Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-2-[(1*H*-Imidazol-4-yl)methylidene]-hydrazinecarbothioamide monohydrate

Benayad Houari,[a] Samira Louhibi,[a]* Leila Boukli-Hacene,[a] Thierry Roisnel[b] and Mustapha Taleb[c]

[a]Laboratoire de Chimie Inorganique et Environnement, Université de Tlemcen, BP 119, 13000, Tlemcen, Algeria, [b]Centre de Diffractométrie X, UMR 6226 CNRS, Unité Sciences Chimiques de Rennes, Université de Rennes I, 263 Avenue du Général Leclerc, 35042 Rennes, France, and [c]USMBA, FSDM, Département de Chimie, BP 1796 Fès - Atlas, Morocco
Correspondence e-mail: samhibi1@yahoo.fr

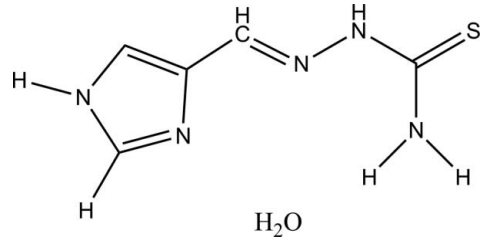
Received 6 August 2013; accepted 14 August 2013

Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.030; wR factor = 0.083; data-to-parameter ratio = 15.0.

In the title compound, $C_5H_7N_5S\cdot H_2O$, the main molecule is approximately planar, with a maximum deviation from the mean plane through the non-H atoms of 0.1478 (12) Å for the amine N atom. In the crystal, the components are connected *via* N—H···O, N—H···S and O—H···N hydrogen bonds, forming a three-dimensional network.

Related literature

For the biological activity of thiosimecarbazone derivatives, see: Finch *et al.* (2000). For the crystal structures of related compounds, see: Alomar *et al.* (2013).



Experimental

Crystal data

$C_5H_7N_5S\cdot H_2O$
M_r = 187.23
Monoclinic, $P2_1/c$
a = 10.8734 (5) Å
b = 11.2416 (5) Å
c = 7.0822 (3) Å
β = 75.601 (2)°
V = 838.50 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 150 K
0.4 × 0.23 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
6537 measured reflections
1903 independent reflections
1726 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.083
S = 1.06
1903 reflections
127 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O21	0.92 (2)	2.34 (2)	3.2325 (16)	163.2 (18)
N8—H8···S1^{i}	0.90 (2)	2.51 (2)	3.3334 (13)	153.0 (18)
O21—H21B···N10	0.87 (2)	2.17 (2)	3.0399 (15)	172 (2)

Symmetry code: (i) $x, y-1, z$.

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2006); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 2012) and *CRYSCAL* (T. Roisnel, local program).

The authors gratefully acknowledge the support of the Algerian Ministry of Higher Education and Scientific Research.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5642).

References

Alomar, K., Landreau, A., Allain, M., Bouet, G. & Larcher, G. (2013). *J. of Inorg. Biochem.* **126**, 76–83.
Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
Bruker (2006). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Finch, R. A., Liu, M., Grill, S. P., Rose, W. C., Loomis, R., Vasquez, K. M., Cheng, Y. & Sartorelli, A. C. (2000). *Biochem. Pharmacol.* **59**, 983–991.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2013). E**69**, o1469 [doi:10.1107/S1600536813022927]

(*E*)-2-[(1*H*-Imidazol-4-yl)methylidene]hydrazinecarbothioamide monohydrate

Benayad Houari, Samira Louhibi, Leila Boukli-Hacene, Thierry Roisnel and Mustapha Taleb

S1. Comment

Our interest in thiosemicarbazone derivatives stems from their wide spectrum of biological activity (Finch *et al.*, 2000; Alomar *et al.* 2013). As part of our study of thiosemicarbazone derivatives, we report herein the crystal structure of the title compound (I). The molecular structure of (I) is shown in Fig. 1. The molecule is approximately planar and the maximum deviation from the least squares plane through the 11 non-hydrogen atoms is -0.1478 (12) Å for N1. The bond angles suggest sp^2 hybridization for the C and N atoms which contributes to the planarity of the molecule. The crystal packing is stabilized by intermolecular N—H···O and N—H···S hydrogen bonds (Fig. 2 and Table 1) forming a three-dimensional network.

