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1-Phenyl-2-phenylsulfinylcyclobutan-1-ol

Wendy A. Loughlin, Catherine C. Rowen, Zane D. van den Berg and Peter C. Healy*

School of Science, Griffith University, Nathan, Brisbane 4111, Australia

Correspondence e-mail: p.healy@sct.gu.edu.au

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The structure of the title compound, $C_{16}H_{16}O_2S$, (I), has been determined as part of an investigation into the synthesis of fused carbocyclic ring systems containing a cyclobutanol ring. Compound (I), a monocyclic example, crystallizes with two molecules in the asymmetric unit. Conformational differences in the phenyl rings on each molecule permit the formation of an infinite chain of intermolecular O—H···O—S hydrogen bonds.

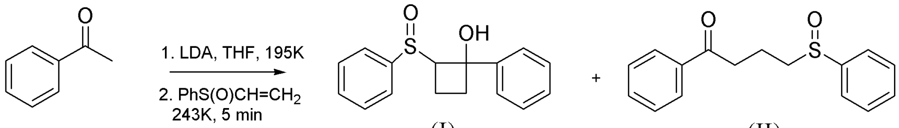
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.047
wR factor = 0.138
Data-to-parameter ratio = 14.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

We have recently shown that a novel cyclization reaction between the lithium enolates of simple ketones and phenyl vinyl sulfoxide provides a simple and convenient route to the preparation of a diverse array of fused carbocyclic ring systems bearing a bridgehead hydroxy group (Loughlin *et al.*, 2002). In the current study, use of the lithium enolate of acetophenone generated the novel monocyclic compound, (I). Noteworthy was the formation of (I) as the major cyclobutanol isomer and the novel monoalkylated product 1-phenyl-4-phenylsulfinylbutan-1-one, (II), in a 64:36 ratio from achiral acetophenone and phenyl vinyl sulfoxide, with less than five percent of other products observed.



(I)

(II)

Compound (I) crystallizes in space group $P2_1/c$ with two molecules, A and B, in the asymmetric unit (Fig. 1). The two molecules are structural isomers with the phenyl groups approximately orthogonal to their corresponding partners. The structure of the cyclobutanol sulfinyl core [$OS(C_4H_5)OH$] of the two molecules is similar, with relative stereochemistry ($1RS_c$,$2RS_c$,RS_s), together with their enantiomers. The bond lengths and angles for this core are similar to those reported for related systems (Loughlin *et al.*, 2002). The cyclobutane rings are distorted from planarity, with C1—C2—C3—C4 −20.1 (2)° (molecule A) and −18.6 (2)° (molecule B). The O1—C1—C2—S1 torsion angles are 28.9 (3)° (molecule A) and 29.9 (3)° (molecule B).

In the crystal lattice, each molecule forms O—H···O—S hydrogen bonds (Fig. 2) with enantiomers of its partner molecule, generating a hydrogen-bonded chain along the a axis. Interestingly, the molecules along this chain are also linked through edge-to-face C—H···π interactions between the phenyl groups attached at C1a and C1b..

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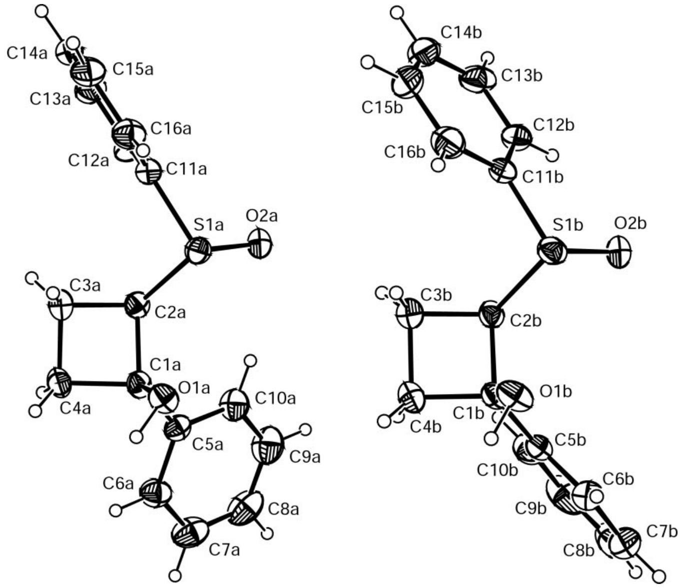

Figure 1

ORTEP-3 (Farrugia, 1997) plot showing the atomic atom-numbering scheme for the two molecules in the asymmetric unit of (I). Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

Experimental

Compounds (I) and (II) were prepared by the reaction of acetophenone (0.50 g, 0.49 ml, 4.20 mmol) in THF (5 ml) with lithium diisopropylamide (1.7 *M*, 4.60 mmol, 2.70 ml) in THF (30 ml) at 195 K under nitrogen over 10 min. Rapid addition of phenyl vinyl sulfoxide (0.63 g, 0.56 ml, 4.20 mmol) at 243 K with a 5 min reaction time and workup as described elsewhere (Loughlin *et al.*, 2002) was followed by purification by silica column chromatography (diethyl ether). Fraction 1 (299 mg) contained a 72:28 mixture of unreacted phenylvinyl sulfoxide and (I). Fraction 2 contained (II) (46 mg), m.p. 349–350 K. Suspension of fraction 1 in ether, filtration and recrystallization (ether) gave (I) as a white solid (m.p. 397–399 K). Crystals of (I) were isolated as colorless plates by slow evaporation of a solution of the pure compound in ether.

