organic papers

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Key indicators

Single-crystal X-ray study

$T = 293$ K

Mean σ(C–C) = 0.005 Å

R factor = 0.062

wR factor = 0.147

Data-to-parameter ratio = 15.6

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

3-(3,4-Methylenedioxyphenyl)-1-phenyl-3-(p-toluenesulfonylamino)propan-1-one

The title compound, $C_{23}H_{21}NO_5S$, was synthesized by the Reformatsky reaction of N-[(3,4-methylenedioxyphenyl)-methylene]-4-methylbenzenesulfonamide and 2-bromoaceto-phenone. The S atom in the sulfonyl group has a distorted tetrahedral geometry. The two independent molecules in the asymmetric unit form two centrosymmetric dimers connected by intermolecular N—H···O hydrogen bonds between the amino and sulfonyl groups.

Comment

Since Gilman & Speeter (1943) reported the Reformatsky addition reaction to imines, it has been employed as a method of synthesizing β-lactams, β-amino acids and their derivatives. In our laboratory, we have recently investigated a new Reformatsky addition reaction to N-sulfonylimines for the preparation of β-sulfonylamino ketones. A new compound, namely 3-(3,4-methylenedioxyphenyl)-1-phenyl-3-(p-toluene-sulfonylamino)propan-1-one, (I), has been synthesized by the reaction of N-[(3,4-methylenedioxyphenyl)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)
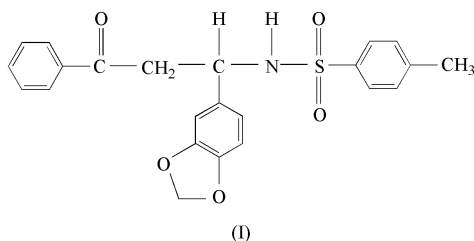
The asymmetric part of the asymmetric unit of (I) contains two independent molecules (Fig. 1). In one molecule, the sulfonylphenyl ring (A) and the 3,4-methylenedioxyphenyl
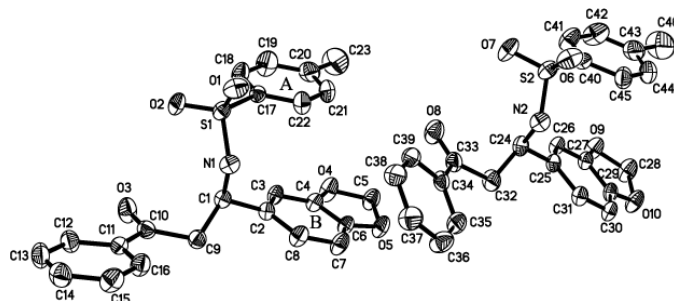
Figure 1

View of the asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

ring (*B*) are inclined at an angle of 11.0 (3)°. Rings *A* and *B* make dihedral angles of 8.5 (3) and 14.7 (3)°, respectively, with the carbonylphenyl ring, different from what is seen in 3-(4-methoxyphenyl)-1-phenyl-3-(*p*-toluenesulfonylamino)-propan-1-one (Yu *et al.*, 2004); the dihedral angles are 97.5 (3) and 85.8 (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-Bromoacetophenone (1.5 mmol) was added to a solution of *N*-[(3,4-methylenedioxyphenyl)methylene]-4-methylbenzenesulfon-amide (1.0 mmol) in dichloromethane (5.0 ml). This mixture was added to a 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 yellow powder was obtained (yield 50%) and single crystals suitable for crystallographic analysis were obtained by slow evaporation of an ethyl acetate–petroleum ether solution (m.p. 396–398 K). Spectroscopic analysis, IR (KBr, v cm^{-1}): 3242, 1678, 1326, 1155; ^{1}H NMR (CDCl$_3$, p.p.m.): 7.82–7.18 (*m*, 12H), 6.61 (*s*, 2H), 5.59 (*br*, 1H), 4.74 (*m*, 1H), 3.50 (*dd*, 1H), 3.43 (*dd*, 1H), 2.37 (*s*, 3H). Analysis required for $C_{23}H_{21}NO_5S$: C 65.18, H 4.96, N 3.31%; found: C 65.13, H 4.91, N 3.28%.

Crystal data

$C_{23}H_{21}NO_5S$	$D_x = 1.372$ Mg m^{-3}
$M_r = 423.48$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 796
$a = 15.968$ (5) Å	reflections
$b = 12.342$ (4) Å	$\theta = 3.3–21.7°$
$c = 21.607$ (7) Å	$\mu = 0.19$ mm^{-1}
$\beta = 105.682$ (6)°	$T = 293$ (2) K
$V = 4100$ (2) Å^3	Prism, yellow
$Z = 8$	$0.26 \times 0.24 \times 0.18$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	8458 independent reflections
φ and ω scans	3849 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.073$
$T_{min} = 0.951$, $T_{max} = 0.966$	$\theta_{max} = 26.5°$
23196 measured reflections	$h = -13 \rightarrow 20$
	$k = -15 \rightarrow 15$
	$l = -27 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.062$	$+ 0.3065P]$
$wR(F^2) = 0.147$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{max} = 0.002$
8458 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
543 parameters	$\Delta\rho_{min} = -0.29$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

S1—O2	1.423 (2)	O4—C5	1.413 (4)
S1—O1	1.440 (2)	O5—C6	1.374 (3)
S1—N1	1.611 (3)	O5—C5	1.427 (4)
S1—C17	1.754 (3)	O8—C33	1.213 (4)
S2—O7	1.415 (2)	O9—C27	1.364 (3)
S2—O6	1.437 (2)	O9—C28	1.420 (4)
S2—N2	1.618 (3)	O10—C29	1.377 (3)
S2—C40	1.757 (3)	O10—C28	1.424 (4)
O3—C10	1.210 (4)	N1—C1	1.471 (4)
O4—C4	1.378 (3)	N2—C24	1.475 (4)
O2—S1—O1	119.38 (14)	O7—S2—O6	119.19 (15)
O2—S1—N1	109.04 (14)	O7—S2—N2	109.63 (16)
O1—S1—N1	104.09 (13)	O6—S2—N2	103.99 (14)
O2—S1—C17	106.52 (14)	O7—S2—C40	107.21 (15)
O1—S1—C17	108.98 (15)	O6—S2—C40	108.78 (16)
N1—S1—C17	108.46 (14)	N2—S2—C40	107.56 (15)
O2—S1—N1—C1	55.2 (3)	S1—N1—C1—C2	98.4 (3)
O1—S1—N1—C1	−176.3 (2)	N1—C1—C9—C10	76.6 (3)
C17—S1—N1—C1	−60.4 (3)	C1—C9—C10—O3	43.3 (4)
O7—S2—N2—C24	53.9 (3)	C1—C9—C10—C11	−138.5 (3)
O6—S2—N2—C24	−177.6 (2)	S2—N2—C24—C25	93.1 (3)
C40—S2—N2—C24	−62.4 (3)	S2—N2—C24—C32	−141.7 (2)
S1—N1—C1—C9	−135.8 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^i	0.86	2.17	3.007 (3)	163
N2—H2···O6^{ii}	0.86	2.20	2.929 (4)	142

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

All H atoms were positioned geometrically and refined as riding (N—H = 0.86 Å and C—H = 0.93–0.98 Å), with U_{iso} values set at 1.2 (NH, CH and CH$_2$) or 1.5 (CH$_3$) times U_{eq} of the parent atom.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SAINT*; 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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