Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 5-(1*H*-Inden-2-yl)-1,3-benzodioxole

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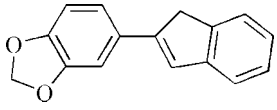
Received 17 November 2007; accepted 15 January 2008

Key indicators: single-crystal X-ray study; $T$ = 294 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.033; $wR$ factor = 0.092; data-to-parameter ratio = 7.8.

In the title compound, $C_{16}H_{12}O_2$, the non-H atoms are coplanar with a mean r.m.s. deviation of 0.0260 (2) Å. The deviations of the bond angles from normal values at the indenyl junction C atom and the indenyl bridgehead C atom nearest the junction are imposed by the five-membered ring geometry. Due to conjugation, the single bond linking the two ring systems [1.455 (3) Å] is significantly shorter than the formal single bonds in the five-membered carbocyclic ring [1.500 (3) and 1.489 (3) Å].

## Related literature

For related literature, see: Rayabarapu *et al.* (2003); Senanayake *et al.* (1995).



## Experimental

### Crystal data

$C_{16}H_{12}O_2$
$M_r$ = 236.26
Orthorhombic, $Pca2_1$
$a$ = 22.277 (10) Å
$b$ = 6.892 (3) Å
$c$ = 7.580 (3) Å
$V$ = 1163.7 (9) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 294 (2) K
0.24 × 0.22 × 0.12 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.979, $T_{max}$ = 0.990
6325 measured reflections
1286 independent reflections
1072 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.035

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.091
$S$ = 1.08
1286 reflections
164 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.11 e Å$^{-3}$

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2152).

## References

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  doi:10.1107/S1600536808001578 

# supporting information

*Acta Cryst.* (2008). E**64**, o492 [doi:10.1107/S1600536808001578]

## 5-(1*H*-Inden-2-yl)-1,3-benzodioxole

**Rui-Xue Deng, Wei-Yi Zhou, Xiao-Juan Deng and Liang-Dong Sun**

### S1. Comment

Indene ring frameworks are present in a large number of biologically active compounds, and their metallocene complexes are able to catalyze olefin polymerization (Senanayake *et al.*, 1995; Rayabarapu *et al.*, 2003). Some derivatives have shown analgesic and myorelaxation activity whereas others are used as valuable intermediates for the synthesis of indenyl chrysanthemates that possess insecticidal properties. In the recent three decades, many chemists have been attracted by the synthesis of indenes. In this context, we report the synthesis and crystal structure of the title compound, (I). The molecul of (I) (Fig. 1) is almost planar (except the H atoms) with the mean value of r.m.s. deviation of 0.0260 (2) Å. The bonding angles of C16—C8—C5 and C16—C8—C9 are 128.3 (2) and 108.88 (19)°, respectively; their deviations from ideal values are imposed by request of a five-ring geometry. The similar deviation is also observed for the C15 with the angles of C14—C15—C16 [132.2 (2)°] and C10—C15—C16 [108.1 (2)°]. Due to the $\pi$-$\pi$ conjugation of the C5?C6 and C8?C16, the single bond distance of the C5—C8 [1.455 (3) Å] is significantly shorter than that of C8—C9 [1.500 (3) Å].

### S2. Experimental

*o*-Bromobenzyl zinc bromide (3.5 mmol, 3.5 equiv) in 3.5 ml $CH_2Cl_2$ was added to a degassed refluxing $CH_2Cl_2$ solution (8 ml) of 5-ethynyl benzo [1,3] dioxole (1.0 mmol, 1.0 equiv) and $Ni(PPh_3)_2I_2$ (0.1 mmol, 0.1 equiv). After stirring at 313 K for 6 h, the solution was cooled to room temperature. The solution obtained was diluted with 50 ml e thyl acetate. The organic layer was washed with 10 ml aqueous HCl solution, saturated by NaCl. The aqueous layer was back-extracted with ethyl acetate. The combined organic layer was dried over anhydrous $Na_2SO_4$. After filtration, the solvent was removed under reduced pressure and the residue was purified *via* flash chromatography ($SiO_2$) to afford the compound. Crystals suitale for X-ray analysis were obtained by slow evaporation from a $CH_2Cl_2$ solution at 298 K.