Crystal data

$C_{16}H_{16}O_2S$
$M_r = 272.36$
Monoclinic, $P2_1/c$
$a = 10.439$ (2) Å
$b = 17.361$ (3) Å
$c = 15.555$ (2) Å
$\beta = 92.74$ (1)°
$V = 2815.8$ (7) Å^3
$Z = 8$
$D_x = 1.285$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 12.5$–16.1°
$\mu = 0.23$ mm^{-1}
$T = 295$ K
Plate, colorless
0.50 × 0.20 × 0.20 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: none
6253 measured reflections
4965 independent reflections
2695 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\rm max} = 25.0°$
$h = -12 \rightarrow 7$
$k = -20 \rightarrow 0$
$l = -18 \rightarrow 18$
3 standard reflections every 150 reflections intensity decay: 1.7%

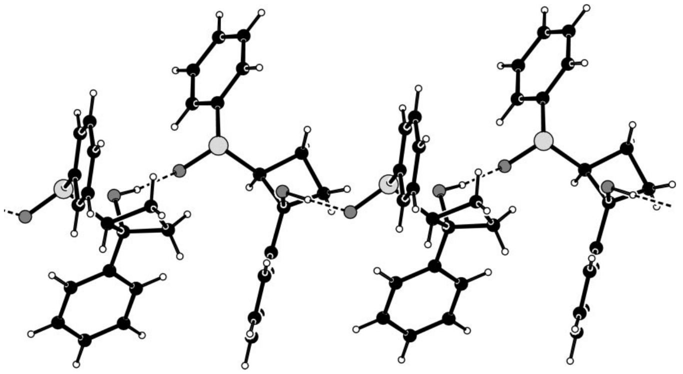

Figure 2

The hydrogen-bonding scheme for (I)

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.138$
$S = 1.00$
4965 reflections
344 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0509P)^2 + 0.6926P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.32$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0017 (4)

Table 1

Selected geometric parameters (Å, °).

S1*A*—O2*A*	1.512 (2)	S1*B*—C2*B*	1.800 (3)
S1*A*—C2*A*	1.802 (3)	S1*B*—C11*B*	1.783 (3)
S1*A*—C11*A*	1.785 (3)	O1*A*—C1*A*	1.429 (4)
S1*B*—O2*B*	1.508 (3)	O1*B*—C1*B*	1.411 (4)
O2*A*—S1*A*—C2*A*	105.92 (14)	S1*A*—C2*A*—C3*A*	117.3 (2)
O2*A*—S1*A*—C11*A*	107.38 (14)	S1*A*—C11*A*—C12*A*	121.3 (3)
C2*A*—S1*A*—C11*A*	96.60 (15)	S1*A*—C11*A*—C16*A*	118.4 (2)
C2*B*—S1*B*—C11*B*	96.69 (15)	O1*B*—C1*B*—C2*B*	106.3 (2)
O2*B*—S1*B*—C11*B*	106.72 (15)	C2*B*—C1*B*—C5*B*	118.8 (3)
O2*B*—S1*B*—C2*B*	104.73 (15)	S1*B*—C2*B*—C3*B*	119.8 (2)
O1*A*—C1*A*—C2*A*	107.8 (2)	S1*B*—C2*B*—C1*B*	116.5 (2)
O1*A*—C1*A*—C5*A*	110.1 (2)	S1*B*—C11*B*—C12*B*	121.3 (2)
C4*A*—C1*A*—C5*A*	118.9 (3)	S1*B*—C11*B*—C16*B*	118.6 (3)

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*A*—H1*A*···O2*B*$^{\rm i}$	0.95	1.79	2.735 (3)	172
O1*B*—H1*B*···O2*A*$^{\rm ii}$	0.95	1.74	2.698 (3)	173

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

H atoms were located at calculated positions with C—H set to 0.95 Å. Hydroxyl H atoms on O1 and O3 were located from a difference synthesis and O—H was set to 0.95 Å. All H atoms were constrained in the refinement. Due to a large fraction of weak data at higher angles, the 2θ maximum was limited to 50°.

Data collection: *MSC/AFC*-7 *Diffractometer Control Software* (Molecular Structure Corporation, 1999); cell refinement: *MSC/AFC*-7 *Diffractometer Control Software*; data reduction: *TEXSAN* for Windows (Molecular Structure Corporation, 2001);

program(s) used to solve structure: *TEXSAN* for Windows; program(s) used to refine structure: *TEXSAN* for Windows and *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2001) and *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *TEXSAN* for Windows and *PLATON* (Spek, 2001).

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