Crystal structure of phenylbenzylsulfone, $(C_6H_5)(C_7H_7)SO_2$

M.-C. Liu*[I], J.-C. Ding[I], W. Feng[II] and H.-Y. Wu[I]

[I] Wenzhou Normal College, Department of Chemistry and Material Science, Wenzhou, 325027 P. R. China
[II] Zhejiang University of Technology, College of Pharmaceutical Sciences, Hangzhou, 310014 P. R. China

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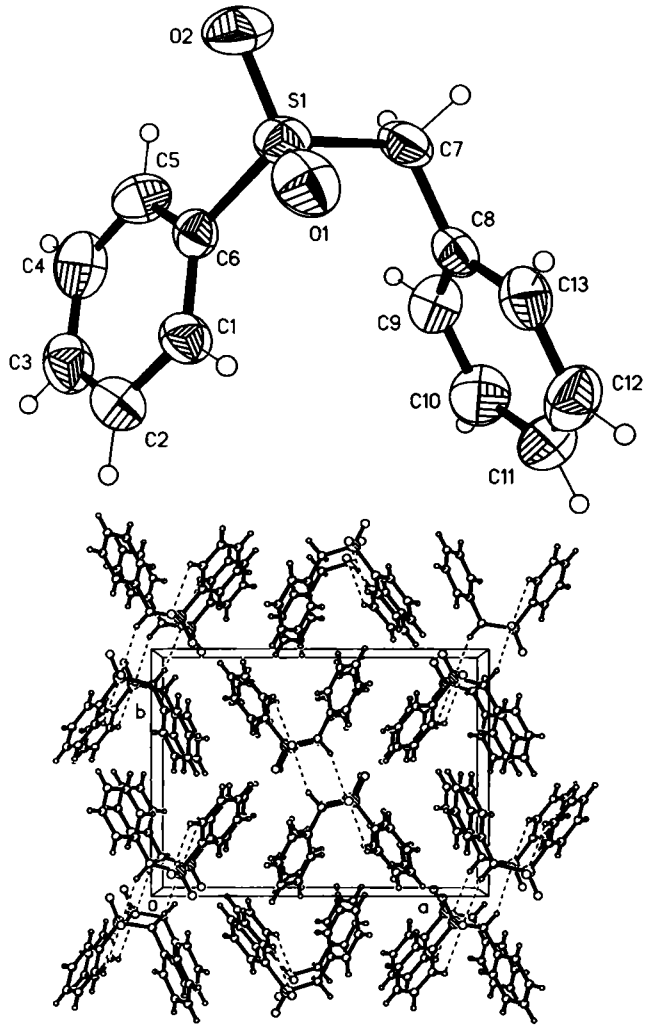

Abstract

$C_{13}H_{12}O_2S$, orthorhombic, $Pna2_1$ (no. 33), $a = 16.5888(6)$ Å, $b = 12.2537(5)$ Å, $c = 5.6054(2)$ Å, $V = 1139.4$ Å^3, $Z = 4$, $R_{gt}(F) = 0.059$, $wR_{ref}(F^2) = 0.128$, $T = 298$ K.

Source of material

The title compound was prepared by the *S*-hydrocarbylation of sodium phenylsulfinate dihydrate, $C_6H_5SO_2Na \cdot 2H_2O$ (2.0 g, 10 mmol), and benzyl bromide (1.2 ml, 11 mmol) in the presence of cetyltrimethylammonium bromide catalyst (1.8 g, 5 mmol). The reaction was carried out in a 630 W microwave oven for 1 min. Single crystals were obtained by using ethanol as solvent for recrystallization.

* Correspondence author (e-mail: mcl@wznc.zj.cn)

Discussion

The utilization of sulfones in organic synthesis has become a classic strategy in the preparation of the most demanding and sophisticated complex molecules. From the methodological point of view, sulfones have been employed in the preparation and functionalization of a wide variety of products [1]. In the study the sulfone synthesis, our group tried some unconventional methods. The synthesis of the title compound is an example of microwave-assisted synthesis [2,3] without use of solvent [4,5].

In the molecule (figure, top) the phenyl and benzyl groups are located on the right and left side of the sulfone, resembling two outstretched hands. The seven atoms C1, C2, C3, C4, C5, C6 and S1, along with the other seven atoms C7, C8, C9, C10, C11, C12 and C13 are essentially coplanar, with average deviations of 0.0153 Å and 0.0119 Å, respectively. The dihedral angle of the two above planes is 45.0(1)°. The S=O bond distances [1.436(3) Å and 1.437(3) Å] are comparable with those found for ethyl phenylsulfonylacetate [6]. There is one intramolecular hydrogen bond (C1–H1···O1), with an H···O distance of 2.54 Å. Moreover, a pair of molecules is linked by the intermolecular C7–H7···O1($-x,-y,-½+z$) bond with an H···O distance of 2.56 Å to form a dimer like a butterfly (figure, bottom).

