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3-(3,4-Methylenedioxyphenyl)-1-(2-furyl)-3-(*p*-toluenesulfonylamino)propan-1-one

Zhifang Yu,* Yi Liu, Xiuyan Gu and Bing Zhao

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

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

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The S atom of the sulfonyl group in the title compound, $C_{21}H_{19}NO_6S$, has a distorted tetrahedral geometry. The amino groups and sulfonyl groups are connected by intermolecular N—H···O hydrogen bonds. The furan ring is disordered in one of the two independent molecules in the asymmetric unit.

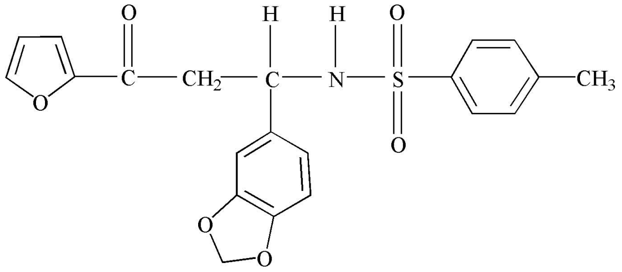
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.061
wR factor = 0.195
Data-to-parameter ratio = 12.4

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

Comment

The Reformatsky addition reaction to imines (Gilman & Speeter, 1943) has been employed as a convenient method for synthesizing β-lactams, β-amino acids and their derivatives. We have recently investigated a new addition reaction to *N*-sulfonylimines with zinc enolates to give the corresponding β-sulfonaminoketones. The title compound, (I), was synthesized by the reaction of 2-(bromoacetyl)furan and *N*-[(3,4-methylenedioxyphenyl)methylene]-4-methylbenzenesulfonamide (Shim & Yamamoto, 2000) 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) and the atom-numbering scheme are shown in Fig. 1. The asymmetric unit of (I) contains two independent molecules. In one molecule, the sulfonylphenyl ring and the 3,4-methylenedioxyphenyl ring are inclined at an angle of 18.9 (3)°; the dihedral angle is 12.9 (3)° in the second molecule. The S atom in each independent molecule has a distorted tetrahedral geometry with angles deviating significantly from the regular tetrahedral value (Table 1). The two independent molecules form two centrosymmetric dimers connected by intermolecular N—H···O hydrogen bonds between the amino and sulfonyl groups (Table 2).

Experimental

2-(Bromoacetyl)furan (1.5 mmol) was added to a solution of *N*-[(3,4-methylenedioxyphenyl)methylene]-4-methylbenzenesulfonamide (1.0 mmol) in dichloromethane (5.0 ml). Zinc powder (3.0 mmol) and a trace amount of iodine were added to the mixture. The reaction mixture was refluxed with stirring for 11 h and dried over magnesium sulfate. After the solvent was evaporated, a white powder was obtained (yield 58%) by flash chromatography (ethyl acetate–trichloromethane). Recrystallization by slow evaporation of an ethyl acetate–petroleum ether mixture afforded the compound as a crystalline solid (m.p.399–401 K). Spectroscopic analysis: IR (KBr, νcm^{-1}): 3247, 1671, 1326, 1157; ^{1}H NMR (CDCl$_3$, δ, p.p.m.): 7.61–7.11 (*m*, 7H), 6.61 (*s*, 2H), 5.90–6.51 (*m*, 3H), 5.66 (*br*, 1H), 4.71 (*q*, 1H), 3.35 (*dd*, *J* = 6.0, 16.4 Hz, 1H), 3.24 (*dd*, *J* = 6.0, 16.8 Hz, 1H), 2.37 (*s*, 3H).

