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6-Methyl-*N*-(3-oxo-1-thia-4-azaspiro[4.4]non-4-yl)-imidazo[2,1-*b*][1,3]thiazole-5-carboxamide monohydrate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.076
Data-to-parameter ratio = 15.6

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

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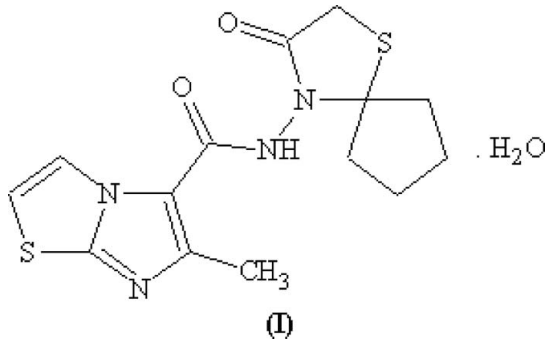
The title compound, $C_{14}H_{16}N_4O_2S_2\cdot H_2O$, is a member of a new series of imidazo[2,1-*b*]thiazoles. The crystal packing is stabilized by intermolecular hydrogen bonds.

Comment

The discovery of the immunomodulatory properties of levamisole [(−)-2,3,5,6-tetrahydro-6-phenylimidazo[2,1-*b*]thiazole; Devlin & Hargrave, 1989] has provoked a great deal of research on imidazo[2,1-*b*]thiazole derivatives. In connection with our previous papers on the synthesis of imidazo[2,1-*b*]-thiazoles and their crystal structures (Akkurt *et al.*, 2005; Öztürk Yıldırım *et al.*, 2005), we report here the crystal structure of the title spiro derivative, 6-methyl-*N*-(3-oxo-1-thia-4-azaspiro[4.4]non-4-yl)imidazo[2,1-*b*][1,3]thiazole-5-carboxamide monohydrate, (I).



(I)

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1. In the

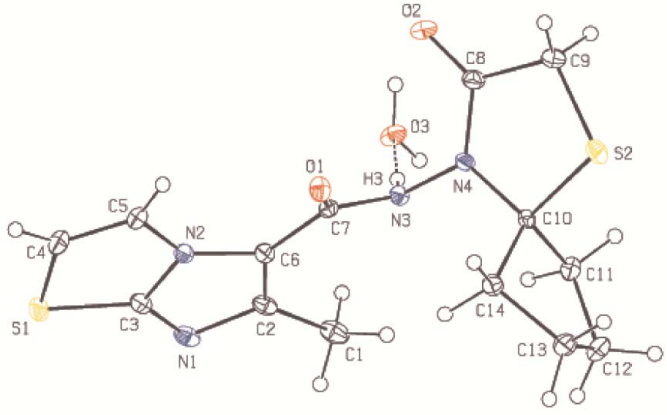


Figure 1
A drawing of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The broken line indicates a hydrogen bond.

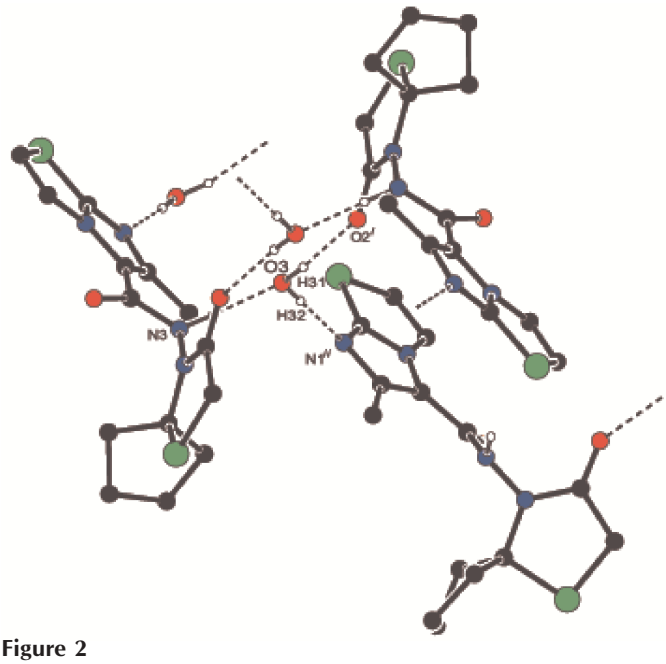


Figure 2
A packing diagram for (I). Broken lines indicate hydrogen bonds. H atoms not involved in the hydrogen bonding have been omitted.

cyclopentane ring, the C—C single-bond length varies between 1.530 (2) and 1.545 (2) Å and has a mean value of 1.538 (2) Å, which is comparable with the corresponding average value for a cyclopentane ring [1.543 (18) Å; Allen *et al.*, 1987].