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.21 × 0.39 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.65 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD, 600 frames, $\Delta\omega = 0.3°$
$2\theta_{max}$:	50.36°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5861, 1978
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1965
$N(param)_{refined}$:	145
Programs:	SHELX-97 [7], ORTEP-II [8]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4*a*	0.1355	0.3033	0.6446	0.052
H(2)	4*a*	0.2246	0.4451	0.5833	0.065
H(3)	4*a*	0.2997	0.4514	0.2343	0.068
H(4)	4*a*	0.2899	0.3124	–0.0378	0.072
H(5)	4*a*	0.2034	0.1671	0.0275	0.059
H(7A)	4*a*	0.0168	0.1405	0.0790	0.056
H(7B)	4*a*	–0.0319	0.0800	0.2785	0.056
H(9)	4*a*	0.0061	0.3317	0.0394	0.061
H(10)	4*a*	–0.0561	0.4938	0.1390	0.077
H(11)	4*a*	–0.1348	0.5041	0.4742	0.079
H(12)	4*a*	–0.1465	0.3546	0.7213	0.072
H(13)	4*a*	–0.0846	0.1921	0.6231	0.061

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S(1)	4*a*	0.09785(6)	0.11027(7)	0.4039(2)	0.0499(6)	0.0294(4)	0.0484(6)	0.0002(4)	–0.0042(5)	0.0048(5)
O(1)	4*a*	0.0818(2)	0.1094(2)	0.6556(6)	0.062(2)	0.047(2)	0.058(2)	–0.010(1)	–0.004(2)	0.019(2)
O(2)	4*a*	0.1316(2)	0.0140(2)	0.2968(7)	0.076(2)	0.032(2)	0.086(3)	0.008(2)	–0.007(2)	–0.003(2)
C(1)	4*a*	0.1673(2)	0.3049(3)	0.5080(9)	0.043(2)	0.040(2)	0.048(2)	–0.009(2)	0.004(2)	–0.001(2)
C(2)	4*a*	0.2197(3)	0.3900(4)	0.4703(8)	0.052(3)	0.054(3)	0.056(4)	–0.005(2)	–0.007(2)	–0.009(2)
C(3)	4*a*	0.2651(3)	0.3932(4)	0.263(1)	0.045(3)	0.049(3)	0.076(4)	–0.006(2)	–0.005(3)	0.014(3)
C(4)	4*a*	0.2589(3)	0.3102(4)	0.1004(9)	0.051(3)	0.077(3)	0.052(3)	0.000(3)	0.009(2)	0.017(3)
C(5)	4*a*	0.2074(3)	0.2234(4)	0.1380(8)	0.061(3)	0.045(2)	0.041(3)	0.006(2)	0.004(2)	0.000(2)
C(6)	4*a*	0.1621(2)	0.2228(3)	0.3446(7)	0.033(2)	0.037(2)	0.039(3)	0.003(2)	–0.008(2)	0.003(2)
C(7)	4*a*	0.0058(3)	0.1390(3)	0.2490(9)	0.047(2)	0.037(2)	0.056(3)	–0.007(2)	–0.014(2)	–0.006(2)
C(8)	4*a*	–0.0335(2)	0.2450(3)	0.3186(8)	0.035(2)	0.041(2)	0.045(2)	–0.008(2)	–0.009(2)	–0.000(2)
C(9)	4*a*	–0.0251(3)	0.3356(4)	0.1769(9)	0.050(2)	0.059(3)	0.045(3)	0.006(2)	0.002(2)	0.009(2)
C(10)	4*a*	–0.0625(3)	0.4329(4)	0.236(1)	0.067(3)	0.048(3)	0.078(4)	0.004(2)	0.001(3)	0.018(3)
C(11)	4*a*	–0.1085(3)	0.4393(4)	0.436(1)	0.056(3)	0.057(3)	0.084(5)	0.013(2)	0.001(3)	–0.010(3)
C(12)	4*a*	–0.1161(3)	0.3497(4)	0.5822(9)	0.051(3)	0.081(4)	0.047(3)	0.007(3)	0.009(2)	–0.006(3)
C(13)	4*a*	–0.0790(2)	0.2525(4)	0.524(1)	0.044(2)	0.055(3)	0.053(3)	–0.008(2)	0.003(2)	0.010(3)

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References

1. Alonso, D. A.; Najera, C.; Varea, M.: Simple, economical and environmentally friendly sulfone synthesis. Tetrahedron Lett. **43** (2002) 3459-3461.
2. Dallinger, D.; Gorobets, N. Y.; Kappe, C. O.: High-throughput synthesis of N_3-acylated dihydropyrimidines combining microwave-assisted synthesis and scavenging techniques. Org. Lett. **5** (2003) 1205-1208.
3. Wu, T. Y. H.; Schultz, P. G.; Ding, S.: One-pot two-step microwave-assisted reaction in constructing 4,5-disubstituted pyrazolopyrimidines. Org. Lett. **5** (2003) 3587-3590.
4. Loupy, A.; Petit, J.; Hamelin, J.; Texier-Boullet, F.; Jacquault, P.; Mathe, D.: New solvent free organic synthesis using focused microwaves. Synthesis **9** (1998) 1213-1214.
5. Melucci, M.; Barbarella, G.; Sotgiu, G.: Solvent-free, microwave-assisted synthesis of thiophene oligomers via Suzuki coupling. J. Org. Chem. **67** (2002) 8877-8884.
6. Ding, J. C.; Su, W. K.; Wu, H. Y.; Liu, M. C.; Ng, S. K.: Ethyl phenylsulfonylacetate. Acta Crystallogr. **E60** (2004) o310-o311.
7. Sheldrick, G. M.: SHELX-97. Programs for the Analysis of Crystal Structures (Release 97-2). University of Göttingen, Germany 1997.
8. Johnson, C. K.: ORTEP-II. Report ORNL-5138. Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA 1976.