organic papers

3-(4-Methoxyphenyl)-1-phenyl-3-(*p*-toluene-sulfonylamino)propan-1-one

Zhifang Yu,* Xiuyan Gu, Bing Zhao and Yan Li

Department of Chemistry, Tianjin University, Tianjin 300072, People's Republic of China

Correspondence e-mail:
zhifang@public.tpt.tj.cn

Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.050
wR factor = 0.127
Data-to-parameter ratio = 13.7

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

The title compound, $C_{23}H_{23}NO_4S$, crystallizes in the space group $P\bar{1}$. The S atom of the sulfonyl group has a distorted tetrahedral geometry. Intermolecular N—H···O hydrogen bonds are formed between the amino and carbonyl groups of two molecules related by a center of symmetry.

Comment

β–Aminoketones are important synthetic intermediates in organic synthesis and are almost always generated by the Mannich reaction (Miura & Tamaki, 2000; Ranu *et al.*, 2002). In our laboratory, we have recently investigated a new metal-mediated reaction for the preparation of β-aminoketones. A new compound, namely 3-(4-methoxyphenyl)-1-phenyl-3-(*p*-toluenesulfonylamino)propan-1-one, (I), has been synthesized by the reaction of *N*-[(4-methoxyphenyl)methylene]-4-methylbenzenesulfonamide (Shim & Yamamoto, 2000) with 2-bromoacetophenone in the presence of active zinc powder. An X-ray crystal structure determination of (I) has been carried out and the results are presented here.

(I)
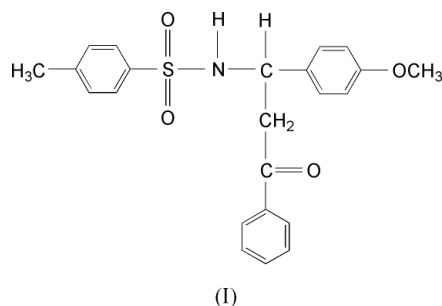
The molecular structure of (I) is illustrated in Fig. 1. The sulfonylphenyl ring (*A*) and the carbonylphenyl ring (*B*) are
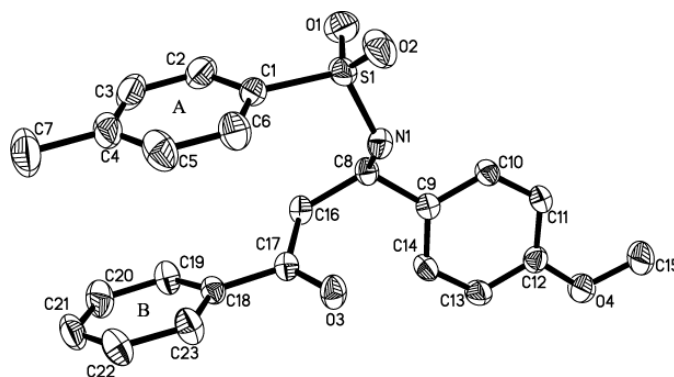

Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 35% probability level. H atoms have been omitted for clarity.

inclined at an angle of 12.1 (3)°. Rings *A* and *B* make dihedral angles of 97.5 (3)° and 85.8 (3)°, respectively, with the methoxyphenyl ring (C9–C14). The S atom has a distorted tetrahedral geometry, with the angles O1—S1—O2 [120.06 (15)°] and O2—S1—N1 [105.50 (13)°] deviating significantly from the regular tetrahedral value. Two molecules related by a center of symmetry are linked by two inter-molecular N—H···O hydrogen bonds (Table 2), resulting in the formation of a dimer.