S2. Experimental

All the chemicals were purchased from Merck and were used as received. An equimolar amount of thiosemicarbazide 10 mmol (0.91 g) and imidazolecarboxaldehyde 10 mmol (0.96 g) were dissolved in a mixture of ethanol and water (30 ml, 50%) and refluxed for 5 h in the presence of a catalytic amount of glacial acetic acid. Yellow crystals suitable for X-ray analysis were obtained after slow evaporation of the solution.

S3. Refinement

H atoms bonded to C atoms were placed in calculated positions with C—H = 0.95 Å and refined in a riding-model approximation with U_{iso}(H) = 1.2U_{eq}(C). The H atoms bonded to O and N atoms were refined independently with fixed isotropic displacement parameters.

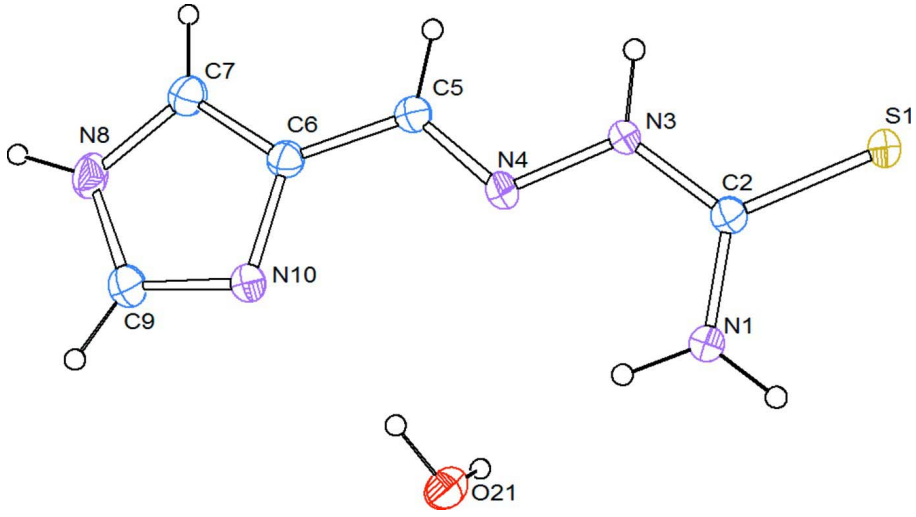


Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

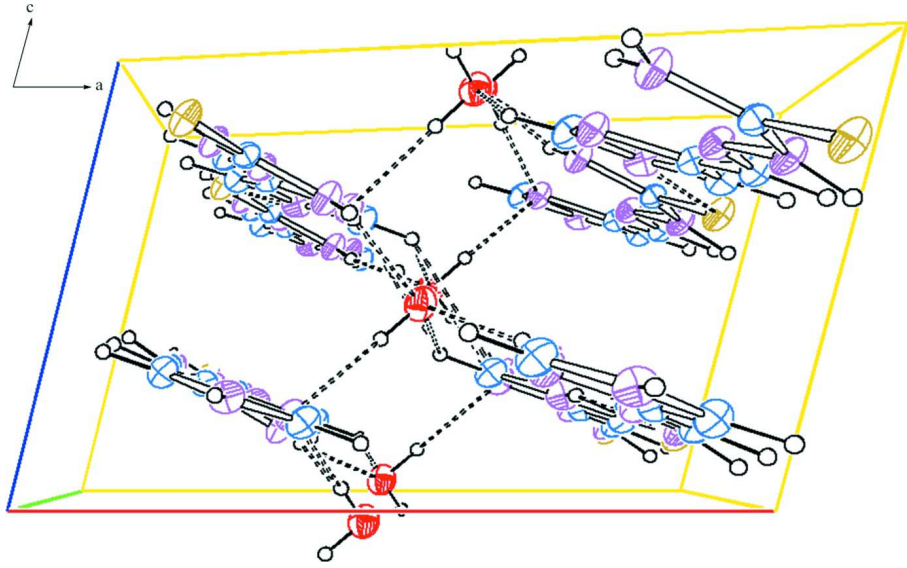


Figure 2

Part of the crystal structure with hydrogen bonds shown as dashed lines.

