organic compounds

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

Rui-Xue Deng,[a] Wei-Yi Zhou,[b] Xiao-Juan Deng[b] and Liang-Dong Sun[a]*

[a]Department of Chemistry, Tianjin University, Tianjin 300072, People's Republic of China, and [b]Analysis Center, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail: dengliu20022002@yahoo.com.cn

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).
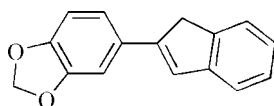
Experimental

Crystal data

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

Data collection

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

Refinement

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

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

Bruker (1997). SMART (Version 5.611), SAINT (Version 6.0) and SADABS (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.

Rayabarapu, D. K., Yang, C. H. & Cheng, C. H. (2003). J. Org. Chem. **68**, 6726–6731.

Senanayake, C. H., Roberts, F. E., DiMichele, L. M., Ryan, K. M., Liu, J., Fredenburgh, L. E., Foster, B. S., Douglas, A. W., Larsen, R. D., Verhoeven, T. R. & Reider, P. J. (1995). Tetrahedron Lett. **36**, 3993–3996.

Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.

Sheldrick, G. M. (2008). Acta Cryst. A**64**, 112–122.

supplementary materials

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

R.-X. Deng, W.-Y. Zhou, X.-J. Deng and L.-D. Sun

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 π-π 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].

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.

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 $U_{iso}(H) = 1.2U_{eq}$(parent). In the absence of significant anomalous scattering effects the Friedel pairs were merged.

Figures
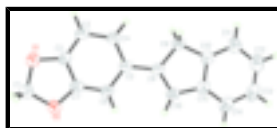

Fig. 1. View of the molecule of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level.

supplementary materials

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

Crystal data

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

Data collection

Bruker SMART CCD area-detector diffractometer	1286 independent reflections
Radiation source: fine-focus sealed tube	1072 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.035$
$T = 294(2)$ K	$\theta_{max} = 26.4°$
φ and ω scans	$\theta_{min} = 1.8°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -27 \rightarrow 13$
$T_{min} = 0.979$, $T_{max} = 0.990$	$k = -8 \rightarrow 8$
6325 measured reflections	$l = -8 \rightarrow 9$

Refinement

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

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 $F^2 > 2\mathrm{sigma}(F^2)$ 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 ($Å^2$)

	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, °)

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)

Fig. 1