

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

3,3'-Oxybi[isobenzofuran-1(3H)-one]

Wenkuan Li, Handong Yin,* Liyuan Wen, Kang Li and Weidong Fan

College of Chemistry and Chemical Engineering, Liaocheng University, Shandong 252059, People's Republic of China

Correspondence e-mail: handongyin@163.com

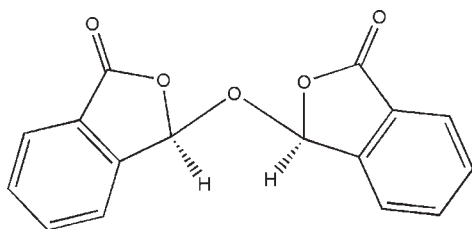
Received 18 September 2009; accepted 25 September 2009

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.031; wR factor = 0.078; data-to-parameter ratio = 6.0.

The title compound, $\text{C}_{16}\text{H}_{10}\text{O}_5$, consists of two isobenzofuran-1(3H)-one moieties which are linked by a bridging O atom. The two halves of the molecule display approximate non-crystallographic mirror symmetry. The dihedral angle between the two isobenzofuran-1(3H)-one ring systems is $53.18(6)^\circ$. Two chiral carbon centres are observed in the compound, but their absolute configurations could not be determined. In the crystal structure, intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link molecules into zigzag chains along c . Additional $\text{C}-\text{H}\cdots\text{O}$ interactions connect adjacent chains.

Related literature

For general background to isobenzofuran-1(3H)-ones, see: Landge *et al.* (2008); Mukhopadhyay & Kundu (2001); Paradkar *et al.* (1998).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{10}\text{O}_5$
 $M_r = 282.24$

 Monoclinic, Pn
 $a = 4.4449(6)$ Å

 $b = 6.4937(8)$ Å
 $c = 22.222(2)$ Å
 $\beta = 91.334(1)^\circ$
 $V = 641.25(13)$ Å³
 $Z = 2$

 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 298$ K
 $0.44 \times 0.29 \times 0.11$ mm

Data collection

 Siemens SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.953$, $T_{\max} = 0.988$

 3081 measured reflections
 1139 independent reflections
 972 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.078$
 $S = 1.01$
 1139 reflections
 190 parameters

 2 restraints
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.13$ e Å⁻³
 $\Delta\rho_{\min} = -0.13$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6}\cdots\text{O5}^{\text{i}}$	0.93	2.68	3.407 (4)	136
$\text{C1}-\text{H1}\cdots\text{O3}^{\text{ii}}$	0.98	2.61	3.337 (4)	131

Symmetry codes: (i) $x - \frac{1}{2}, -y + 1, z - \frac{1}{2}$; (ii) $x, y - 1, z$.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); 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.

We acknowledge the financial support of the Natural Science Foundation of China (No. 20771053) and the Natural Science Foundation of Shandong Province (Y2008B48). This work was also supported by the 'Students Technology Cultural Innovation Fund' of Liaocheng University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2658).

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supplementary materials

Acta Cryst. (2009). E65, o2577 [doi:10.1107/S1600536809038914]

3,3'-Oxybi[isobenzofuran-1(3*H*)-one]

W. Li, H. Yin, L. Wen, K. Li and W. Fan

Comment

Isobenzofuran-1(3*H*)-ones represent an important class of natural products that possess significant biological properties (Landge *et al.*, 2008). A number of isobenzofuran derivatives have been reported in the literature (Paradkar *et al.*, 1998; Mukhopadhyay & Kundu, 2001). Herein, the crystal structure of the title compound is presented.

The title compound (Fig. 1) is composed of two isobenzofuran-1(3*H*)-one moieties which are linked together by a bridging O1 atom. The two moieties display approximate mirror symmetry. The two isobenzofuran-1(3*H*)-one ring systems are not coplanar and are twisted from each other by a dihedral angle of 53.18 (6)°. The title compound is a chiral compound with two chiral centers at C₁ and C₉ but the absolute configuration could not be determined. In the crystal structure, C6—H6···O5 hydrogen bonds link molecules into zig-zag chains along *c*. Additional C1—H1···O3 interactions connect adjacent chains (Fig. 2).

Experimental

The product was obtained by a simple synthetic method: A mixture of triphenylantimony (3.00 mmol) and phthalic acid (3.00 mmol) in a stoppered flask was heated to 383 K. The mixture was shaken for 2.5 h. The product was crystallized from a solution of dichloromethane/petroleum ether (1:1), and to afford the title compound unexpectedly.

