1-phenyl-2-phenylsulfinylcyclobutan-1-ol

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The structure of the title compound, C_{10}H_{16}O_{2}S, (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.

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-phenylsulfonylbutan-1-one, (II), in a 64:36 ratio from achiral acetophenone and phenyl vinyl sulfoxide, with less than five percent of other products observed.

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_6H_5)OH] of the two molecules is similar, with relative stereochemistry (1RS,2RS,3SR), 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.
Compounds (I) and (II) were prepared by the reaction of aceto-phenone (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 recrystal- lization (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.
program(s) used to solve structure: TEXSAN for Windows; program(s) used to refine structure: TEXSAN for Windows and SHELXL97 (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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