Experimental

2–Bromoacetophenone (1.5 mmol) was added to a solution of *N*-[(4-methoxyphenyl)methylene]-4-methylbenzenesulfonamide (1.0 mmol) in dichloromethane (5.0 ml). This mixture was added to a round-bottomed flask, which was filled with zinc powder (3.0 mmol) and a trace amount of iodine. The reaction mixture was refluxed, with stirring, for about 11 h and then quenched with a saturated solution of NH_4Cl (5.0 ml) and 25% NH_4OH (5.0 ml). The mixture was extracted with dichloromethane, washed with brine and dried over magnesium sulfate. After removal of the solvent under reduced pressure, the residue was purified by flash chromatography (ethyl acetate–trichloromethane). A colorless powder was obtained (yield 60%) and single crystals suitable for crystallographic analysis were obtained by slow evaporation of an ethyl acetate-petroleum ether solution (m.p. 398–400 K). Spectroscopic analysis, IR (KBr, ν cm^{-1}): 3235, 1675, 1331, 1160; ^{1}H NMR (CDCl$_3$, p.p.m.): 7.80–6.68 (*m*, 13H), 5.67 (br, 1 H), 4.79 (*m*, 1H), 3.72 (*s*, 3H), 3.56 (*dd*, 1H), 3.43 (*dd*, 1H), 2.36 (*s*, 3H). Analysis required for $C_{23}H_{23}NO_4S$: C 67.48, H 5.62, N 3.42%; found: C 67.64, H 5.75, N 3.40%.

Crystal data

$C_{23}H_{23}NO_4S$	$Z = 2$
$M_r = 409.49$	$D_x = 1.329$ Mg m^{-3}
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 9.884$ (3) Å	Cell parameters from 991
$b = 9.924$ (3) Å	reflections
$c = 11.738$ (3) Å	$\theta = 3.0$–26.4°
$\alpha = 94.267$ (5)°	$\mu = 0.19$ mm^{-1}
$\beta = 91.985$ (4)°	$T = 293$ (2) K
$\gamma = 116.676$ (4)°	Prism, colorless
$V = 1023.0$ (5) Å^3	$0.26 \times 0.16 \times 0.12$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer	3607 independent reflections
	2537 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.018$
Absorption correction: multi-scan	$\theta_{max} = 25.0°$
(*SADABS*; Sheldrick, 1996)	$h = -11 \rightarrow 11$
$T_{min} = 0.965$, $T_{max} = 0.978$	$k = -11 \rightarrow 8$
5353 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.050$	$+ 0.4833P]$
$wR(F^2) = 0.127$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} = 0.002$
3607 reflections	$\Delta\rho_{max} = 0.30$ e Å^{-3}
264 parameters	$\Delta\rho_{min} = -0.36$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

S1—O1	1.413 (2)	O3—C17	1.215 (3)
S1—O2	1.430 (2)	O4—C12	1.363 (3)
S1—N1	1.611 (2)	O4—C15	1.415 (4)
S1—C1	1.758 (3)	N1—C8	1.467 (3)
O1—S1—O2	120.06 (15)	O2—S1—C1	107.40 (13)
O1—S1—N1	107.86 (12)	N1—S1—C1	107.75 (12)
O2—S1—N1	105.50 (13)	C8—N1—S1	122.13 (18)
O1—S1—C1	107.74 (14)		
O1—S1—N1—C8	−39.4 (2)	S1—N1—C8—C16	−79.2 (3)
O2—S1—N1—C8	−168.84 (19)	N1—C8—C16—C17	−50.6 (3)
C1—S1—N1—C8	76.7 (2)	C8—C16—C17—O3	−18.0 (3)
S1—N1—C8—C9	148.75 (18)	C8—C16—C17—C18	159.4 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^i	0.86	2.30	3.019 (3)	141

Symmetry code: (i) $1 - x, -y, -z$.

All H atoms were positioned geometrically and refined as riding (N—H = 0.86 Å and C—H = 0.93–0.98 Å). For NH, CH and CH$_2$ groups, U_{iso}(H) values were set equal to $1.2U_{eq}$(carrier atom) and for the methyl groups they were set equal to $1.5U_{eq}$(carrier atom).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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