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N-Benzoyl-*N*′-methyl-*N*′-(*p*-tolylsulfonyl)hydrazine

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.100
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.

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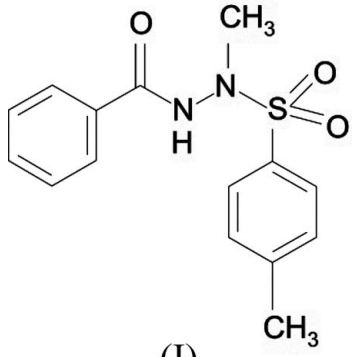
The crystal structure of the title compound, $C_{15}H_{16}N_2O_3S$, contains chains running along the *a* axis which are formed *via* intermolecular N—H···O hydrogen bonds. The side chain comprising the N–N–C(=O)–C sequence of atoms is planar to within 0.0172 (14) Å and the benzene rings are inclined at 33.09 (11)° to one another.

Comment

Sulfonamides constitute an important class of drugs with diverse biological activities, such as anti-HIV, antidiabetic, carbonic-anhydrase inhibitory, antimicrobial and antitumour activities (Nishimori *et al.*, 2006; Turner, 2002; Supuran & Scozzafava, 2000, 2001, 2003). In continuation of our interest in the chemical and pharmacological properties of benzenesulfonamides and 1,3,4-oxadiazole derivatives (Zareef *et al.*, 2006*a*,*b*), a novel benzenediazasulfonamide was obtained during an attempt to reduce [5-(phenyl)-1,3,4-oxadiazol-2-ylthio]acetamide with sodium acyloxyborohydride (Norihide *et al.*, 1976). During the reduction reaction, the heterocyclic ring, 1,3,4-oxadiazole, has been cleaved. The product was further reacted with tosyl chloride to produce the title compound, (I), and the structure of this compound is presented here.



(I)

The crystal structure is composed of discrete molecules of (I) (Fig. 1), forming chains along the *a* axis *via* strong intermolecular N—H···O hydrogen bonds (Fig. 2); details of the hydrogen-bonding geometry are provided in Table 1. The hydrogen-bonding pattern observed in the structure of (I) is in contrast with those observed in 4-methyl-*N*-[1-(5-thioxo-4,5-dihydro-1,3,4-oxadiazol-2-yl)propyl]benzenesulfonamide (Zareef *et al.*, 2006*a*), where the tosylate O atoms are involved in hydrogen bonds, and in *N*-{[1-(5-benzylsulfanyl)-1,3,4-oxadiazol-2-yl]ethyl}-4-chlorobenzenesulfonamide (Zareef *et al.*, 2006*b*), where one of the N atoms in the heterocyclic ring is

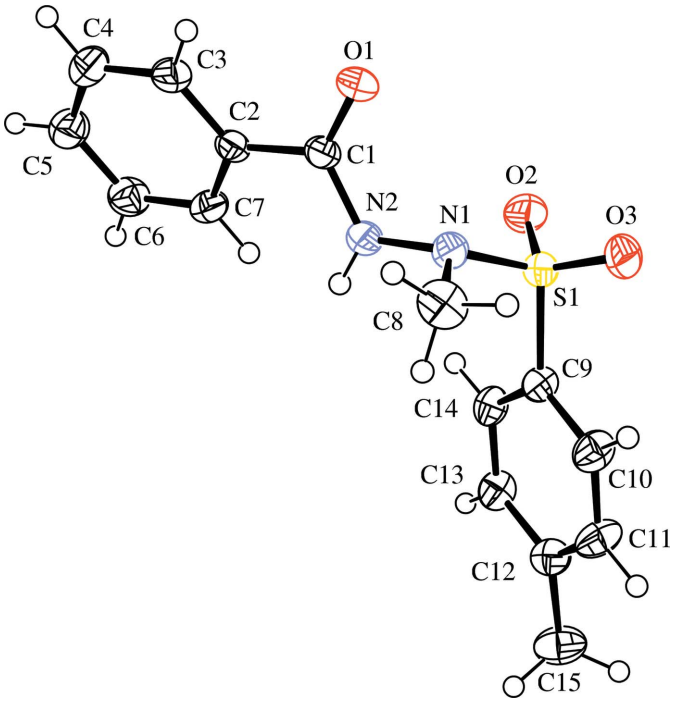


Figure 1
The molecular structure of (I), with displacement ellipsoids plotted at the 50% probability level.

