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2-(2-Amino-5-methyl-1,2,4-triazolo[1,5-*a*]-pyrimidin-7-yl)acetohydrazide monohydrate

Sanae Lahmidi,[a]* Nada Kheira Sebbar,[a] Mohammed Boulhaoua,[a] El Mokhtar Essassi,[a] Joel T. Mague[b] and Hafid Zouihri[c]

[a]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Mohammed V University in Rabat, BP 1014, Avenue Ibn Batouta, Rabat, Morocco, [b]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA, and [c]Département de chimie, Faculté des Sciences, Université Ibn Zohr, BP 8106, Cité Dakhla, 80000 Agadir, Morocco. *Correspondence e-mail: lahmidi_sanae@yahoo.fr

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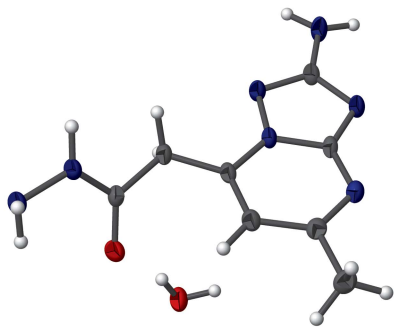
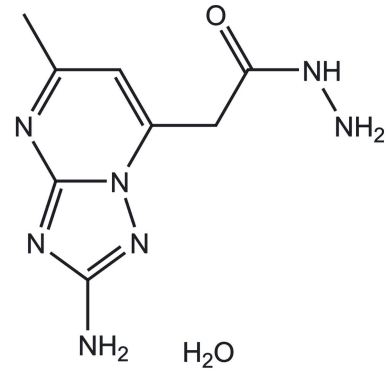
In the crystal structure of the title molecule, $C_8H_{11}N_7O\cdot H_2O$, a network of O—H···O, O—H···N, N—H···O and N—H···N hydrogen bonds links the components, forming layers which include the lattice water molecules. The layers are held together by π–π stacking interactions.

3D view



Chemical scheme



Structure description

Pyrimidine is one of the most important heterocycles that are widely used as a key building unit for the preparation of many pharmaceutical compounds. Fusion of pyrimidine with 1,2,4-triazole gives 1,2,4-triazolo[1,5-*a*]pyrimidine (Salas *et al.*, 1999). These molecules are thermodynamically stable and, thus, the most studied (Fischer *et al.*, 2008). Recently, due to their diverse pharmacological activities, such as antitumor potency (Zhang *et al.*, 2007) and antimicrobial activity (Luo *et al.*, 2013), it is understandable that research on the synthesis of these compounds has intensified.

As part of our ongoing research program on heterocyclic compounds which may serve as leads for designing novel chemotherapeutic agents, we were particularly interested to examined the action of hydrazine hydrate on ethyl 2-(2-amino-5-methyl-1,2,4-triazolo[1,5-*a*] pyrimidin-7-yl) acetate leading to the corresponding 2-(2-amino-5-methyl[1,2,4]triazolo[1,5-*a*]pyrimidin-7-yl)acetohydrazide (Fig. 1).

In the crystal, a network of O—H···O, O—H···N, N—H···O and N—H···N hydrogen bonds (Table 1) links the components into layers which include the lattice water molecules (Fig. 2). The layers are held together by π–π stacking interactions as shown in Fig. 3. The dihedral angle between the mean planes of the two molecules is 1.48 (8)° and the slippage is 0.89 Å.

