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4-Methyl-*N*-[1-(5-thioxo-4,5-dihydro-1,3,4-oxadiazol-2-yl)propyl]benzenesulfonamide

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The structure of the title compound, $C_{12}H_{15}N_3O_2S_2$, incorporating oxadiazole and sulfonamide units, contains two conformers in different proportions. The heterocyclic five-membered ring is inclined at 17.6 (1)° with respect to the benzene ring. The structure is stabilized by N—H···O hydrogen bonds.

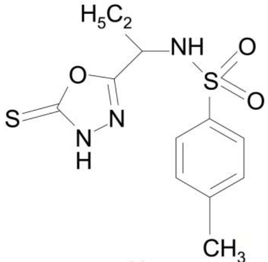
Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.048
wR factor = 0.117
Data-to-parameter ratio = 21.7

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

Comment

Based on the diverse biological activities of sulfonamides and 2,5-disubstituted-1,3,4-oxadiazoles (Turner, 2002; Supuran & Scozzafava, 2000, 2001, 2003; Masereel *et al.*, 2002; Singh *et al.*, 1997), we have designed and synthesized some new compounds that have both oxadiazole and sulfonamide entities in one molecule. Here, we report the structure of the title compound, (I).



(I)

The structure of (I) is composed of two conformers of (I) in unequal proportions; the major component is shown in Fig. 1. The oxadiazole ring is essentially planar and is inclined at 17.6 (1)° with respect to the benzene ring. The N2═C2 and S1═C1 double bonds (Table 1) agree with the corresponding distances in three structures containing similar systems reported in the Cambridge Structural Database [2006 Release (Allen, 2002); refcodes: AVULIM (Öztürk *et al.*, 2004), AVULUY (Du *et al.*, 2004) and YITMUJ (Ziyaev *et al.*, 1992)]. The structure is stabilized by a network of strong hydrogen bonds of the type N—H···O, involving both H atoms bonded to N atoms and the O atoms bonded to S2 (Table 2). However, it is interesting to note that N3 and N3′, in the major and minor conformers, are hydrogen bonded to O3 and N2, respectively, and may be responsible for the disorder, resulting in two conformations for (I).

Experimental

For the preparation of the title compound, (I), DL-2-(4-methylphenylsulfonamido)butanoic acid was esterified with ethanol in acidic media using a standard method (Furniss *et al.*, 1978). A mixture of DL-2-(4-methylphenylsulfonamido)butanoic acid (25 mmol), absolute ethanol (80 ml) and concentrated sulfuric acid (0.5 ml) was refluxed

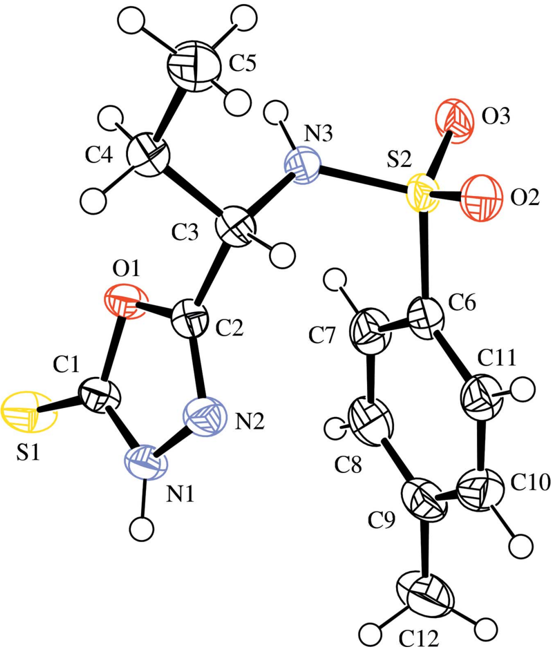


Figure 1
ORTEPII (Johnson, 1976) drawing of the major conformer of (I), with displacement ellipsoids drawn at the 50% probability level.