Refinement

In the absence of significant anomalous scattering effects, 1139 Friedel pairs were merged. H atoms were positioned geometrically, and constrained to ride on their parent atoms, with C—H = 0.93 and 0.98 Å for aromatic and methine H atoms, respectively, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

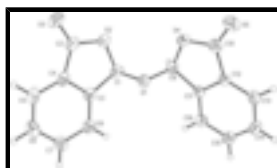


Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids.



Fig. 2. Packing diagram of the title compound viewed approximately along the *b* axis. H atoms are omitted for clarity.

3,3'-Oxybi[isobenzofuran-1(3H)-one]

Crystal data

$C_{16}H_{10}O_5$	$F_{000} = 292$
$M_r = 282.24$	$D_x = 1.462 \text{ Mg m}^{-3}$
Monoclinic, Pn	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2yac	Cell parameters from 1280 reflections
$a = 4.4449 (6) \text{ \AA}$	$\theta = 2.8\text{--}23.0^\circ$
$b = 6.4937 (8) \text{ \AA}$	$\mu = 0.11 \text{ mm}^{-1}$
$c = 22.222 (2) \text{ \AA}$	$T = 298 \text{ K}$
$\beta = 91.334 (1)^\circ$	Block, colorless
$V = 641.25 (13) \text{ \AA}^3$	$0.44 \times 0.29 \times 0.11 \text{ mm}$
$Z = 2$	

Data collection

Siemens SMART CCD area-detector diffractometer	1139 independent reflections
Radiation source: fine-focus sealed tube	972 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.025$
$T = 298 \text{ K}$	$\theta_{\text{max}} = 25.0^\circ$
φ and ω scans	$\theta_{\text{min}} = 1.8^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -3 \rightarrow 5$
$T_{\text{min}} = 0.953$, $T_{\text{max}} = 0.988$	$k = -7 \rightarrow 7$
3081 measured reflections	$l = -26 \rightarrow 26$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.078$	$w = 1/[\sigma^2(F_o^2) + (0.0502P)^2 + 0.0032P]$
$S = 1.01$	where $P = (F_o^2 + 2F_c^2)/3$
1139 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
190 parameters	$\Delta\rho_{\text{max}} = 0.13 \text{ e \AA}^{-3}$
2 restraints	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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O1	0.4119 (4)	0.1516 (3)	0.04946 (8)	0.0445 (5)
O2	0.7298 (5)	0.4086 (3)	0.01338 (9)	0.0511 (6)
O3	0.7306 (7)	0.7164 (3)	-0.03192 (13)	0.0742 (8)
O4	0.6010 (5)	0.2116 (3)	0.14674 (10)	0.0581 (6)