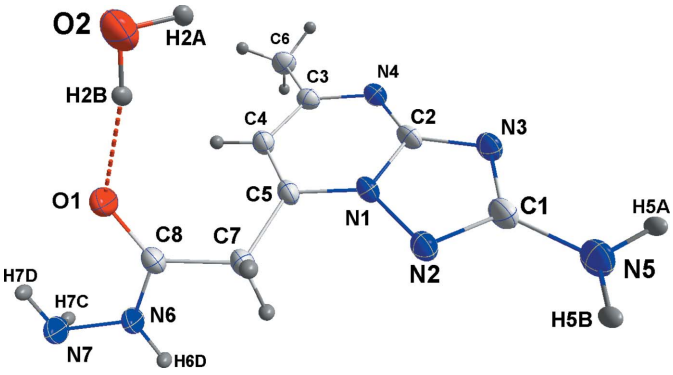


Figure 1
The title molecule with labeling scheme and 50% probability ellipsoids. The O—H···O hydrogen bond is shown as a dotted line.

Synthesis and crystallization

Ethyl 2-(2-amino-5-methyl-1,2,4-triazolo[1,5-*a*]pyrimidin-7-yl) acetate 1 g (0.004 mol) was refluxed with hydrazine hydrate 0.44 ml (0.008 mol) in absolute ethanol for 4–5 h. On cooling the mixture, white crystals of 2-(2-amino-5-methyl[1,2,4]-triazolo[1,5-*a*]pyrimidin-7-yl)acetohydrazide monohydrate separated out in 80% yield.

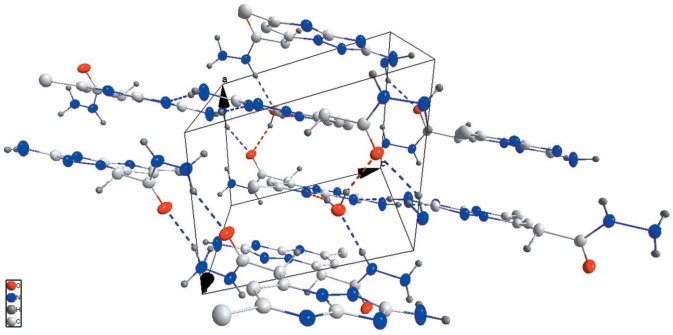


Figure 2
Packing projected onto (382) showing a portion of the layer structure. O—H···O and O—H···N hydrogen bonds are shown as red dotted lines while N—H···O and N—H···N hydrogen bonds are shown as blue dotted lines.

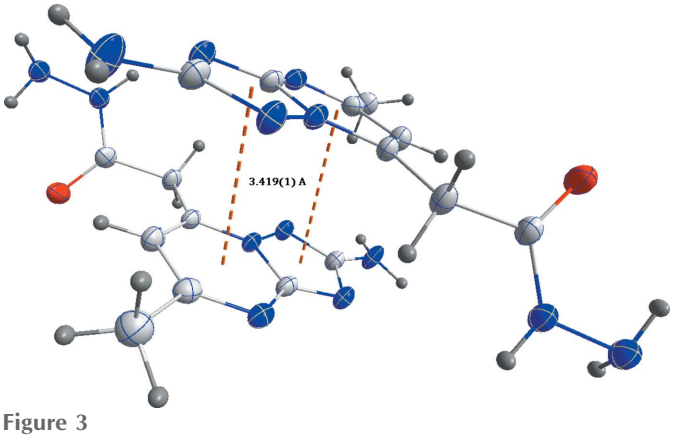


Figure 3
A detail of the π–π stacking interactions between molecules at x, y, z and $2-x$, $1-y$, $1-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2*B*···O1	0.87	2.00	2.8478 (15)	166
O2—H2*A*···N4^{i}	0.87	2.06	2.9004 (16)	162
N5—H5*B*···N7^{ii}	0.91	2.17	3.0636 (17)	167
N5—H5*A*···N3^{iii}	0.91	2.06	2.9713 (17)	177
N6—H6*D*···O2^{iv}	0.91	2.02	2.9193 (16)	169
N7—H7*D*···O1^{v}	0.91	2.15	2.8715 (16)	135
C7—H7*A*···O2^{vi}	0.99	2.43	3.4215 (18)	175