The cyclopentane ring adopts an envelope conformation with atom C14 at the flap, with puckering parameters of q_2 = 0.430 (2) and φ_2 = −31.4 (2)° (Cremer & Pople, 1975). The thiazole and imidazole rings are essentially coplanar, with a dihedral angle of 0.56 (7)°. The mean C—S bond length [1.783 (2) Å] may be compared with the corresponding values in similar molecules [1.7588 (2) Å (Öztürk Yıldırım *et al.*, 2005) and 1.729 (2) Å (Akkurt *et al.*, 2005)].

Intermolecular hydrogen bonds (Table 2) are highly effective in forming polymeric networks in (I) (Fig. 2) and in stabilizing the crystal packing.

Experimental

A mixture of 6-methyl-*N*-(cyclopentylidene)imidazo[2,1-*b*][1,3]thiazole-5-carbohydrazide (1.31 g, 0.005 mol) (Ur *et al.*, 2004) and $HSCH_2COOH$ (13.82 g, 0.15 mol) was refluxed in dry benzene (30 ml) using a Dean–Stark trap for 6 h. Excess benzene was evaporated *in vacuo*. The residue was triturated with saturated $NaHCO_3$ until CO_2 evolution ceased and then allowed to stand overnight. The solid thus obtained was filtered, washed with H_2O and crystallized from C_2H_5OH–H_2O mixture (1:2) (Ur *et al.*, 2004; m.p. 399–401 K). IR (KBr, ν_{max}, cm^{-1}): 3366, 3125 (NH), 1692, 1665 (C═O); ^{1}H NMR (DMSO-d_6, δ, p.p.m.): 1.57–1.76 (4H, *m*, cyclopent.), 1.83–1.94 (2H, *m*, cyclopent.), 2.05–2.23 (2H, *m*, cyclopent.), 2.55 (3H, *s*, CH_3), 3.68 (2H, *s*, thiazolidinone CH_2), 7.37 (1H, *d*, *J* = 4.4 Hz, C2-H), 7.99 (1H, *d*, *J* = 4.4 Hz, C3-H), 9.83 (1H, *s*, CONH); ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 16.45 (6-CH_3), 22.99 (cyclopent. C3 and C4), 29.38 (thiazolidinone C5), 38.84 (cyclopent. C2 and C5), 77.61 (thiazolidinone C2), 113.17 (imidazothiazole C2), 116.21 (imidazothiazole C5), 121.22 (imidazothiazole C3), 148.31 (imidazothiazole C6), 152.61 (imidazothiazole C7a), 160.82, 169.45 (CONH/C═O). EI–MS (70 eV), *m*/*z* (%): 336 (M^+, 31), 262 (2), 238 (15), 197 (3), 181 (9), 179 (1), 165 (100), 137 (7), 111 (4), 97 (5). Analysis calculated for $C_{14}H_{16}N_4O_2S_2{\cdot}H_2O$: C 47.43, H 5.11, N 15.80%; found: C 47.80, H 5.28, N 15.46%.

Crystal data

$C_{14}H_{16}N_4O_2S_2{\cdot}H_2O$
M_r = 354.46
Orthorhombic, *Pbca*
a = 12.959 (5) Å
b = 10.908 (5) Å
c = 23.797 (5) Å
V = 3364 (2) Å^3
Z = 8
D_x = 1.400 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 338 reflections
θ = 6.0–20.0°
μ = 0.34 mm^{-1}
T = 100 K
Prism, colourless
0.35 × 0.24 × 0.14 mm

Data collection

Bruker Nonius KappaCCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.891, T_{max} = 0.955
32544 measured reflections
4118 independent reflections
3269 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 28.5°
h = −17 → 17
k = −14 → 14
l = −29 → 31

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.076
S = 1.04
4118 reflections
264 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0323P)^2 + 1.4591P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C3	1.7332 (16)	N1—C3	1.321 (2)
S1—C4	1.745 (2)	N2—C5	1.396 (2)
S2—C9	1.8082 (19)	N2—C3	1.3616 (19)
S2—C10	1.8458 (16)	N2—C6	1.3992 (18)
N1—C2	1.3851 (19)	N3—N4	1.3916 (17)
C3—S1—C4	89.39 (7)	C9—S2—C10	93.85 (7)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···O3	0.91 (2)	1.85 (2)	2.759 (2)	174 (2)
O3—H31···O2^{i}	0.86 (2)	1.98 (2)	2.8197 (19)	167 (2)
O3—H32···N1^{ii}	0.79 (2)	1.98 (2)	2.767 (2)	176 (2)

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

All H atoms were located in a difference synthesis and refined isotropically [C—H = 0.912 (18)–1.026 (17) Å and O—H = 0.79 (2)–0.86 (2) Å]. The U_{iso}(H) values were constrained to between 0.96 and 1.36 times U_{eq} of the carrier atom, while the displacement parameters of the water H atoms were refined freely.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *EVALCCD* (Duisenberg *et al.*, 2003); data reduction: *SADABS* (Sheldrick, 2002); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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