Crystal data

$C_{21}H_{19}NO_6S$
$M_r = 413.44$
Monoclinic, $P2_1/n$
$a = 15.721$ (4) Å
$b = 12.082$ (3) Å
$c = 21.386$ (6) Å
$\beta = 104.346$ (6)°
$V = 3935.4$ (18) Å^3
$Z = 8$

$D_x = 1.396$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 890 reflections
$\theta = 2.5$–21.0°
$\mu = 0.20$ mm^{-1}
$T = 293$ (2) K
Prism, colorless
0.30 × 0.24 × 0.22 mm

Data collection

Bruker SMART CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.943$, $T_{\max} = 0.956$
20215 measured reflections

6939 independent reflections
3691 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.051$
$\theta_{\max} = 25.0°$
$h = -16 \rightarrow 18$
$k = -14 \rightarrow 14$
$l = -25 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.195$
$S = 1.00$
6939 reflections
560 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0807P)^2 + 4.1226P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.50$ e Å^{-3}
$\Delta\rho_{\min} = -0.79$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—O4	1.419 (3)	S2—O10	1.419 (3)
S1—O3	1.442 (3)	S2—O9	1.432 (3)
S1—N1	1.616 (4)	S2—N2	1.613 (3)
S1—C15	1.753 (4)	S2—C36	1.754 (4)
N1—C7	1.471 (5)	N2—C28	1.470 (5)
O4—S1—O3	118.6 (2)	O9—S2—N2	103.61 (18)
O4—S1—N1	109.5 (2)	O10—S2—C36	107.14 (19)
O3—S1—N1	104.17 (19)	O9—S2—C36	109.5 (2)
O4—S1—C15	107.6 (2)	N2—S2—C36	108.22 (18)
O3—S1—C15	108.8 (2)	C28—N2—S2	124.2 (3)
N1—S1—C15	107.64 (18)	C32—O11—C35	105.8 (3)
C7—N1—S1	120.9 (3)	C31—O12—C35	106.2 (3)
O10—S2—O9	119.0 (2)	C22—O7—C25	100.0 (7)
O10—S2—N2	109.0 (2)	C23—C22—O7	115.2 (8)
O4—S1—N1—C7	57.8 (3)	S2—N2—C28—C29	96.9 (4)
O3—S1—N1—C7	−174.4 (3)	S2—N2—C28—C27	−136.7 (3)
C15—S1—N1—C7	−58.9 (4)	O10—S2—C36—C41	−174.5 (4)
S1—N1—C7—C8	93.6 (4)	O9—S2—C36—C41	55.2 (4)
S1—N1—C7—C6	−140.5 (3)	N2—S2—C36—C41	−57.1 (4)
O10—S2—N2—C28	57.2 (3)	O10—S2—C36—C37	3.0 (4)
O9—S2—N2—C28	−175.2 (3)	O9—S2—C36—C37	−127.3 (4)
C36—S2—N2—C28	−59.0 (4)	N2—S2—C36—C37	120.4 (4)

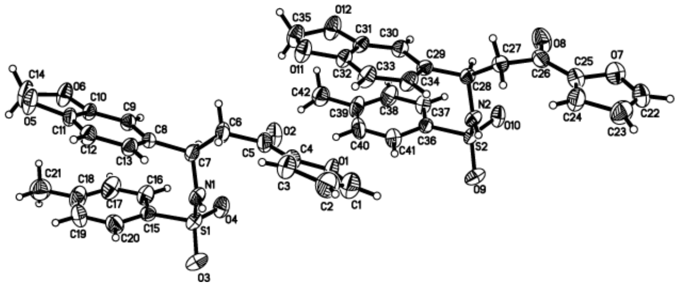


Figure 1
View of the asymmetric unit of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. Only one disorder component is shown.

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1$\cdots$O3$^{\rm i}$	0.86	2.26	2.957 (5)	137.8
N2—H2$\cdots$O9$^{\rm ii}$	0.86	2.21	3.012 (5)	154.5

Symmetry codes: (i) $1 - x, 2 - y, -z$; (ii) $-x, 1 - y, -z$.

All H atoms were located in a difference Fourier map and were refined as riding (N—H = 0.86 Å and C—H = 0.93–0.98 Å). For NH, CH and CH$_2$ groups, $U_{\rm iso}$(H) values were set equal to 1.2$U_{\rm eq}$(carrier atom) and for the methyl groups they were set equal to 1.5$U_{\rm eq}$(carrier atom). In one molecule, the furyl ring is disordered over two sites. The occupancies are 0.62 (2) and 0.38 (2).

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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