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $-x+2, -y+2, -z+1$; (iii) $-x+2, -y+1, -z$; (iv) $x+1, y, z$; (v) $-x+1, -y+2, -z+2$; (vi) $-x+1, -y+2, -z+1$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_8H_{11}N_7O\cdot H_2O$
M_r	239.25
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	7.2648 (3), 8.7228 (4), 8.9297 (4)
α, β, γ (°)	82.834 (2), 71.465 (2), 85.478 (1)
V (Å^3)	531.87 (4)
Z	2
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.96
Crystal size (mm)	0.21 × 0.16 × 0.08
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.83, 0.92
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8613, 2135, 1966
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.039, 0.104, 1.06
No. of reflections	2135
No. of parameters	155
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.22

Computer programs: *APEX3* (Bruker, 2016), *SAINT* (Bruker, 2016), *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009), *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x160870 [doi:10.1107/S2414314616008701]

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Crystal data

$C_8H_{11}N_7O·H_2O$
M_r = 239.25
Triclinic, $P\overline{1}$
a = 7.2648 (3) Å
b = 8.7228 (4) Å
c = 8.9297 (4) Å
α = 82.834 (2)°
β = 71.465 (2)°
γ = 85.478 (1)°
V = 531.87 (4) Å^3
Z = 2
F(000) = 252
D_x = 1.494 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 7288 reflections
θ = 5.1–74.4°
μ = 0.96 mm^{-1}
T = 150 K
Plate, colourless
0.21 × 0.16 × 0.08 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro–focus source
Mirror monochromator
Detector resolution: 10.4167 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.83, T_{max} = 0.92
8613 measured reflections
2135 independent reflections
1966 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 74.4°, θ_{min} = 5.1°
h = −9→9
k = −10→10
l = −11→11

