—C13—H13	120.0
C5—C4—H4	119.5	C12—C13—H13	120.0
C3—C4—H4	119.5	O4—C14—C13	124.76 (19)
C4—C5—C6	121.6 (2)	O4—C14—C15	115.50 (19)
C4—C5—H5	119.2	C13—C14—C15	119.7 (2)
C6—C5—H5	119.2	C16—C15—C14	120.2 (2)
C7—C6—C5	117.9 (2)	C16—C15—H15	119.9
C7—C6—H6	121.0	C14—C15—H15	119.9
C5—C6—H6	121.0	C15—C16—C11	120.7 (2)
C6—C7—C2	120.8 (2)	C15—C16—H16	119.6
C6—C7—C8	129.5 (2)	C11—C16—H16	119.6
C2—C7—C8	109.7 (2)	O4—C17—H17A	109.5
O3—C8—C9	130.1 (2)	O4—C17—H17B	109.5
O3—C8—C7	123.5 (2)	H17A—C17—H17B	109.5
C9—C8—C7	106.35 (17)	O4—C17—H17C	109.5
C10—C9—C1	120.02 (19)	H17A—C17—H17C	109.5
C10—C9—C8	132.62 (19)	H17B—C17—H17C	109.5
C1—C9—C8	107.00 (19)	C10—O1—H1	109.5
O1—C10—C9	118.17 (18)	C14—O4—C17	117.63 (17)

O2—C1—C2—C7	176.0 (2)	C7—C8—C9—C10	−172.1 (2)
C9—C1—C2—C7	−1.1 (2)	O3—C8—C9—C1	−179.1 (2)
O2—C1—C2—C3	−3.3 (4)	C7—C8—C9—C1	0.9 (2)
C9—C1—C2—C3	179.6 (2)	C1—C9—C10—O1	1.6 (3)
C7—C2—C3—C4	−0.9 (4)	C8—C9—C10—O1	173.8 (2)
C1—C2—C3—C4	178.3 (2)	C1—C9—C10—C11	−175.8 (2)
C2—C3—C4—C5	0.0 (4)	C8—C9—C10—C11	−3.6 (4)
C3—C4—C5—C6	0.3 (4)	O1—C10—C11—C12	148.4 (2)
C4—C5—C6—C7	0.3 (3)	C9—C10—C11—C12	−34.1 (3)
C5—C6—C7—C2	−1.3 (3)	O1—C10—C11—C16	−28.8 (3)
C5—C6—C7—C8	179.4 (2)	C9—C10—C11—C16	148.8 (2)
C3—C2—C7—C6	1.6 (3)	C16—C11—C12—C13	−0.2 (3)
C1—C2—C7—C6	−177.80 (19)	C10—C11—C12—C13	−177.40 (18)
C3—C2—C7—C8	−179.0 (2)	C11—C12—C13—C14	−0.8 (3)
C1—C2—C7—C8	1.6 (2)	C12—C13—C14—O4	179.78 (18)
C6—C7—C8—O3	−2.3 (4)	C12—C13—C14—C15	0.4 (3)
C2—C7—C8—O3	178.4 (2)	O4—C14—C15—C16	−178.35 (18)
C6—C7—C8—C9	177.8 (2)	C13—C14—C15—C16	1.1 (3)
C2—C7—C8—C9	−1.6 (2)	C14—C15—C16—C11	−2.2 (3)
O2—C1—C9—C10	−3.0 (3)	C12—C11—C16—C15	1.7 (3)
C2—C1—C9—C10	174.07 (19)	C10—C11—C16—C15	179.00 (18)
O2—C1—C9—C8	−177.0 (2)	C13—C14—O4—C17	3.1 (3)
C2—C1—C9—C8	0.1 (2)	C15—C14—O4—C17	−177.49 (19)
O3—C8—C9—C10	8.0 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2	0.82	1.78	2.539 (2)	152