O5	0.4721 (8)	0.2569 (4)	0.24282 (12)	0.0859 (9)
C1	0.6000 (7)	0.2076 (5)	0.00288 (13)	0.0401 (7)
H1	0.7583	0.1044	-0.0022	0.048*
C2	0.6456 (7)	0.5423 (5)	-0.03175 (14)	0.0494 (8)
C3	0.4189 (7)	0.2325 (4)	-0.05400 (13)	0.0414 (7)
C4	0.4482 (7)	0.4308 (5)	-0.07433 (13)	0.0445 (7)
C5	0.3011 (8)	0.4956 (6)	-0.12687 (14)	0.0555 (9)
H5	0.3231	0.6295	-0.1409	0.067*
C6	0.1225 (9)	0.3563 (6)	-0.15739 (15)	0.0620 (10)
H6	0.0207	0.3965	-0.1925	0.074*
C7	0.0922 (9)	0.1584 (6)	-0.13675 (16)	0.0598 (9)
H7	-0.0305	0.0669	-0.1582	0.072*
C8	0.2407 (7)	0.0915 (5)	-0.08456 (14)	0.0499 (8)
H8	0.2207	-0.0430	-0.0708	0.060*
C9	0.5534 (7)	0.0616 (5)	0.10002 (14)	0.0443 (7)
H9	0.7448	-0.0014	0.0890	0.053*
C10	0.4522 (8)	0.1563 (5)	0.19777 (15)	0.0561 (9)
C11	0.3520 (6)	-0.0951 (4)	0.12751 (13)	0.0383 (7)
C12	0.2908 (6)	-0.0361 (5)	0.18517 (12)	0.0398 (7)
C13	0.1088 (8)	-0.1539 (6)	0.22104 (14)	0.0527 (8)
H13	0.0671	-0.1132	0.2601	0.063*
C14	-0.0088 (8)	-0.3326 (5)	0.19756 (16)	0.0567 (9)
H14	-0.1309	-0.4152	0.2209	0.068*
C15	0.0531 (8)	-0.3905 (5)	0.13930 (15)	0.0565 (9)
H15	-0.0294	-0.5119	0.1240	0.068*
C16	0.2336 (7)	-0.2733 (5)	0.10336 (14)	0.0475 (8)
H16	0.2740	-0.3131	0.0642	0.057*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0407 (11)	0.0523 (13)	0.0405 (11)	0.0026 (9)	0.0024 (9)	0.0118 (10)
O2	0.0571 (13)	0.0440 (13)	0.0520 (12)	-0.0096 (11)	-0.0019 (10)	0.0043 (11)
O3	0.097 (2)	0.0396 (14)	0.0862 (17)	-0.0174 (15)	0.0044 (15)	0.0040 (13)
O4	0.0705 (15)	0.0480 (13)	0.0551 (14)	-0.0231 (12)	-0.0137 (11)	0.0084 (11)
O5	0.132 (3)	0.0649 (18)	0.0596 (15)	-0.0248 (17)	-0.0195 (16)	-0.0118 (14)
C1	0.0425 (17)	0.0349 (15)	0.0430 (16)	-0.0007 (13)	0.0061 (13)	0.0023 (13)
C2	0.055 (2)	0.0401 (19)	0.0534 (18)	-0.0032 (15)	0.0119 (16)	0.0017 (16)
C3	0.0455 (17)	0.0412 (17)	0.0382 (15)	0.0043 (15)	0.0114 (13)	-0.0021 (14)
C4	0.0517 (18)	0.0407 (17)	0.0416 (16)	0.0028 (15)	0.0144 (14)	0.0031 (14)
C5	0.070 (2)	0.0505 (19)	0.0468 (18)	0.0112 (18)	0.0135 (17)	0.0141 (17)
C6	0.068 (2)	0.076 (3)	0.0421 (19)	0.012 (2)	0.0024 (17)	0.0031 (18)
C7	0.060 (2)	0.073 (3)	0.0467 (18)	-0.0015 (18)	0.0011 (16)	-0.0067 (19)
C8	0.056 (2)	0.0453 (19)	0.0483 (18)	-0.0011 (16)	0.0072 (15)	-0.0006 (15)