### S3. Refinement

All H atoms were positioned geometrically and refined as riding (C—H = 0.93 and 0.97%A) and allowed to ride on their parent atoms, with $U_{iso}$(H) = 1.2$U_{eq}$(parent). In the absence of significant anomalous scattering effects the Friedel pairs were merged.

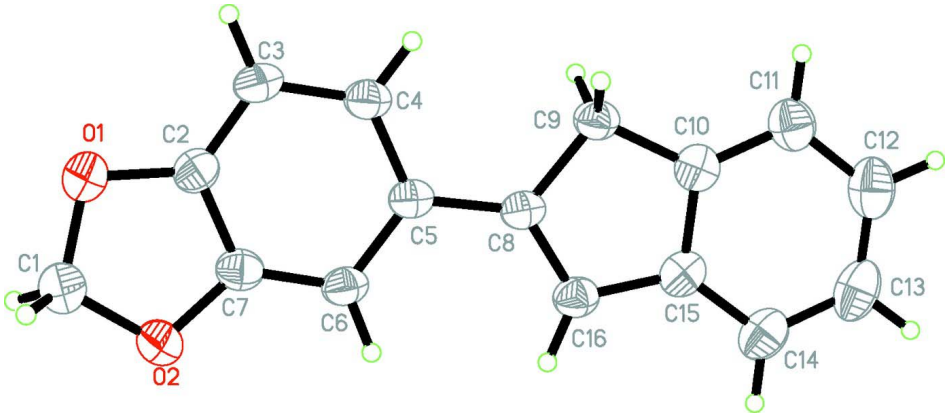


**Figure 1**

View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

### 5-(1*H*-Inden-2-yl)-1,3-benzodioxole

*Crystal data*

$C_{16}H_{12}O_2$
$M_r = 236.26$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac
$a = 22.277\ (10)$ Å
$b = 6.892\ (3)$ Å
$c = 7.580\ (3)$ Å
$V = 1163.7\ (9)$ Å$^3$
$Z = 4$
$F(000) = 496$
$D_x = 1.348$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2528 reflections
$\theta = 2.7–25.2°$
$\mu = 0.09$ mm$^{-1}$
$T = 294$ K
Plate, colourless
0.24 × 0.22 × 0.12 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.979$, $T_{max} = 0.990$
6325 measured reflections
1286 independent reflections
1072 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 26.4°$, $\theta_{min} = 1.8°$
$h = -27\rightarrow13$
$k = -8\rightarrow8$
$l = -8\rightarrow9$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.091$
$S = 1.08$
1286 reflections
164 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2 + 0.0272P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å$^{-3}$
$\Delta\rho_{min} = -0.11$ e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.006 (2)
Absolute structure: indeterminate

