Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3-(*p*-Tolyl)-1-phenyl-3-(*p*-tolylsulfonylamino)-propan-1-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.058
wR factor = 0.165
Data-to-parameter ratio = 16.4

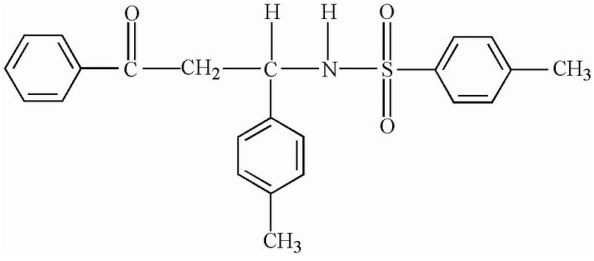
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 July 2004
Accepted 27 July 2004
Online 7 August 2004

The S atom of the sulfonyl group in the title compound, $C_{23}H_{23}NO_3S$, has a distorted tetrahedral geometry. The amino group forms an intermolecular hydrogen bond with the carbonyl O atom of an adjacent molecule.

Comment

β-Aminoketones are an important class of synthetic intermediates in organic synthesis. Most β-aminoketones are synthesized by the Mannich reaction (Miura *et al.*, 2000; Ranu *et al.*, 2002). However, we have recently investigated a new metal-mediated reaction for the preparation of β-aminoketones (Shim & Yamamoto, 2000) with 2-bromoacetophenone in the presence of activated zinc powder. We report here the synthesis and structure of 3-(*p*-tolyl)-1-phenyl-3-(*p*-tolylsulfonylamino)propan-1-one, (I). The structure determination of (I) was undertaken as part of our studies on the above-mentioned reaction.



(I)

The molecular structure of (I) and the atom-numbering scheme are shown in Fig. 1. In this structure, the benzene rings attached to the sulfonyl and carbonyl groups make dihedral angles of 85.5 (4) and 100.9 (4)°, respectively, with the third ring. Intermolecular hydrogen bonds are formed (Table 2) between the NH group and the carbonyl O atom of an adjacent molecule.

Experimental

To a solution of *N*-[(*p*-tolyl)methylene]-*p*-toluenesulfonamide (1.0 mmol) in dichloromethane (5.0 ml) was added 2-bromoacetophenone (1.5 mmol). Zinc powder (3.0 mmol) and a trace amount of iodine were added to the mixture. After the reaction mixture had been refluxed with stirring for 11 h and quenched with a saturated solution of NH_4Cl (5.0 ml) and 25% NH_4OH (5.0 ml), the mixture was extracted with dichloromethane. The extract was washed with water and dried over magnesium sulfate. After evaporation of the solvent, a white powder was obtained (yield 69%) by flash chroma-

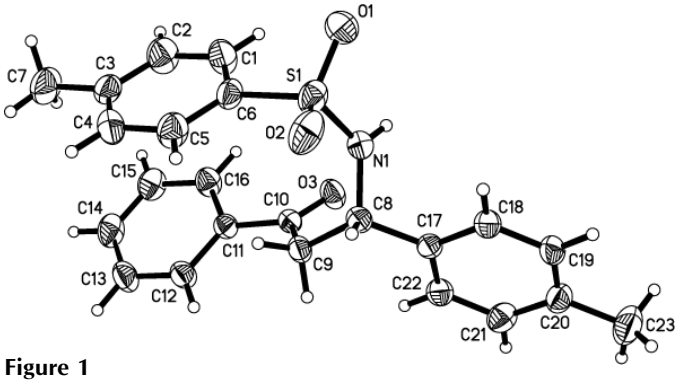


Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 35% probability level.

tography (ethyl acetate–trichloromethane). Slow evaporation of an ethyl acetate–petroleum ether mixture afforded the title compound as a crystalline solid (m.p. 376–377 K). Spectroscopic analysis, IR (KBr, ν, cm^{-1}): 3246, 1679, 1334, 1160; ^{1}H NMR ($CDCl_3$, p.p.m.): 7.82–7.00 (*m*, 13H), 5.60 (*br*, 1H), 4.81 (*m*, 1H), 3.59 (*dd*, 1H), 3.45 (*dd*, 1H), 2.37 (*s*, 3H), 2.26 (*s*, 3H). Analysis required for $C_{23}H_{23}NO_3S$: C 70.20, H 5.89, N 3.56%; found: C 70.14, H 5.92, N 3.52%.

Crystal data

$C_{23}H_{23}NO_3S$
M_r = 393.49
Triclinic, $P\overline{1}$
a = 9.939 (3) Å
b = 9.988 (3) Å
c = 11.546 (4) Å
α = 80.848 (5)°
β = 86.550 (6)°
γ = 62.444 (5)°
V = 1003.1 (6) Å^3
Z = 2
D_x = 1.303 Mg m^{-3}
Mo Kα radiation
Cell parameters from 953 reflections
θ = 2.3–23.5°
μ = 0.19 mm^{-1}
T = 293 (2) K
Prism, colorless
0.42 × 0.36 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.925, T_{max} = 0.967
5815 measured reflections
4191 independent reflections
2674 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 26.6°
$h = -12 \rightarrow 11$
$k = -12 \rightarrow 11$
$l = -14 \rightarrow 7$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.165
S = 1.01
4191 reflections
255 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0725P)^2 + 0.5148P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.408 (3)	S1—C6	1.761 (3)
S1—O1	1.429 (3)	O3—C10	1.220 (3)
S1—N1	1.615 (3)	N1—C8	1.455 (3)
O2—S1—O1	120.94 (16)	O2—S1—C6	107.43 (15)
O2—S1—N1	107.33 (14)	O1—S1—C6	106.75 (14)
O1—S1—N1	105.90 (15)	N1—S1—C6	107.93 (13)
O2—S1—N1—C8	34.3 (2)	O2—S1—C6—C5	−9.7 (3)
O1—S1—N1—C8	164.7 (2)	O1—S1—C6—C5	−140.8 (3)
C6—S1—N1—C8	−81.2 (2)	N1—S1—C6—C5	105.7 (3)
O2—S1—C6—C1	171.0 (3)	S1—N1—C8—C9	80.9 (3)
O1—S1—C6—C1	39.9 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	2.32	3.052 (3)	144

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

All H atoms were positioned geometrically and refined as riding (N—H = 0.86 Å and C—H = 0.93–0.98 Å). For the 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*.

The authors thank the State Key Laboratory of Elemento-Organic Chemistry, Nankai University. This research was supported by the Visiting Scholar Foundation of the Key Laboratory in that university. The authors thank Tianjin University for support of the work by the '985' Project.

References

Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Miura, K., Tamaki, K., Nakagawa, T. & Hosomi, A. (2000). *Angew. Chem. Int. Ed.* **39**, 1958–1960.
Ranu, B. C., Samanta, S. & Guchhait, S. K. (2002). *Tetrahedron*, **58**, 983–988.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97. University of Göttingen, Germany.
Shim, J. G. & Yamamoto, Y. (2000). *Heterocycles*, **52**, 885–895.