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N-(2,2-Dimethyl-4-oxo-1,3-thiazolidin-3-yl)-6-methylimidazo[2,1-b][1,3]thiazole-5-carbox-amide monohydrate

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The title compound, $C_{12}H_{14}N_4O_2S_2 \cdot H_2O$, is a member of a new series of imidazo[2,1-b]thiazoles. The thiazolidine ring system adopts an envelope conformation. The packing is stabilized by intra- and intermolecular hydrogen-bond interactions.

Comment

Imidazothiazole derivatives have demonstrated a broad range of biological activities, including immunoregulatory (Devlin & Hargrave, 1989), antihelmintic (Marin *et al.*, 1992), antimicrobial (Ulusoy *et al.*, 1997), cardiotonic (Andreani *et al.*, 1998) and anticancer (Andreani *et al.*, 1992). These biological functions of imidazothiazole derivatives stimulated our research interest and we have synthesized the title imidazothiazole derivative N-(2,2-dimethyl-4-oxo-1,3-thiazolidin-3-yl)-6-methylimidazo[2,1-b][1,3]thiazole-5-carboxamide monohydrate, (3).
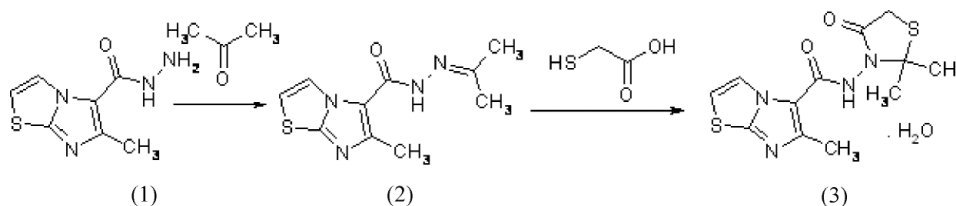
The main geometric parameters of (3) are listed in Table 1 and the molecular structure is illustrated in Fig. 1. The thiazole and imidazole rings are essentially coplanar and the least-squares plane containing all the non-H atoms has a maximum deviation of 0.005 (1) Å for atom N2. There is a dihedral angle of 0.38 (1)° between the thiazole and imidazole rings. The thiazolidine ring system adopts an envelope conformation,
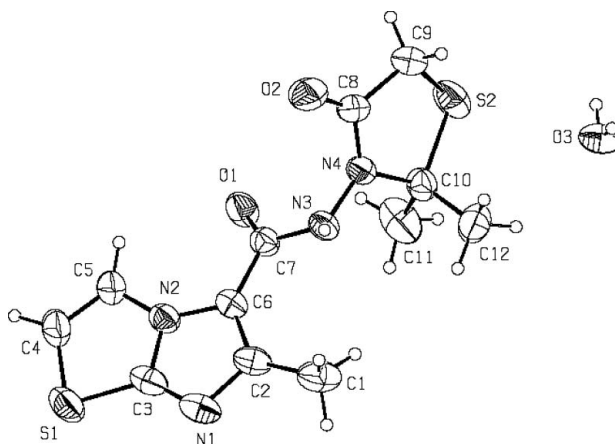

Figure 1
An *ORTEP*-3 drawing (Farrugia, 1997) of (3), with the atom-numbering scheme and 30% probability displacement ellipsoids.
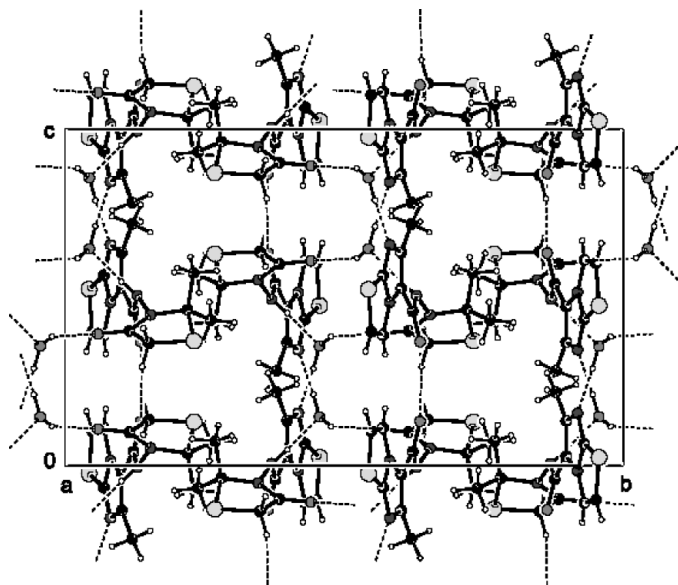
Figure 2
Packing and hydrogen-bond contacts (dashed lines) of (3).

with atom S2 0.173 (1) Å from the plane of the other four atoms. There is an angle of 72.46 (1)° between the imidazo[2,1-*b*][1,3]thiazole and thiazolidine ring systems.

The mean C—S bond length [1.7588 (5) Å] is longer than distances reported for similar molecules [1.729 (2) (Akkurt *et al.*, 2005) and 1.739 (5) Å (Vasu *et al.*, 2004)]. The other bond lengths and angles are in agreement with expected values (Allen *et al.*, 1987).

In the crystal structure, the molecules are linked by intra- and intermolecular hydrogen-bond contacts; relevant data are listed in Table 2 (Fig. 2).