*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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.33678 (7) | 0.3967 (2) | 0.9508 (3) | 0.0629 (5) |
| O2 | 0.41993 (8) | 0.2623 (2) | 1.0832 (3) | 0.0676 (6) |
| C1 | 0.35772 (11) | 0.2393 (4) | 1.0539 (5) | 0.0686 (8) |
| H1A | 0.3366 | 0.2362 | 1.1658 | 0.082* |
| H1B | 0.3502 | 0.1180 | 0.9929 | 0.082* |
| C2 | 0.38388 (9) | 0.5252 (3) | 0.9457 (3) | 0.0472 (5) |
| C3 | 0.38568 (10) | 0.7034 (3) | 0.8718 (4) | 0.0512 (6) |
| H3 | 0.3523 | 0.7568 | 0.8162 | 0.061* |
| C4 | 0.43988 (10) | 0.8035 (3) | 0.8829 (3) | 0.0480 (5) |
| H4 | 0.4425 | 0.9262 | 0.8326 | 0.058* |
| C5 | 0.49008 (9) | 0.7272 (3) | 0.9662 (3) | 0.0415 (5) |
| C6 | 0.48641 (10) | 0.5410 (3) | 1.0401 (3) | 0.0468 (5) |
| H6 | 0.5192 | 0.4849 | 1.0966 | 0.056* |
| C7 | 0.43337 (10) | 0.4460 (3) | 1.0263 (3) | 0.0472 (5) |
| C8 | 0.54537 (9) | 0.8392 (3) | 0.9756 (3) | 0.0417 (5) |
| C9 | 0.55116 (10) | 1.0396 (3) | 0.9004 (3) | 0.0482 (5) |
| H9A | 0.5435 | 1.0394 | 0.7744 | 0.058* |
| H9B | 0.5234 | 1.1284 | 0.9570 | 0.058* |
| C10 | 0.61447 (10) | 1.0948 (3) | 0.9384 (3) | 0.0488 (6) |
| C11 | 0.64540 (11) | 1.2630 (4) | 0.9035 (4) | 0.0606 (7) |
| H11 | 0.6271 | 1.3649 | 0.8438 | 0.073* |
| C12 | 0.70444 (12) | 1.2778 (4) | 0.9592 (4) | 0.0703 (8) |
| H12 | 0.7259 | 1.3910 | 0.9372 | 0.084* |
| C13 | 0.73163 (11) | 1.1275 (4) | 1.0462 (4) | 0.0692 (8) |
| H13 | 0.7714 | 1.1399 | 1.0822 | 0.083* |
| C14 | 0.70099 (10) | 0.9578 (4) | 1.0814 (4) | 0.0611 (7) |
| H14 | 0.7198 | 0.8564 | 1.1405 | 0.073* |
| C15 | 0.64208 (10) | 0.9406 (3) | 1.0277 (3) | 0.0489 (6) |
| C16 | 0.59838 (10) | 0.7872 (3) | 1.0480 (3) | 0.0483 (5) |
| H16 | 0.6057 | 0.6691 | 1.1032 | 0.058* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0482 (8) | 0.0638 (10) | 0.0767 (12) | −0.0047 (7) | −0.0055 (9) | 0.0074 (10) |
| O2 | 0.0584 (10) | 0.0524 (10) | 0.0920 (15) | −0.0043 (8) | −0.0100 (10) | 0.0205 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.0586 (15) | 0.0618 (15) | 0.085 (2) | −0.0056 (12) | −0.0049 (15) | 0.0124 (16) |
| C2 | 0.0460 (12) | 0.0512 (12) | 0.0443 (12) | 0.0027 (9) | −0.0027 (11) | −0.0031 (11) |
| C3 | 0.0485 (13) | 0.0532 (12) | 0.0519 (13) | 0.0110 (10) | −0.0098 (11) | 0.0034 (12) |
| C4 | 0.0531 (13) | 0.0433 (11) | 0.0477 (12) | 0.0069 (10) | −0.0060 (11) | 0.0016 (10) |
| C5 | 0.0479 (12) | 0.0410 (10) | 0.0357 (10) | 0.0080 (8) | −0.0030 (10) | −0.0034 (9) |
| C6 | 0.0482 (12) | 0.0446 (12) | 0.0476 (12) | 0.0082 (9) | −0.0083 (10) | 0.0005 (11) |
| C7 | 0.0523 (12) | 0.0428 (11) | 0.0466 (12) | 0.0080 (10) | −0.0005 (11) | −0.0005 (10) |
| C8 | 0.0470 (11) | 0.0414 (10) | 0.0365 (10) | 0.0086 (9) | −0.0040 (9) | −0.0039 (9) |
| C9 | 0.0536 (12) | 0.0425 (11) | 0.0485 (13) | 0.0069 (10) | −0.0049 (11) | −0.0001 (10) |
| C10 | 0.0521 (13) | 0.0539 (12) | 0.0404 (12) | 0.0021 (10) | 0.0062 (10) | −0.0082 (11) |
| C11 | 0.0640 (16) | 0.0615 (15) | 0.0564 (15) | −0.0072 (12) | 0.0080 (13) | −0.0007 (12) |
| C12 | 0.0664 (17) | 0.0803 (18) | 0.0644 (16) | −0.0188 (13) | 0.0157 (16) | −0.0118 (16) |
| C13 | 0.0431 (13) | 0.094 (2) | 0.0701 (18) | −0.0063 (13) | 0.0081 (14) | −0.0234 (17) |
| C14 | 0.0461 (14) | 0.0761 (18) | 0.0612 (16) | 0.0082 (12) | 0.0014 (12) | −0.0127 (13) |
| C15 | 0.0450 (12) | 0.0569 (13) | 0.0450 (12) | 0.0079 (10) | 0.0026 (11) | −0.0102 (11) |
| C16 | 0.0517 (12) | 0.0459 (11) | 0.0473 (12) | 0.0076 (10) | −0.0047 (11) | −0.0022 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.374 (3) | C8—C9 | 1.500 (3) |
| O1—C1 | 1.416 (3) | C9—C10 | 1.489 (3) |
| O2—C7 | 1.370 (3) | C9—H9A | 0.9700 |
| O2—C1 | 1.412 (3) | C9—H9B | 0.9700 |
| C1—H1A | 0.9700 | C10—C11 | 1.375 (3) |
| C1—H1B | 0.9700 | C10—C15 | 1.402 (3) |
| C2—C3 | 1.351 (3) | C11—C12 | 1.385 (4) |
| C2—C7 | 1.373 (3) | C11—H11 | 0.9300 |
| C3—C4 | 1.393 (3) | C12—C13 | 1.369 (4) |
| C3—H3 | 0.9300 | C12—H12 | 0.9300 |
| C4—C5 | 1.387 (3) | C13—C14 | 1.380 (4) |
| C4—H4 | 0.9300 | C13—H13 | 0.9300 |
| C5—C6 | 1.402 (3) | C14—C15 | 1.379 (3) |
| C5—C8 | 1.455 (3) | C14—H14 | 0.9300 |
| C6—C7 | 1.355 (3) | C15—C16 | 1.446 (3) |
| C6—H6 | 0.9300 | C16—H16 | 0.9300 |
| C8—C16 | 1.350 (3) | | |
| | | | |
| C2—O1—C1 | 104.93 (18) | C5—C8—C9 | 122.86 (17) |
| C7—O2—C1 | 105.56 (18) | C10—C9—C8 | 104.06 (17) |
| O2—C1—O1 | 108.9 (2) | C10—C9—H9A | 110.9 |
| O2—C1—H1A | 109.9 | C8—C9—H9A | 110.9 |
| O1—C1—H1A | 109.9 | C10—C9—H9B | 110.9 |
| O2—C1—H1B | 109.9 | C8—C9—H9B | 110.9 |
| O1—C1—H1B | 109.9 | H9A—C9—H9B | 109.0 |
| H1A—C1—H1B | 108.3 | C11—C10—C15 | 120.8 (2) |
| C3—C2—C7 | 121.5 (2) | C11—C10—C9 | 130.8 (2) |
| C3—C2—O1 | 128.4 (2) | C15—C10—C9 | 108.4 (2) |
| C7—C2—O1 | 110.1 (2) | C10—C11—C12 | 118.6 (3) |

