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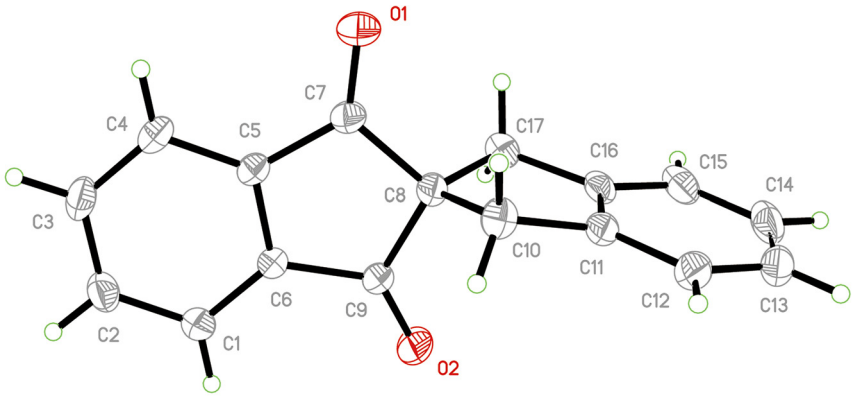
Crystal structure of 1′,3′-dihydro-2,2′-spirobi[indene]-1,3-dione, $C_{17}H_{12}O_2$



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Abstract

$C_{17}H_{12}O_2$, monoclinic, $P2_1/c$ (no. 14), a = 11.0333(13) Å, b = 15.7993(19) Å, c = 7.0619(9) Å, β = 92.302(2)°, V = 1230.0(3) Å^3, Z = 4, $R_{gt}(F)$ = 0.0420, w$R_{ref}(F^2)$ = 0.1081, T = 298 K.

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A part of the molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless cube
Size:	0.26 × 0.22 × 0.21 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX2, φ and ω
θ_{max}, completeness:	27.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7285, 2780, 0.026
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 2,037
$N(param)_{refined}$:	173
Programs:	Bruker,[1] OLEX2,[2] SHELX[3,4]

1 Source of materials

The synthesis of 1′,3′-dihydro-2,2′-spirobi[indene]-1,3-dione (SPIN) primarily referred to the literature of J. Wilbuer and coworkers.[5] Reagents were purchased from Macklin Inc. and used as received without further purification. An amount of 0.5 g SPIN (2.0 mmol) was added to a 15 mL glass tube with a mixed solution of methanol and dichloromethane (10 mL, 1:1, v/v). After several days, cube-shaped crystals were obtained, washed with anhydrous methanol, dried in air, yield 46 % (based on SPIN).

2 Experimental details

The crystal structure was determined using the SHELXT program, followed by refinement using the SHELXL program. The refinement process included anisotropic displacement parameters for all non-hydrogen atoms, while the hydrogen atoms were placed in idealized positions with isotropic thermal parameters.

3 Comment

The special stereoelectronic effects of spiro-conjugated compounds make them potentially useful in the fields of optics, dyes, organic conductors, etc.[6–8] At present, there are

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.62906 (13)	0.35645 (9)	0.8624 (2)	0.0424 (4)
H1	0.673358	0.405470	0.888209	0.051*
C2	0.68129 (14)	0.27763 (10)	0.8851 (2)	0.0499 (4)
H2	0.762019	0.273442	0.927252	0.060*
C3	0.61516 (15)	0.20427 (10)	0.8461 (2)	0.0487 (4)
H3	0.652697	0.151872	0.861321	0.058*
C4	0.49504 (14)	0.20786 (8)	0.7853 (2)	0.0417 (4)
H4	0.450825	0.158738	0.760184	0.050*
C5	0.44216 (12)	0.28701 (8)	0.76271 (18)	0.0336 (3)
C6	0.50871 (12)	0.36036 (8)	0.79994 (18)	0.0333 (3)
C7	0.31751 (12)	0.30919 (9)	0.6971 (2)	0.0394 (3)
C8	0.30486 (12)	0.40571 (8)	0.7027 (2)	0.0376 (3)
C9	0.43326 (12)	0.43564 (8)	0.7580 (2)	0.0378 (3)
C10	0.21476 (14)	0.43489 (9)	0.8536 (2)	0.0450 (4)
H10A	0.155896	0.390953	0.877511	0.054*
H10B	0.257508	0.449384	0.971906	0.054*
C11	0.15425 (12)	0.51137 (9)	0.7660 (2)	0.0400 (3)
C12	0.08075 (14)	0.57069 (10)	0.8498 (3)	0.0529 (4)
H12	0.067266	0.567986	0.978858	0.063*
C13	0.02785 (15)	0.63384 (11)	0.7392 (3)	0.0656 (6)
H13	−0.021631	0.673839	0.794158	0.079*
C14	0.04781 (15)	0.63797 (10)	0.5487 (4)	0.0662 (6)
H14	0.011214	0.680578	0.475762	0.079*
C15	0.12184 (13)	0.57941 (10)	0.4638 (3)	0.0536 (4)
H15	0.135333	0.582581	0.334890	0.064*
C16	0.17539 (11)	0.51610 (9)	0.5740 (2)	0.0397 (3)
C17	0.25703 (13)	0.44554 (10)	0.5137 (2)	0.0426 (4)
H17A	0.323319	0.467347	0.441801	0.051*
H17B	0.212164	0.404334	0.436939	0.051*
O1	0.23814 (10)	0.26026 (7)	0.64641 (19)	0.0636 (4)
O2	0.46702 (10)	0.50840 (6)	0.76245 (19)	0.0617 (4)

relatively few reported crystal structures of spiro-conjugated compounds. To better understand the properties of these compounds, it is necessary to synthesize more novel spiro-conjugated compounds and investigate their crystal structures in detail. Therefore, we report the single-crystal structure of the title 2,2′-spirobi[indene] compound. The asymmetric unit contains one SPIN molecule. The C–C bond and C–O bond lengths are in the normal range of 1.373(3) – 1.5320(19) Å and 1.2084(16) – 1.2115(17) Å, compared to the reported structures with similar molecules.[9–13] The torsion angle of C16–C17–C8–C9 and C9–C8–C17 are 100.642° and 111.992°. The SPIN molecules are linked by the C–H···O short contact (C4–H4···O2$^{1-x,\ 1/2+y,\ 1/2-z}$). Ultimately, a three-dimensional network was formed, facilitated by π-π stacking interactions, with centroid-centroid distances ranging from 3.6752(5) Å to 3.8876(5) Å. The crystal data for the compound has been deposited to the Cambridge Crystallographic Data Centre (CCDC no. 2391002).

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