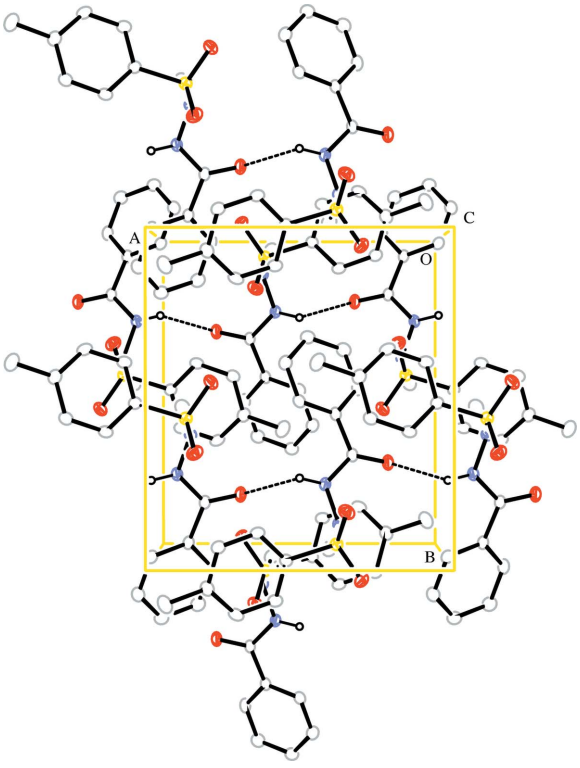


Figure 2
The packing of (I), showing N—H···O hydrogen bonds as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

involved in N—H···N hydrogen bonds. Both of these compounds are closely related to (I).

The molecular dimensions in (I) are as expected, with distances S1—N1 = 1.655 (3) Å and S1—C9 = 1.756 (3) Å, and agree with the corresponding distances reported in the structures quoted above, as well as those observed in *N*′-(benzenesulfonyl)-4-methylthiazole-5-carbohydrazide (Song *et al.*, 2004). The side chain comprising atoms N1/N2/C1/C2 is planar to within 0.0172 (14) Å and makes an angle of 21.9 (2)° with respect to the phenyl ring C2–C7. The mean planes of the two benzene rings are inclined at an angle of 33.09 (11)° to each other.

Experimental

The title compound was obtained in six steps. 2-Mercapto-5-phenyl-1,3,4-oxadiazole was prepared by the reported method (Ahmad *et al.*, 2001) from benzoic acid hydrazide. The free mercapto (SH) at position 2 of the oxadiazole ring was converted to its ethyl ester with ethyl bromoacetate in a basic medium, following the reported procedure (Mir & Siddiqui, 1970). 2-Mercapto-5-phenyl-1,3,4-oxadiazole (20 mmol) was dissolved in an aqueous solution of sodium hydrogen carbonate (20 mmol in 75 ml of water) by stirring at 305 K, followed by addition of ethyl bromoacetate (20 mmol in 10 ml of absolute ethanol). The resulting mixture was stirred at 305 K for 5 h to form the desired ethyl ester. The [5-(phenyl)-1,3,4-oxadiazol-2-ylthio]acetamide was prepared from its ethyl ester by reacting with dry ammonia in ethanol (Furniss *et al.*, 1978). To a stirred suspension of [5-(phenyl)-1,3,4-oxadiazol-2-ylthio]acetamide (10 mmol) and sodium borohydride (50 mmol) in dioxane (25 ml) was added acetic acid (50 mmol) in dioxane (10 ml) over a period of 10 min at 278–283 K, and the resulting mixture was stirred under reflux for 2 h. Following the reported treatment of the mixture (Norihide *et al.*, 1976), the resulting product was treated with tosyl chloride in the presence of triethylamine to yield the title compound, (I). Crystals suitable for crystallographic study were grown from a solution of (I) in ethanol by slow evaporation at room temperature (305 K).

Crystal data

$C_{15}H_{16}N_2O_3S$
M_r = 304.36
Orthorhombic, $Pna2_1$
a = 9.839 (6) Å
b = 10.937 (4) Å
c = 13.836 (8) Å
V = 1488.9 (14) Å^3
Z = 4
D_x = 1.358 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 173 (2) K
Block, colourless
0.18 × 0.16 × 0.12 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.960, T_{max} = 0.973
3178 measured reflections
3176 independent reflections
2603 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.100
S = 1.04
3176 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 0.72P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Flack (1983), with 1404 Friedel pairs
Flack parameter: 0.53 (9)

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.88	2.13	2.879 (3)	143

Symmetry code: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, z$.

Refinement of the Flack (1983) parameter indicates that the stucture contains roughly equal components of inversion twins. H atoms were included in the refinement in geometrically idealized positions, with N—H = 0.88 Å and C—H = 0.95–0.98 Å, and with U_{iso}(H) = 1.2U_{eq}(parent).

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

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