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S-*p*-Tolyl 3-methanesulfonyl-2-oxoimidazolidine-1-carbothioate

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Key indicators

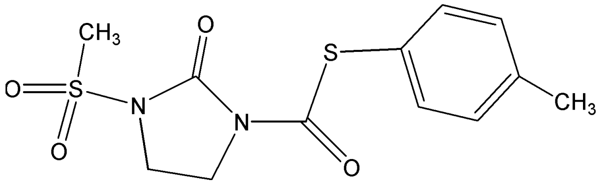
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.116
Data-to-parameter ratio = 14.3

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

In the title compound, $C_{12}H_{14}N_2O_4S_2$, the five atoms of the imidazolidine ring are nearly coplanar, the average deviation being 0.004 Å. This ring makes a dihedral angle of 79.54 (9)° with the plane of the benzene ring. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds and C—H···π interactions.

Comment

Thiol esters are important intermediates in organic synthesis and have been used as mild acyl-transfer reagents (Mukaiyama *et al.*, 1973), as intermediates in the synthesis of ketones (McGarvey *et al.*, 1986) and in asymmetric aldol reactions (Kobayashi *et al.*, 1991). Thiol esters also constitute an important group of natural products (Halcomb *et al.*, 1995). Some of them show physiological activity and are widely applied in medicines and pesticides; examples include Timobensone, which is a corticoid antibiotic, and compounds such as 2-phenyl-1-ethyl (2*S*)-1-(3,3-dimethyl-1,2-dioxopentyl)-2-pyrrolidinecarbothioate, used for the treatment of Parkinsonism (Hamilton & Li, 1999). Previously, we have reported the synthesis (Su *et al.*, 2002) and crystal structure of *S*-phenyl 3-methanesulfonyl-2-oxoimidazolidine-1-carbothioate (Li *et al.*, 2004). Since then, a new derivative of the former compound, *S*-*p*-tolyl 3-methanesulfonyl-2-oxoimidazolidine-1-carbothioate, (I), has been obtained and its crystal structure is reported here.



(I)

In (I), the r.m.s deviation of the benzene ring from planarity is 0.002 Å, which indicates that atom S1 has little influence on benzene-ring planarity. The S1—C8 and S1—C5 bond lengths are 1.785 (2) and 1.781 (2) Å, respectively, and are comparable to those found in *S*-phenyl 3-methanesulfonyl-2-oxoimidazolidine-1-carbothioate (Li *et al.*, 2004), where the S—C bond lengths are 1.7777 (18) and 1.7767 (18) Å, respectively. The five atoms (N1, C9, N2, C11 and C10) of the imidazolidine ring are essentially coplanar, with an r.m.s deviation of 0.004 Å. The dihedral angle between the imidazolidine and benzene rings is 79.54 (9)°, which is larger than that reported for the phenyl analog [40.77 (5)°]. In the crystal structure, there are two intermolecular C—H···O hydrogen bonds and a

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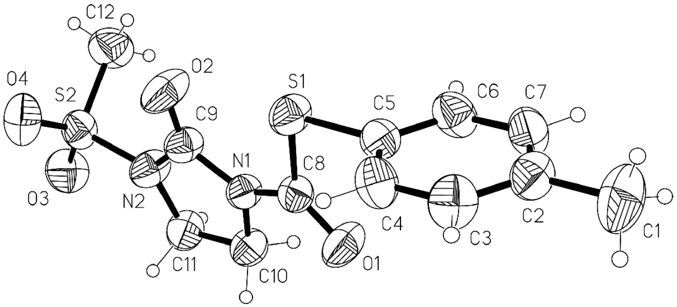

Figure 1
The molecule structure of (I), with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

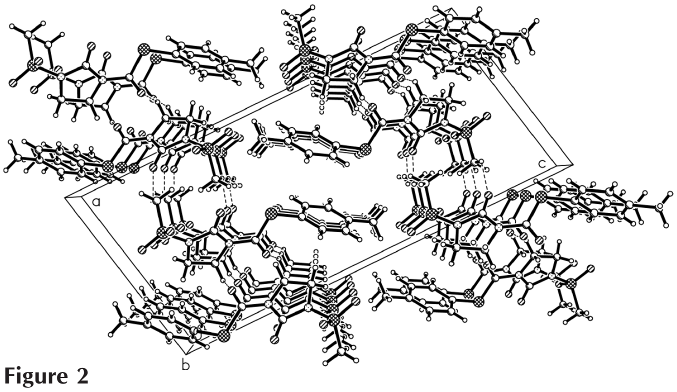

Figure 2
The three-dimensional network of (I). Hydrogen bonds are indicated by dashed lines.

C—H$\cdots\pi$ interaction involving the benzene ring (Table 1), resulting in the formation of a two-dimensional network structure (Fig. 2).

Experimental

The title compound was synthesized by the reaction of 3-methanesulfonyl-2-oxoimidazolidine-1-carbonyl chloride (2.27 g, 0.01 mol) and 4-methylbenzenethiol (1.24 g, 0.01 mol) in the presence of tetrahydrofuran (20 ml) with active Zn powder as catalyst (0.65 g). The reaction was carried out under reflux for 6 h. Single crystals were obtained by recrystallization from tetrahydrofuran.

Crystal data

$C_{12}H_{14}N_2O_4S_2$
$M_r = 314.37$
Monoclinic, $P2_1/n$
$a = 10.6170$ (8) Å
$b = 6.0354$ (5) Å
$c = 23.0654$ (17) Å
$\beta = 101.649$ (1)°
$V = 1447.54$ (19) Å^3
$Z = 4$
$D_x = 1.443$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2487 reflections
$\theta = 2.3$–25.0°
$\mu = 0.38$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.35 × 0.22 × 0.17 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.878$, $T_{\max} = 0.938$
7344 measured reflections
2620 independent reflections
2309 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 25.3°$
$h = -9 \rightarrow 12$
$k = -7 \rightarrow 7$
$l = -25 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.117$
$S = 1.08$
2620 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.7722P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.28$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C12—H12$B\cdots$O2$^{\rm i}$	0.96	2.55	3.280 (4)	133
C10—H10$A\cdots$O1$^{\rm ii}$	0.97	2.54	3.037 (3)	112
C11—H11$B\cdots Cg$P$^{\rm ii}$	0.97	2.80	3.767 (3)	176

Symmetry codes: (i) $-\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$. CgP is the centroid of the benzene ring.

The H atoms were positioned geometrically and allowed to ride on their parent atoms, with Csp^2—H = 0.93 Å and $U_{\rm iso} = 1.2U_{\rm eq}$(parent atom), and C$sp^3$—H = 0.96 or 0.97 Å and $U_{\rm iso} = 1.5U_{\rm eq}$(parent atom).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); 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: *XP* (Bruker, 2002); software used to prepare material for publication: *SHELXL*97.

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