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6-Methyl-*N*′-(1-methylethylidene)imidazo-[2,1-*b*][1,3]thiazole-5-carbohydrazide monohydrate

Mehmet Akkurt,[a]* Sema Öztürk Yıldırım,[a] Fusun Ur,[b] Zafer Cesur,[b] Nesrin Cesur[b] and Orhan Büyükgüngör[c]

[a]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, [b]Department of Pharmaceutical Chemistry, Faculty of Pharmaceutical Sciences, Istanbul University, 34116 Istanbul, Turkey, and [c]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Samsun, Turkey

Correspondence e-mail: akkurt@erciyes.edu.tr

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.107
Data-to-parameter ratio = 15.7

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

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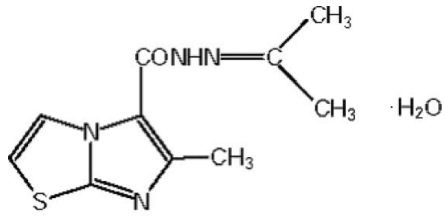
The title compound, $C_{10}H_{12}N_4OS\cdot H_2O$, was obtained from 6-methylimidazo[2,1-*b*]thiazole-5-carbohydrazide and 2-propanone. The crystal structure is stabilized by intra- and intermolecular hydrogen-bond interactions.

Comment

Functionalized imidazo[2,1-*b*]thiazoles have emerged as potentially interesting compounds particularly with regard to their immunoregulatory activity (levamisole) (Devlin & Hargrave, 1989) and also for their anthelmintic (Marin *et al.*, 1992), antimicrobial (Ulusoy *et al.*, 1997) and cardiotonic (Andreani *et al.*, 1998) properties, as well as their anticancer activity (Andreani *et al.*, 1992). In connection with our recent paper on 4-thiazolidinones incorporating an imidazo[2,1-*b*]-thiazole moiety as antimycobacterials (Ur *et al.*, 2004), we now report the crystal structure of 6-methyl-*N*′-(1-methylethylidene)imidazo[2,1-*b*][1,3]thiazole-5-carbohydrazide monohydrate, (I), which is a synthetic precursor of the 4-thiazolidinone ring system.



(I)

The structure of (I) is shown in Fig. 1. The organic molecule (non-H atoms) is essentially planar [the maximum deviations are 0.660 (1) and −0.260 (1) Å for atoms O1 and S1, respectively], with a dihedral angle of 1.38 (9)° between the thiazole and imidazole rings. The terminal carbohydrazide group is extended away from the heterocyclic ring system, with a C7—N3—N4—C8 torsion angle of −165.58 (16)°.

The mean C—S bond length [1.729 (2) Å] is shorter than distances reported for similar molecules [1.739 (5) Å (Vasu *et al.*, 2004) and 1.736 (2) Å (Liu *et al.*, 2003)]. The other bond lengths and angles are comparable with literature values (Allen *et al.*, 1987).

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

Experimental

6-Methylimidazo[2,1-*b*]thiazole-5-carbohydrazide (0.005 mol; Cesur *et al.*, 1994) was heated in 2-propanone (15 ml) for 5 h. The crude

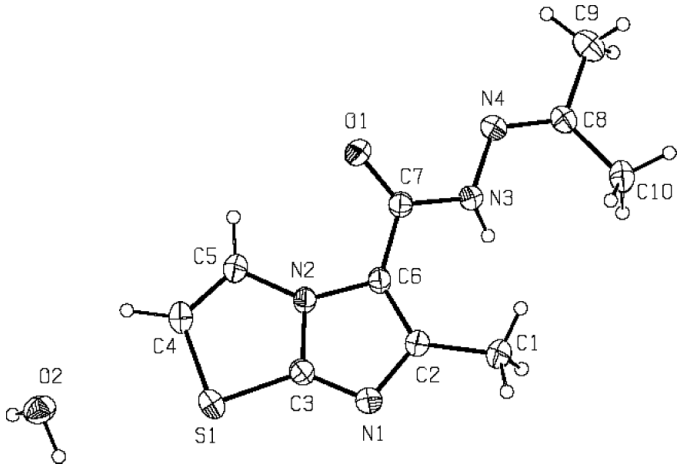


Figure 1
An *ORTEP-3* plot (Farrugia, 1997) of (I), showing the atom-numbering scheme and 30% probability displacement ellipsoids