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.104
S = 1.06
2135 reflections
155 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 0.2766P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å) while those attached to nitrogen and to oxygen were placed in locations derived from a difference map and their parameters adjusted to give N—H = 0.91 and O—H = 0.87 Å. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.56050 (15)	0.88518 (13)	0.82814 (13)	0.0295 (3)
N1	0.85056 (17)	0.64386 (13)	0.42124 (13)	0.0188 (3)
N2	0.96315 (17)	0.73473 (13)	0.29304 (13)	0.0212 (3)
N3	0.91216 (18)	0.50117 (13)	0.22122 (14)	0.0221 (3)
N4	0.71916 (17)	0.39440 (13)	0.48112 (14)	0.0211 (3)
N5	1.1119 (2)	0.68303 (15)	0.02980 (15)	0.0302 (3)
H5B	1.1518	0.7819	0.0107	0.036*
H5A	1.1098	0.6274	−0.0493	0.036*
N6	0.86297 (17)	0.91552 (14)	0.83455 (14)	0.0231 (3)
H6D	0.9931	0.9192	0.7843	0.028*
N7	0.79660 (18)	0.97790 (14)	0.98230 (14)	0.0237 (3)
H7D	0.6648	0.9874	1.0086	0.028*
H7C	0.8283	0.9061	1.0542	0.028*
C1	0.9968 (2)	0.64125 (16)	0.17728 (16)	0.0218 (3)
C2	0.8213 (2)	0.50413 (15)	0.37634 (16)	0.0195 (3)
C3	0.6465 (2)	0.42573 (16)	0.63186 (17)	0.0216 (3)
C4	0.6719 (2)	0.56896 (16)	0.68086 (17)	0.0221 (3)
H4	0.6155	0.5878	0.7888	0.027*
C5	0.77773 (19)	0.68022 (15)	0.57282 (16)	0.0192 (3)
C6	0.5390 (2)	0.30117 (17)	0.75096 (18)	0.0276 (3)
H6A	0.4841	0.2334	0.6979	0.041*
H6B	0.4339	0.3480	0.8334	0.041*
H6C	0.6286	0.2405	0.7996	0.041*
C7	0.8302 (2)	0.83592 (16)	0.59765 (16)	0.0222 (3)
H7A	0.7896	0.9152	0.5236	0.027*
H7B	0.9732	0.8376	0.5703	0.027*
C8	0.7395 (2)	0.87902 (15)	0.76432 (17)	0.0215 (3)
O2	0.27856 (16)	0.88291 (12)	0.66816 (13)	0.0296 (3)
H2B	0.3787	0.8856	0.7012	0.044*
H2A	0.3067	0.7999	0.6191	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0224 (5)	0.0370 (6)	0.0313 (6)	−0.0006 (4)	−0.0067 (4)	−0.0172 (5)
N1	0.0234 (6)	0.0169 (5)	0.0185 (6)	−0.0004 (4)	−0.0083 (4)	−0.0057 (4)
N2	0.0278 (6)	0.0185 (6)	0.0179 (6)	−0.0035 (4)	−0.0066 (5)	−0.0035 (4)
N3	0.0296 (6)	0.0192 (6)	0.0198 (6)	−0.0024 (5)	−0.0089 (5)	−0.0059 (4)
N4	0.0246 (6)	0.0190 (6)	0.0223 (6)	−0.0010 (4)	−0.0103 (5)	−0.0040 (4)
N5	0.0475 (8)	0.0251 (6)	0.0188 (6)	−0.0116 (6)	−0.0078 (6)	−0.0056 (5)
N6	0.0222 (6)	0.0259 (6)	0.0225 (6)	−0.0022 (5)	−0.0053 (5)	−0.0108 (5)
N7	0.0250 (6)	0.0260 (6)	0.0214 (6)	−0.0017 (5)	−0.0062 (5)	−0.0097 (5)
C1	0.0274 (7)	0.0201 (6)	0.0204 (7)	−0.0010 (5)	−0.0098 (6)	−0.0054 (5)
C2	0.0237 (7)	0.0165 (6)	0.0223 (7)	0.0008 (5)	−0.0114 (5)	−0.0066 (5)
C3	0.0214 (7)	0.0213 (7)	0.0242 (7)	−0.0003 (5)	−0.0098 (6)	−0.0027 (5)
C4	0.0240 (7)	0.0220 (7)	0.0214 (7)	−0.0001 (5)	−0.0073 (6)	−0.0059 (5)
C5	0.0208 (6)	0.0192 (6)	0.0198 (6)	0.0017 (5)	−0.0081 (5)	−0.0075 (5)
C6	0.0304 (8)	0.0240 (7)	0.0279 (8)	−0.0058 (6)	−0.0084 (6)	0.0004 (6)
C7	0.0259 (7)	0.0200 (7)	0.0213 (7)	−0.0030 (5)	−0.0060 (6)	−0.0076 (5)
C8	0.0237 (7)	0.0170 (6)	0.0249 (7)	−0.0011 (5)	−0.0072 (6)	−0.0070 (5)
O2	0.0307 (6)	0.0266 (5)	0.0366 (6)	0.0024 (4)	−0.0147 (5)	−0.0132 (4)

Geometric parameters (Å, º)

O1—C8	1.2425 (18)	N7—H7D	0.9099
N1—C5	1.3558 (17)	N7—H7C	0.9100
N1—N2	1.3742 (16)	C3—C4	1.4180 (19)
N1—C2	1.3814 (16)	C3—C6	1.496 (2)
N2—C1	1.3458 (17)	C4—C5	1.370 (2)
N3—C2	1.3342 (18)	C4—H4	0.9500
N3—C1	1.3677 (18)	C5—C7	1.4997 (18)
N4—C3	1.3351 (18)	C6—H6A	0.9800
N4—C2	1.3377 (18)	C6—H6B	0.9800
N5—C1	1.3396 (19)	C6—H6C	0.9800
N5—H5B	0.9099	C7—C8	1.5058 (18)
N5—H5A	0.9098	C7—H7A	0.9900
N6—C8	1.3237 (18)	C7—H7B	0.9900
N6—N7	1.4151 (15)	O2—H2B	0.8701
N6—H6D	0.9100	O2—H2A	0.8702
C5—N1—N2	126.59 (11)	C4—C3—C6	120.25 (13)
C5—N1—C2	122.89 (12)	C5—C4—C3	120.14 (13)
N2—N1—C2	110.52 (11)	C5—C4—H4	119.9
C1—N2—N1	101.11 (11)	C3—C4—H4	119.9
C2—N3—C1	103.21 (11)	N1—C5—C4	115.77 (12)
C3—N4—C2	116.98 (12)	N1—C5—C7	114.62 (12)
C1—N5—H5B	115.8	C4—C5—C7	129.60 (12)
C1—N5—H5A	118.1	C3—C6—H6A	109.5
H5B—N5—H5A	122.4	C3—C6—H6B	109.5