| | | | |
|---|---|---|---|
| C2—C3—C4 | 116.8 (2) | C10—C11—H11 | 120.7 |
| C2—C3—H3 | 121.6 | C12—C11—H11 | 120.7 |
| C4—C3—H3 | 121.6 | C13—C12—C11 | 120.8 (3) |
| C5—C4—C3 | 122.6 (2) | C13—C12—H12 | 119.6 |
| C5—C4—H4 | 118.7 | C11—C12—H12 | 119.6 |
| C3—C4—H4 | 118.7 | C12—C13—C14 | 121.0 (2) |
| C4—C5—C6 | 118.8 (2) | C12—C13—H13 | 119.5 |
| C4—C5—C8 | 120.23 (19) | C14—C13—H13 | 119.5 |
| C6—C5—C8 | 120.99 (18) | C15—C14—C13 | 119.1 (3) |
| C7—C6—C5 | 117.51 (19) | C15—C14—H14 | 120.4 |
| C7—C6—H6 | 121.2 | C13—C14—H14 | 120.4 |
| C5—C6—H6 | 121.2 | C14—C15—C10 | 119.7 (2) |
| C6—C7—O2 | 127.8 (2) | C14—C15—C16 | 132.2 (2) |
| C6—C7—C2 | 122.8 (2) | C10—C15—C16 | 108.1 (2) |
| O2—C7—C2 | 109.38 (19) | C8—C16—C15 | 110.6 (2) |
| C16—C8—C5 | 128.3 (2) | C8—C16—H16 | 124.7 |
| C16—C8—C9 | 108.88 (19) | C15—C16—H16 | 124.7 |

