Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

3-(4-Chlorophenyl)-1-phenyl-3-(*p*-tolylsulfonyl-amino)propan-1-one

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Received 19 August 2004
Accepted 6 September 2004
Online 11 September 2004

Key indicators

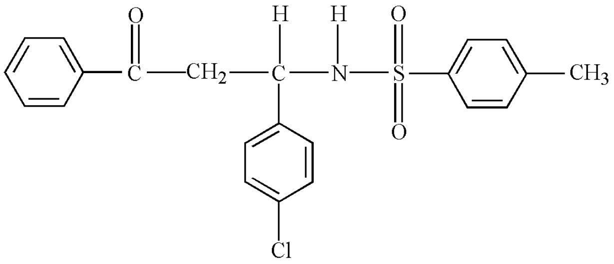
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.148
Data-to-parameter ratio = 16.5

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

The title compound, $C_{22}H_{20}ClNO_3S$, was synthesized by the Reformatsky reaction of *N*-[(4-chlorophenyl)methylene]-4-methylbenzenesulfonamide and 2-bromoacetophenone. The S atom in the sulfonyl group has a distorted tetrahedral geometry. Two intermolecular N—H···O hydrogen bonds are formed between the amine and sulfonyl groups of two molecules related by a center of symmetry.

Comment

The addition of nucleophilic reagents to unsaturated carbon–nitrogen bonds is a common practice in the synthesis of amine derivatives (Kobayashi & Ishitani, 1999). In our laboratory, we have recently investigated a new Reformatsky addition reaction to *N*-sulfonylimines with zinc enolates of ketones to give the corresponding β-sulfonaminoketones. A new compound, namely the title compound, (I), has been synthesized by the reaction of *N*-[(4-chlorophenyl)methylene]-4-methylbenzenesulfonamide (Shim & Yamamoto, 2000) with 2-bromoacetophenone in the presence of active zinc powder. An X-ray crystal structure determination of (I) was carried out in order to elucidate the structure, and the results are presented here.



(I)

The molecular structure of (I) is illustrated in Fig. 1. The S atom has a distorted tetrahedral geometry, with the O3—S1—O2 [119.2 (1)°] and O2—S1—N1 [105.0 (1)°] angles deviating significantly from the regular tetrahedral value. In this structure, the ring attached to the sulfonyl group and the 4-chlorophenyl ring make dihedral angles of 94.5 (3) and 103.2 (3)°, respectively, with the carbonylphenyl ring. Two molecules related by a center of symmetry are linked by two intermolecular N—H···O hydrogen bonds (Table 2), resulting in the formation of a dimer.

Experimental

To a solution of *N*-[(4-chlorophenyl)methylene]-4-methylbenzenesulfonamide (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 was refluxed with stirring for 11 h and quenched with a saturated aqueous solution of NH_4Cl (5.0 ml) and 25% ammonia (5.0 ml), the mixture was extracted with dichloromethane. The extract was washed with water and dried over magnesium sulfate. After the solvent was evaporated, a white powder was obtained (yield 86%) by flash chromatography (ethyl acetate–trichloromethane). Slow evaporation of an ethyl acetate–petroleum ether mixture afforded the title compound as a crystalline solid (m.p. 401–403 K). IR (KBr, cm^{-1}): ν 3261, 1690, 1318, 1154; ^{1}H NMR ($CDCl_3$, p.p.m.): δ 7.81–7.07 (*m*, 13H), 5.73 (*br*, 1H), 4.81 (*m*, 1H), 3.52 (*dd*, 1H), 3.40 (*dd*, 1H), 2.36 (*s*, 3H). Analysis calculated for $C_{22}H_{20}ClNO_3S$: C 63.78, H 4.83, N 3.38%; found: C 63.84, H 4.87, N 3.35%.

Crystal data

$C_{22}H_{20}ClNO_3S$
$M_r = 413.91$
Monoclinic, $P2_1/c$
$a = 15.975\ (5)$ Å
$b = 9.654\ (3)$ Å
$c = 14.566\ (4)$ Å
$\beta = 115.752\ (4)^\circ$
$V = 2023.3\ (11)$ Å^3
$Z = 4$

$D_x = 1.359$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 923 reflections
$\theta = 3.1$–25.0°
$\mu = 0.32$ mm^{-1}
$T = 293\ (2)$ K
Prism, colorless
0.40 × 0.34 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.882$, $T_{max} = 0.945$
11 492 measured reflections

4185 independent reflections
2521 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$
$\theta_{max} = 26.5^\circ$
$h = -17 \rightarrow 20$
$k = -12 \rightarrow 6$
$l = -18 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.148$
$S = 1.06$
4185 reflections
253 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.077P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O3	1.426 (2)	S1—C16	1.758 (3)
S1—O2	1.440 (2)	O1—C9	1.212 (3)
S1—N1	1.608 (2)	N1—C7	1.475 (3)
O3—S1—O2	119.2 (1)	O2—S1—C16	107.3 (1)
O3—S1—N1	107.9 (1)	N1—S1—C16	108.6 (1)
O2—S1—N1	105.0 (1)	C7—N1—S1	123.0 (2)
O3—S1—C16	108.5 (1)		
O3—S1—N1—C7	42.4 (2)	S1—N1—C7—C6	78.1 (2)
O2—S1—N1—C7	170.5 (2)	N1—C7—C8—C9	67.8 (3)
C16—S1—N1—C7	−74.9 (2)	C7—C8—C9—O1	−0.3 (4)
S1—N1—C7—C8	−157.12 (17)	C7—C8—C9—C10	178.4 (2)

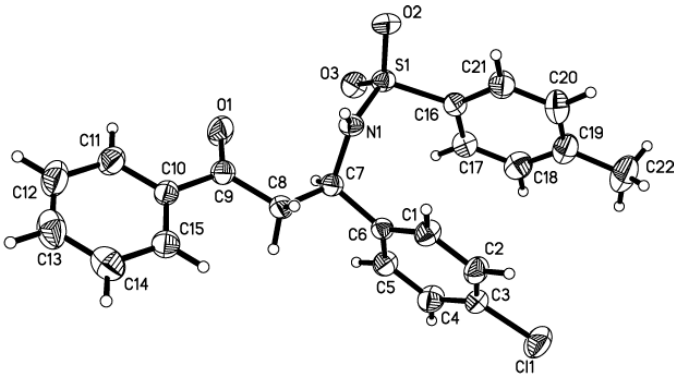


Figure 1
View of the molecule of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 35% probability level.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O2^{i}	0.86	2.18	2.951 (3)	149

Symmetry code: (i) $-x, 1-y, 2-z$.

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 at $1.2U_{eq}$(carrier atom), and for the methyl groups they were set at $1.5U_{eq}$(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 Key Lab in the university. The authors thank Tianjin University. The work was also supported by the "985" Project of Tianjin University.

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