Experimental

A mixture of 6-methylimidazo[2,1-*b*]thiazole-5-carboxylic acid iso-propylidenehydrazide (1.18 g, 0.05 mol) and 2-mercaptoacetic acid (13.82 g, 0.15 mol) was refluxed in dry benzene (30 ml) for 6 h using a Dean–Stark trap. Excess benzene was evaporated *in vacuo*. The residue was triturated with saturated $NaHCO_3$ until CO_2 evaluation ceased and allowed to stand overnight. The solid thus obtained was filtered off, washed with water and crystallized from a C_2H_5OH–H_2O mixture (Ur *et al.*, 2004) (m.p. 403–405 K). IR (KBr, cm^{-1}): 3312, 3125 (NH); 1691, 1663 (C=O). ^{1}H NMR ($CDCl_3$): 1.97 (3H, *s*, CH_3), 2.15 (3H, *s*, CH_3), 2.64 (3H, *s*, 6-CH_3), 6.89 (1H, *d*, *J* = 4.4 Hz, C_2-H), 8.24 (1H, *d*, *J* = 4.4 Hz, C_3-H), 8.34 (1H, *s*, CONH). EIMS (70 eV) *m/z* (%): 310 (M^+, 43), 238 (1), 181 (6), 166 (30), 165 (100), 137 (8), 111 (6), 57 (12). Analysis calculated for $C_{12}H_{14}N_4O_2S_2.H_2O$: C 43.88, H 4.91, N 17.06%; found: C 44.15, H 5.20, N 17.00%.

Crystal data

$C_{12}H_{14}N_4O_2S_2\cdot H_2O$	Mo $K\alpha$ radiation
$M_r = 328.43$	Cell parameters from 19865
Orthorhombic, *Pbca*	reflections
$a = 11.3660$ (7) Å	$\theta = 1.6–27.6°$
$b = 21.4096$ (12) Å	$\mu = 0.35$ mm^{-1}
$c = 12.9034$ (11) Å	$T = 293$ K
$V = 3139.9$ (4) Å^3	Prism, colorless
$Z = 8$	$0.38 \times 0.35 \times 0.29$ mm
$D_x = 1.390$ Mg m^{-3}	

Data collection

Stoe IPDS-II diffractometer	3558 independent reflections
ω scans	2155 reflections with $I > 2\sigma(I)$
Absorption correction: by	$R_{int} = 0.038$
integration (*X-RED32*;	$\theta_{max} = 27.5°$
Stoe & Cie, 2002)	$h = -14 \rightarrow 14$
$T_{min} = 0.877$, $T_{max} = 0.904$	$k = -27 \rightarrow 24$
19955 measured reflections	$l = -16 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1017P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.055$	$+ 0.0559P]$
$wR(F^2) = 0.178$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.08$	$(\Delta/\sigma)_{max} < 0.001$
3558 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
214 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}
H atoms treated by a mixture of	Extinction correction: *SHELXL97*
independent and constrained	Extinction coefficient: 0.0090 (18)
refinement	

Table 1
Selected geometric parameters (Å, °).

S1—C3	1.716 (3)	N2—C5	1.388 (4)
S1—C4	1.725 (5)	N2—C6	1.397 (3)
S2—C9	1.779 (4)	N2—C3	1.351 (4)
S2—C10	1.815 (3)	N3—N4	1.389 (3)
O1—C7	1.218 (3)	N3—C7	1.360 (3)
O2—C8	1.218 (4)	N4—C8	1.337 (3)
N1—C2	1.384 (3)	N4—C10	1.469 (3)
N1—C3	1.319 (4)		
C3—S1—C4	89.08 (18)	N2—C5—C4	110.9 (4)
C9—S2—C10	93.04 (15)	N2—C6—C2	105.0 (2)
C2—N1—C3	105.0 (2)	N2—C6—C7	119.6 (2)
C3—N2—C6	106.8 (2)	N3—C7—C6	114.4 (2)
C5—N2—C6	139.1 (2)	O1—C7—C6	121.8 (2)
C3—N2—C5	114.1 (2)	O1—C7—N3	123.7 (2)
N4—N3—C7	117.9 (2)	N4—C8—C9	111.2 (3)
N3—N4—C10	117.8 (2)	O2—C8—N4	124.1 (2)
C8—N4—C10	119.6 (2)	O2—C8—C9	124.7 (3)
N3—N4—C8	120.4 (2)	S2—C9—C8	107.3 (2)
N1—C2—C6	110.5 (2)	S2—C10—C12	111.3 (2)
N1—C2—C1	119.4 (2)	S2—C10—C11	108.5 (3)
N1—C3—N2	112.8 (2)	N4—C10—C11	110.0 (2)
S1—C3—N2	111.8 (3)	S2—C10—N4	102.78 (17)
S1—C3—N1	135.4 (3)	N4—C10—C12	110.7 (3)
S1—C4—C5	114.1 (3)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N3—H3$\cdots$O3^i	0.83 (4)	2.01 (4)	2.828 (3)	167 (3)
O3—H3$A\cdots$N1^{ii}	0.91 (5)	1.89 (5)	2.795 (4)	174 (4)
O3—H3$B\cdots$O2^{iii}	0.78 (4)	2.16 (4)	2.871 (4)	152 (4)
C5—H5$\cdots$O1	0.95 (4)	2.49 (3)	3.001 (4)	114 (3)
C9—H9$B\cdots$O1^{iv}	0.97	2.29	3.225 (4)	163

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

Methyl and methylene H atoms were positioned geometrically and constrained to an idealized geometry, with C—H distances of 0.96 Å for methyl and 0.97 Å for methylene groups. The U_{iso}(H) values were constrained to be 1.2 (1.5 for methyl group) times U_{eq} of the carrier atoms. The other H atoms were found in a difference Fourier map and refined isotropically.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s)

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used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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