| | | | |
|---|---|---|---|
| C7—O2—C1—O1 | 10.4 (3) | C4—C5—C8—C9 | 0.9 (3) |
| C2—O1—C1—O2 | −9.7 (3) | C6—C5—C8—C9 | −179.3 (2) |
| C1—O1—C2—C3 | −176.1 (3) | C16—C8—C9—C10 | 0.3 (2) |
| C1—O1—C2—C7 | 5.4 (3) | C5—C8—C9—C10 | −179.4 (2) |
| C7—C2—C3—C4 | −0.7 (3) | C8—C9—C10—C11 | −178.7 (2) |
| O1—C2—C3—C4 | −179.0 (2) | C8—C9—C10—C15 | −0.2 (2) |
| C2—C3—C4—C5 | −0.4 (4) | C15—C10—C11—C12 | −0.3 (4) |
| C3—C4—C5—C6 | 0.9 (4) | C9—C10—C11—C12 | 178.0 (3) |
| C3—C4—C5—C8 | −179.3 (2) | C10—C11—C12—C13 | 0.4 (4) |
| C4—C5—C6—C7 | −0.3 (3) | C11—C12—C13—C14 | −0.2 (4) |
| C8—C5—C6—C7 | 179.9 (2) | C12—C13—C14—C15 | −0.1 (4) |
| C5—C6—C7—O2 | 177.9 (2) | C13—C14—C15—C10 | 0.1 (4) |
| C5—C6—C7—C2 | −0.8 (4) | C13—C14—C15—C16 | −178.2 (3) |
| C1—O2—C7—C6 | 174.2 (3) | C11—C10—C15—C14 | 0.1 (3) |
| C1—O2—C7—C2 | −7.0 (3) | C9—C10—C15—C14 | −178.6 (2) |
| C3—C2—C7—C6 | 1.3 (4) | C11—C10—C15—C16 | 178.8 (2) |
| O1—C2—C7—C6 | 179.9 (2) | C9—C10—C15—C16 | 0.1 (3) |
| C3—C2—C7—O2 | −177.6 (2) | C5—C8—C16—C15 | 179.4 (2) |
| O1—C2—C7—O2 | 1.0 (3) | C9—C8—C16—C15 | −0.2 (3) |
| C4—C5—C8—C16 | −178.7 (2) | C14—C15—C16—C8 | 178.6 (2) |
| C6—C5—C8—C16 | 1.1 (4) | C10—C15—C16—C8 | 0.1 (3) |