(I)

Crystal data

$C_5H_7N_5S·H_2O$
M_r = 187.23
Monoclinic, $P2_1/c$
a = 10.8734 (5) Å
b = 11.2416 (5) Å
c = 7.0822 (3) Å
β = 75.601 (2)°
V = 838.50 (6) Å^3
Z = 4
F(000) = 392

D_x = 1.483 Mg m^{-3}
Melting point: 0 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3274 reflections
θ = 2.7–27.5°
μ = 0.35 mm^{-1}
T = 150 K
Prism, colourless
0.4 × 0.23 × 0.16 mm

Data collection

Bruker APEXII CCD diffractometer
Graphite monochromator
CCD rotation images, thin slices scans
6537 measured reflections
1903 independent reflections
1726 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.5°$
$h = -13 \rightarrow 14$
$k = -14 \rightarrow 13$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.083$
$S = 1.06$
1903 reflections
127 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0438P)^2 + 0.2645P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	−0.87437 (3)	0.64372 (3)	0.33879 (5)	0.02064 (13)
N1	−0.66924 (10)	0.51441 (11)	0.17680 (17)	0.0192 (3)
H1A	−0.6294 (19)	0.443 (2)	0.140 (3)	0.05*
H1B	−0.6319 (19)	0.583 (2)	0.135 (3)	0.05*
C2	−0.79042 (12)	0.51613 (11)	0.27634 (18)	0.0154 (3)
N3	−0.84841 (10)	0.41073 (10)	0.32669 (17)	0.0177 (2)
H3	−0.928 (2)	0.408 (2)	0.406 (3)	0.05*
N4	−0.77834 (10)	0.30715 (10)	0.28202 (16)	0.0170 (2)
C5	−0.84105 (12)	0.21003 (12)	0.32281 (19)	0.0168 (3)
H5	−0.9299	0.2125	0.38	0.02*
C6	−0.77714 (12)	0.09669 (12)	0.28214 (18)	0.0165 (3)
C7	−0.82981 (13)	−0.01458 (12)	0.3055 (2)	0.0203 (3)
H7	−0.9172	−0.0336	0.3521	0.024*
N8	−0.73164 (11)	−0.09260 (11)	0.24822 (17)	0.0214 (3)
H8	−0.743 (2)	−0.172 (2)	0.251 (3)	0.05*
C9	−0.62411 (13)	−0.02867 (13)	0.1925 (2)	0.0218 (3)
H9	−0.5421	−0.0627	0.1467	0.026*
N10	−0.64591 (10)	0.08743 (10)	0.20915 (17)	0.0191 (3)