supplementary materials

C9	0.0421 (16)	0.0425 (18)	0.0479 (17)	-0.0022 (14)	-0.0052 (13)	0.0075 (14)
C10	0.072 (2)	0.0447 (19)	0.051 (2)	-0.0063 (17)	-0.0157 (18)	0.0046 (17)
C11	0.0325 (14)	0.0384 (17)	0.0437 (16)	0.0012 (13)	-0.0046 (12)	0.0066 (13)
C12	0.0415 (15)	0.0410 (17)	0.0367 (15)	-0.0018 (14)	-0.0058 (12)	0.0053 (13)
C13	0.0552 (19)	0.064 (2)	0.0389 (16)	-0.0008 (18)	-0.0001 (14)	0.0066 (16)
C14	0.054 (2)	0.056 (2)	0.060 (2)	-0.0156 (17)	0.0015 (17)	0.0150 (17)
C15	0.062 (2)	0.0431 (18)	0.064 (2)	-0.0142 (17)	-0.0052 (17)	0.0016 (17)
C16	0.0485 (19)	0.0449 (18)	0.0490 (18)	-0.0032 (15)	-0.0011 (15)	-0.0036 (15)

Geometric parameters (Å, °)

O1—C1	1.394 (3)	C6—H6	0.9300
O1—C9	1.403 (4)	C7—C8	1.391 (5)
O2—C2	1.372 (4)	C7—H7	0.9300
O2—C1	1.444 (4)	C8—H8	0.9300
O3—C2	1.192 (4)	C9—C11	1.495 (4)
O4—C10	1.374 (4)	C9—H9	0.9800
O4—C9	1.436 (4)	C10—C12	1.465 (5)
O5—C10	1.197 (4)	C11—C12	1.371 (4)
C1—C3	1.491 (4)	C11—C16	1.375 (4)
C1—H1	0.9800	C12—C13	1.380 (4)
C2—C4	1.467 (4)	C13—C14	1.371 (5)
C3—C4	1.372 (4)	C13—H13	0.9300
C3—C8	1.379 (4)	C14—C15	1.382 (5)
C4—C5	1.390 (4)	C14—H14	0.9300
C5—C6	1.373 (5)	C15—C16	1.375 (4)
C5—H5	0.9300	C15—H15	0.9300
C6—C7	1.372 (5)	C16—H16	0.9300
C1—O1—C9	116.0 (2)	C7—C8—H8	121.3
C2—O2—C1	110.6 (2)	O1—C9—O4	110.6 (2)
C10—O4—C9	110.7 (2)	O1—C9—C11	110.4 (2)
O1—C1—O2	111.1 (2)	O4—C9—C11	104.2 (2)
O1—C1—C3	109.6 (2)	O1—C9—H9	110.5
O2—C1—C3	104.2 (2)	O4—C9—H9	110.5
O1—C1—H1	110.6	C11—C9—H9	110.5
O2—C1—H1	110.6	O5—C10—O4	121.4 (3)
C3—C1—H1	110.6	O5—C10—C12	130.7 (3)
O3—C2—O2	121.4 (3)	O4—C10—C12	107.9 (3)
O3—C2—C4	130.6 (3)	C12—C11—C16	121.1 (3)
O2—C2—C4	108.0 (2)	C12—C11—C9	109.0 (3)
C4—C3—C8	121.3 (3)	C16—C11—C9	129.8 (3)
C4—C3—C1	109.1 (3)	C11—C12—C13	121.1 (3)
C8—C3—C1	129.6 (3)	C11—C12—C10	108.1 (3)
C3—C4—C5	120.9 (3)	C13—C12—C10	130.8 (3)
C3—C4—C2	108.1 (3)	C14—C13—C12	118.2 (3)
C5—C4—C2	130.9 (3)	C14—C13—H13	120.9
C6—C5—C4	118.1 (3)	C12—C13—H13	120.9
C6—C5—H5	121.0	C13—C14—C15	120.3 (3)
C4—C5—H5	121.0	C13—C14—H14	119.9

C7—C6—C5	120.8 (3)	C15—C14—H14	119.9
C7—C6—H6	119.6	C16—C15—C14	121.7 (3)
C5—C6—H6	119.6	C16—C15—H15	119.2
C6—C7—C8	121.5 (4)	C14—C15—H15	119.2
C6—C7—H7	119.2	C11—C16—C15	117.5 (3)
C8—C7—H7	119.2	C11—C16—H16	121.2
C3—C8—C7	117.3 (3)	C15—C16—H16	121.2
C3—C8—H8	121.3		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C6—H6 \cdots O5 ⁱ	0.93	2.68	3.407 (4)	136
C1—H1 \cdots O3 ⁱⁱ	0.98	2.61	3.337 (4)	131

Symmetry codes: (i) $x-1/2, -y+1, z-1/2$; (ii) $x, y-1, z$.

Fig. 1

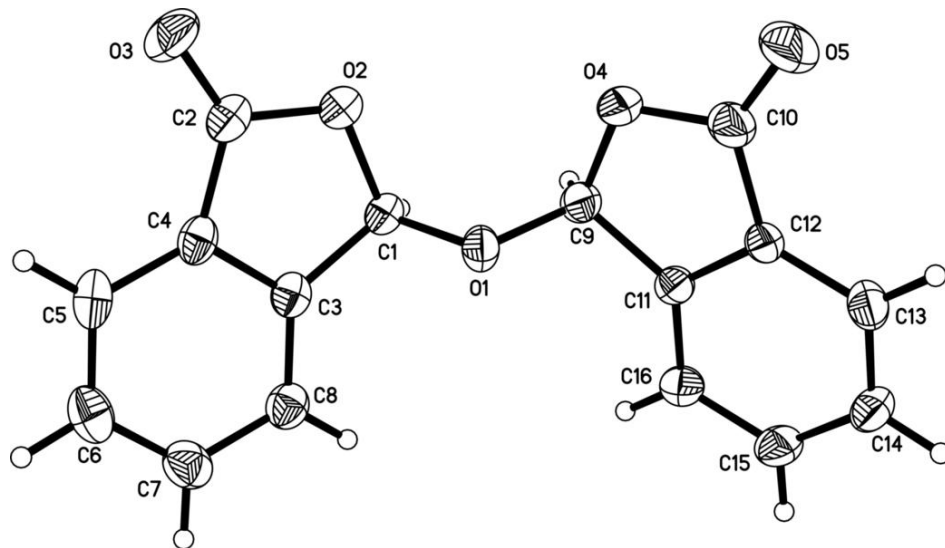


Fig. 2

