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S-(4-Methoxyphenyl) 3-methylsulfonyl-2-oxoimidazolidine-1-carbothioate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.119
Data-to-parameter ratio = 13.4

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

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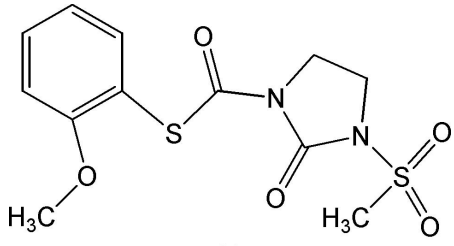
In the title compound, $C_{12}H_{14}N_2O_5S_2$, the five ring atoms of the imidazolidine are nearly coplanar, the average deviation being 0.077 Å. The imidazolidine and benzene rings make a dihedral angle of 73.98 (10)°. The crystal packing is stabilized by weak intermolecular C—H···O hydrogen bonds.

Comment

Thiol esters are important intermediates in organic synthesis (Mukaiyama *et al.*, 1973; McGarvey *et al.*, 1986; Kobayashi *et al.*, 1991). They constitute a group of natural products (Halcomb *et al.*, 1995). Some of them demonstrate 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 (Hamiton & Li, 1999). Previously, we have reported the synthesis (Su *et al.*, 2002) and crystal structure of *S*-*p*-tolyl 3-methylsulfonyl-2-oxoimidazolidine-1-carbothioate (Liang *et al.*, 2004). We present here a new derivative of a thiol ester, (I) (Fig. 1).



(I)

In (I), the bond lengths S1—C7 and S1—C8 are 1.776 (3) and 1.781 (3) Å, respectively, corresponding to those observed in *S*-*p*-tolyl 3-methylsulfonyl-2-oxoimidazolidine-1-carbothioate (Liang *et al.*, 2004) of 1.781 (2) and 1.785 (2) Å, respectively. The five atoms (N1, C9, N2, C10 and C11) of the imidazolidine ring are essentially coplanar, with an r.m.s. deviation of 0.077 Å. The dihedral angle between the imidazolidine and benzene rings is 73.98 (10)°. The crystal packing (Fig. 2) is stabilized by weak intermolecular C—H···O hydrogen bonds (Table 1).

Experimental

The title compound was synthesized by the reaction of 3-methylsulfonyl-2-oxoimidazolidine-1-carbonyl chloride (2.27 g, 0.01 mol) and 2-methoxybenzenethiol (1.4 g, 0.01 mol) in the presence of toluene (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

$C_{12}H_{14}N_2O_5S_2$
M_r = 330.37
Monoclinic, $P2_1/n$
a = 10.5529 (8) Å
b = 11.9861 (9) Å
c = 11.9789 (9) Å
β = 108.662 (1)°
V = 1435.52 (19) Å^3
Z = 4
D_x = 1.529 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2478 reflections
θ = 2.2–25.0°
μ = 0.39 mm^{-1}
T = 298 (2) K
Block, colorless
0.29 × 0.15 × 0.14 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.894, T_{max} = 0.947
7468 measured reflections
2581 independent reflections
2348 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 25.2°
$h = -12 \rightarrow 12$
$k = -14 \rightarrow 14$
$l = -9 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.119
S = 1.18
2581 reflections
192 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0443P)^2 + 0.9471P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11A···O2^{i}	0.97	2.48	3.346 (4)	148
C3—H3···O5^{ii}	0.93	2.55	3.449 (4)	164
C1—H1C···O3^{ii}	0.96	2.40	3.328 (5)	162

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

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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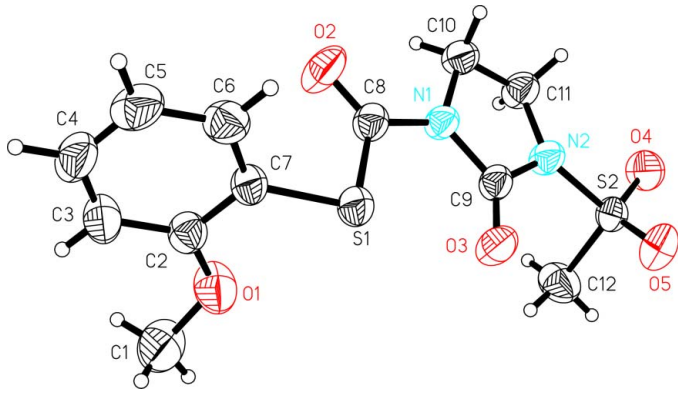

Figure 1
View of (I), with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radius.

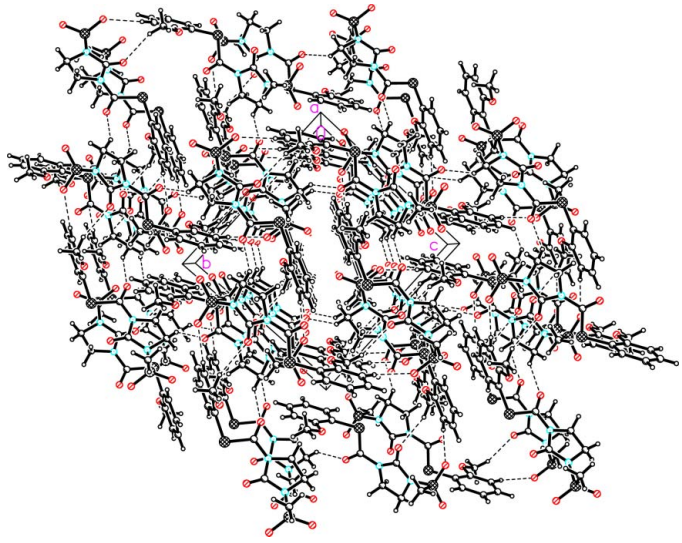

Figure 2
Crystal packing of (I). Intermolecular C—H···O hydrogen bonds are indicated by dashed lines.

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