for 11 h to obtain the pure ester. A mixture of ethyl 2-(4-chlorophenylsulfonamido)butanoate (10 mmol) and hydrazine monohydrate (80%) in absolute ethanol (50 ml) was then refluxed for 9 h. The excess solvent was distilled off and the residue was filtered off, washed with water and recrystallized from 60% aqueous ethanol to yield 2-(4-methylphenylsulfonamido)butane hydrazide. Compound (I) was then obtained by the reaction of the hydrazide (5.5 mmol) in 80 ml absolute ethanol, with carbon disulfide (6.6 mmol) and aqueous potassium hydroxide (5.5 mmol) at reflux temperature for 16.5 h. The excess solvent was distilled off and the product was recrystallized by slow evaporation of an aqueous ethanol solution at room temperature to give a racemic mixture of (I).

Crystal data

$C_{12}H_{15}N_3O_3S_2$
$M_r = 313.39$
Monoclinic, $C2/c$
$a = 14.098$ (5) Å
$b = 16.575$ (8) Å
$c = 13.695$ (5) Å
$\beta = 114.05$ (2)°
$V = 2922$ (2) Å^3
$Z = 8$
$D_x = 1.425$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.37$ mm^{-1}
$T = 173$ (2) K
Block, colorless
0.18 × 0.16 × 0.14 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min} = 0.936$, $T_{\max} = 0.950$
5916 measured reflections
4257 independent reflections
3265 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 30.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.117$
$S = 1$
4257 reflections
196 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.038P)^2 + 4.48P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.53$ e Å^{-3}
$\Delta\rho_{\min} = -0.54$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.642 (2)	S2′—O3′	1.440 (2)
O1—C2	1.369 (2)	S2′—N3′	1.610 (2)
O1—C1	1.382 (2)	S2′—C6	1.766 (2)
S2—O3	1.440 (2)	N1—C1	1.330 (2)
S2—O2	1.445 (2)	N1—N2	1.387 (2)
S2—N3	1.615 (2)	N2—C2	1.279 (2)
S2—C6	1.766 (2)	N3—C3	1.450 (3)
S2′—O2′	1.439 (2)		
C2—O1—C1	106.02 (14)	O2′—S2′—N3′	112.6 (10)
O3—S2—O2	117.87 (11)	O3′—S2′—N3′	112.6 (10)
O3—S2—N3	105.07 (11)	O2′—S2′—C6	104.6 (7)
O2—S2—N3	108.03 (11)	O3′—S2′—C6	96.7 (9)
O3—S2—C6	110.29 (11)	N3′—S2′—C6	98.8 (7)
O2—S2—C6	107.04 (10)	C1—N1—N2	112.98 (15)
N3—S2—C6	108.20 (10)	C2—N2—N1	103.17 (15)
O2′—S2′—O3′	125.6 (11)	C3—N3—S2	122.00 (14)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O2$^{\rm i}$	0.88	1.96	2.840 (2)	173
N1—H1···O2′$^{\rm i}$	0.88	1.83	2.615 (10)	148
N3—H3···O3$^{\rm ii}$	0.88	2.06	2.920 (3)	165
N3′—H3′···N2$^{\rm iii}$	0.88	2.44	3.141 (14)	137

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $-x + 2, -y + 1, -z + 1$; (iii) $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 1$.

Atoms S2, O2, O3 and N3 were disordered over two sites with occupancy factors of 0.898 (2) and 0.102 (2). Corresponding atoms in the minor occupancy fraction are labelled with primes. The bond lengths involving the primed atoms were restrained to be the same as those of the unprimed atoms. The remaining atoms were not affected by this disorder. H atoms were included in the refinement at geometrically idealized positions, with N—H = 0.88, C—H = 0.95–1.00 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$ of the atoms to which they were bonded.

Data collection: *COLLECT* (Hooft, 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*.

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