O21	−0.50826 (10)	0.29013 (9)	−0.04044 (17)	0.0250 (2)
H21A	−0.552 (2)	0.3077 (19)	−0.118 (3)	0.05*
H21B	−0.552 (2)	0.237 (2)	0.038 (3)	0.05*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.01797 (19)	0.01144 (19)	0.0285 (2)	0.00167 (11)	0.00184 (13)	0.00089 (13)
N1	0.0159 (5)	0.0133 (6)	0.0254 (6)	−0.0002 (4)	0.0004 (4)	0.0000 (5)
C2	0.0159 (6)	0.0146 (6)	0.0152 (6)	0.0002 (5)	−0.0030 (4)	−0.0003 (5)
N3	0.0154 (5)	0.0117 (6)	0.0224 (6)	0.0018 (4)	0.0025 (4)	−0.0001 (4)
N4	0.0174 (5)	0.0131 (6)	0.0185 (6)	0.0028 (4)	−0.0009 (4)	−0.0010 (4)
C5	0.0158 (6)	0.0158 (7)	0.0178 (6)	−0.0005 (5)	−0.0022 (5)	−0.0003 (5)
C6	0.0171 (6)	0.0162 (7)	0.0158 (6)	−0.0002 (5)	−0.0030 (5)	−0.0002 (5)
C7	0.0207 (6)	0.0163 (7)	0.0230 (7)	−0.0019 (5)	−0.0034 (5)	−0.0006 (5)
N8	0.0279 (6)	0.0114 (6)	0.0240 (6)	−0.0005 (5)	−0.0046 (5)	−0.0013 (5)
C9	0.0224 (7)	0.0167 (7)	0.0240 (7)	0.0032 (5)	−0.0015 (5)	−0.0012 (5)
N10	0.0177 (5)	0.0139 (6)	0.0233 (6)	0.0013 (4)	−0.0006 (4)	−0.0001 (4)
O21	0.0221 (5)	0.0221 (6)	0.0298 (6)	−0.0038 (4)	−0.0047 (4)	0.0068 (4)

Geometric parameters (Å, º)

S1—C2	1.6986 (13)	C6—C7	1.3685 (18)
N1—C2	1.3309 (17)	C6—N10	1.3955 (16)
N1—H1A	0.92 (2)	C7—N8	1.3630 (18)
N1—H1B	0.88 (2)	C7—H7	0.95
C2—N3	1.3480 (17)	N8—C9	1.3453 (18)
N3—N4	1.3844 (15)	N8—H8	0.90 (2)
N3—H3	0.90 (2)	C9—N10	1.3264 (18)
N4—C5	1.2816 (17)	C9—H9	0.95
C5—C6	1.4456 (18)	O21—H21A	0.83 (2)
C5—H5	0.95	O21—H21B	0.87 (2)
C2—N1—H1A	119.8 (13)	C7—C6—C5	127.99 (12)
C2—N1—H1B	118.5 (14)	N10—C6—C5	122.42 (12)
H1A—N1—H1B	121.2 (19)	N8—C7—C6	106.20 (12)
N1—C2—N3	117.63 (12)	N8—C7—H7	126.9
N1—C2—S1	123.18 (10)	C6—C7—H7	126.9
N3—C2—S1	119.18 (10)	C9—N8—C7	107.62 (12)
C2—N3—N4	118.96 (11)	C9—N8—H8	129.8 (14)
C2—N3—H3	120.5 (14)	C7—N8—H8	122.6 (14)
N4—N3—H3	119.7 (14)	N10—C9—N8	112.12 (12)
C5—N4—N3	115.68 (11)	N10—C9—H9	123.9
N4—C5—C6	120.23 (12)	N8—C9—H9	123.9
N4—C5—H5	119.9	C9—N10—C6	104.47 (11)
C6—C5—H5	119.9	H21A—O21—H21B	106 (2)
C7—C6—N10	109.59 (11)		

N1—C2—N3—N4	3.30 (18)	C5—C6—C7—N8	−179.86 (12)
S1—C2—N3—N4	−177.56 (9)	C6—C7—N8—C9	−0.07 (15)
C2—N3—N4—C5	−175.73 (11)	C7—N8—C9—N10	−0.24 (16)
N3—N4—C5—C6	180.00 (11)	N8—C9—N10—C6	0.43 (15)
N4—C5—C6—C7	−175.90 (13)	C7—C6—N10—C9	−0.46 (15)
N4—C5—C6—N10	3.9 (2)	C5—C6—N10—C9	179.72 (12)
N10—C6—C7—N8	0.33 (15)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O21	0.92 (2)	2.34 (2)	3.2325 (16)	163.2 (18)
N8—H8···S1^{i}	0.90 (2)	2.51 (2)	3.3334 (13)	153.0 (18)
O21—H21*B*···N10	0.87 (2)	2.17 (2)	3.0399 (15)	172 (2)

Symmetry code: (i) *x*, *y*−1, *z*.