C8—N6—N7	121.17 (12)	H6A—C6—H6B	109.5
C8—N6—H6D	122.6	C3—C6—H6C	109.5
N7—N6—H6D	115.3	H6A—C6—H6C	109.5
N6—N7—H7D	106.4	H6B—C6—H6C	109.5
N6—N7—H7C	106.6	C5—C7—C8	114.22 (12)
H7D—N7—H7C	108.5	C5—C7—H7A	108.7
N5—C1—N2	121.12 (13)	C8—C7—H7A	108.7
N5—C1—N3	122.86 (12)	C5—C7—H7B	108.7
N2—C1—N3	115.99 (13)	C8—C7—H7B	108.7
N3—C2—N4	129.03 (12)	H7A—C7—H7B	107.6
N3—C2—N1	109.16 (12)	O1—C8—N6	122.79 (13)
N4—C2—N1	121.79 (12)	O1—C8—C7	121.80 (12)
N4—C3—C4	122.42 (13)	N6—C8—C7	115.36 (12)
N4—C3—C6	117.31 (12)	H2B—O2—H2A	101.3
C5—N1—N2—C1	178.41 (13)	C2—N4—C3—C6	−177.54 (12)
C2—N1—N2—C1	−0.65 (14)	N4—C3—C4—C5	−1.4 (2)
N1—N2—C1—N5	−176.73 (13)	C6—C3—C4—C5	177.09 (13)
N1—N2—C1—N3	1.19 (15)	N2—N1—C5—C4	−178.90 (12)
C2—N3—C1—N5	176.63 (13)	C2—N1—C5—C4	0.05 (19)
C2—N3—C1—N2	−1.25 (16)	N2—N1—C5—C7	−0.43 (19)
C1—N3—C2—N4	−177.86 (14)	C2—N1—C5—C7	178.52 (12)
C1—N3—C2—N1	0.72 (14)	C3—C4—C5—N1	0.81 (19)
C3—N4—C2—N3	178.34 (13)	C3—C4—C5—C7	−177.38 (13)
C3—N4—C2—N1	−0.08 (19)	N1—C5—C7—C8	176.75 (11)
C5—N1—C2—N3	−179.15 (12)	C4—C5—C7—C8	−5.0 (2)
N2—N1—C2—N3	−0.05 (15)	N7—N6—C8—O1	−5.9 (2)
C5—N1—C2—N4	−0.4 (2)	N7—N6—C8—C7	171.46 (12)
N2—N1—C2—N4	178.65 (12)	C5—C7—C8—O1	−58.21 (18)
C2—N4—C3—C4	0.98 (19)	C5—C7—C8—N6	124.40 (13)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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N7—H7*D*···O1^{v}	0.91	2.15	2.8715 (16)	135
C7—H7*A*···O2^{vi}	0.99	2.43	3.4215 (18)	175

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+2, −*y*+2, −*z*+1; (iii) −*x*+2, −*y*+1, −*z*; (iv) *x*+1, *y*, *z*; (v) −*x*+1, −*y*+2, −*z*+2; (vi) −*x*+1, −*y*+2, −*z*+1.