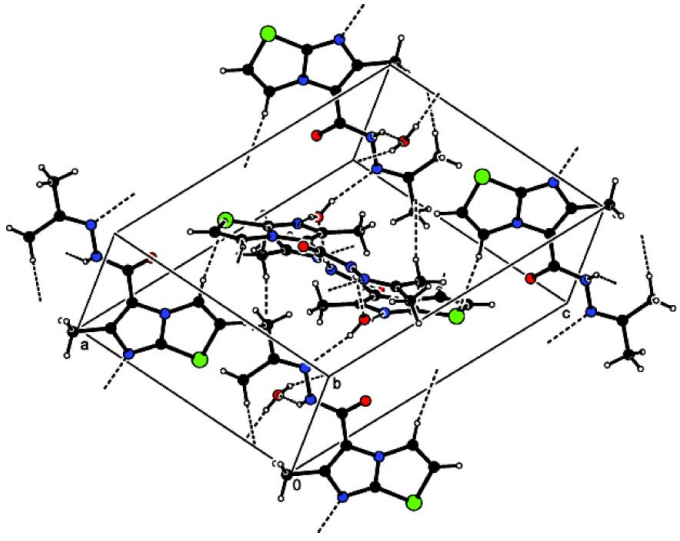


Figure 2
A view of the packing and hydrogen-bond contacts (dashed lines) of (I).

product which precipitated on cooling was filtered off, washed with diethyl ether and crystallized from a C_2H_5OH–H_2O mixture (Ur *et al.*, 2004). IR (KBr, cm^{-1}): 3273, 3143 (NH), 1659 (C═O). ^1H NMR ($CDCl_3$): δ 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* (%): 236 (M^+, 74), 181 (2), 165 (100), 137 (22), 71 (3), 57 (12). Analysis calculated for $C_{10}H_{12}N_4OS.H_2O$: C 47.22, H 5.54, N 22.03%; found: C 47.57, H 5.97, N 21.22%.

Crystal data

$C_{10}H_{12}N_4OS·H_2O$
M_r = 254.32
Monoclinic, $P2_1/n$
a = 10.0955 (7) Å
b = 9.8500 (6) Å
c = 13.3756 (8) Å
β = 111.226 (5)°
V = 1239.85 (14) Å^3
Z = 4
D_x = 1.362 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15184 reflections
θ = 2.2–27.3°
μ = 0.26 mm^{-1}
T = 293 K
Plate, pale yellow
0.38 × 0.24 × 0.08 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.908, T_{max} = 0.980
15514 measured reflections
2771 independent reflections
2217 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 27.3°
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.107
S = 1.05
2771 reflections
177 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2 + 0.2624P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C3	1.7262 (19)	N2—C3	1.353 (2)
S1—C4	1.732 (2)	N2—C6	1.392 (2)
O1—C7	1.222 (2)	N3—C7	1.351 (3)
N1—C2	1.378 (2)	N3—N4	1.396 (2)
N1—C3	1.321 (2)	N4—C8	1.276 (3)
N2—C5	1.395 (2)		
C3—S1—C4	89.49 (9)	S1—C3—N1	135.99 (14)
C2—N1—C3	104.57 (14)	S1—C4—C5	113.69 (16)
C3—N2—C6	106.96 (14)	N2—C5—C4	111.18 (17)
C5—N2—C6	138.64 (15)	N2—C6—C7	120.07 (14)
C3—N2—C5	114.32 (15)	N2—C6—C2	104.66 (15)
N4—N3—C7	117.67 (14)	N3—C7—C6	114.54 (15)
N3—N4—C8	115.75 (15)	O1—C7—C6	121.35 (17)
N1—C2—C1	119.71 (15)	O1—C7—N3	124.09 (16)
N1—C2—C6	111.08 (16)	N4—C8—C9	116.47 (16)
S1—C3—N2	111.31 (13)	N4—C8—C10	125.96 (17)
N1—C3—N2	112.70 (16)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N1^{i}	0.91 (3)	1.98 (3)	2.883 (2)	172 (3)
O2—H2B···N4^{ii}	0.83 (4)	2.21 (4)	3.018 (2)	166 (3)
N3—H3···O2^{iii}	0.83 (2)	2.12 (2)	2.890 (2)	155 (2)
C5—H5···O1	0.97 (2)	2.53 (2)	3.034 (2)	112.2 (15)
C5—H5···S1^{iv}	0.97 (2)	2.82 (2)	3.782 (2)	172.0 (17)
C10—H10C···O1^{v}	0.96	2.54	3.400 (3)	149

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

Methyl H atoms were positioned geometrically and constrained to an idealized geometry, with C—H distances of 0.96 Å. The U_{iso}(H) values were constrained to be 1.5 times U_{eq} of the carrier atom. The other H atoms were found in difference Fourier maps and refined isotropically.

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