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3-(4-Amino-5-thioxo-4,5-dihydro-1H-1,2,4-triazol-3-yl)pyridinium chloride

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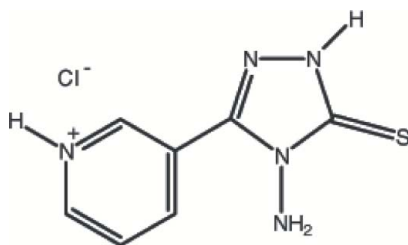
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}—\text{C}) = 0.002$ Å; R factor = 0.029; wR factor = 0.089; data-to-parameter ratio = 16.4.

In the title compound, $\text{C}_7\text{H}_8\text{N}_5\text{S}^+\cdot\text{Cl}^-$, the dihedral angle formed by the pyridine ring with the triazole ring is 10.0 (1)°. There are weak intermolecular hydrogen-bond interactions in the crystal structure, involving the NH and NH_2 groups as donors, and the chloride anion, the S atom in the thioketone group and the unsubstituted ring N atom as acceptors.

Related literature

For related literature, see: Gilchrist (1998); Jian *et al.* (2007).

Experimental

Crystal data

 $\text{C}_7\text{H}_8\text{N}_5\text{S}^+\cdot\text{Cl}^-$ $M_r = 229.69$ Monoclinic, $P2_1/c$ $a = 7.2290$ (14) Å $b = 12.922$ (3) Å $c = 11.253$ (4) Å $\beta = 114.90$ (2)° $V = 953.5$ (4) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.59$ mm⁻¹ $T = 293$ (2) K $0.20 \times 0.15 \times 0.11$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Absorption correction: none

6085 measured reflections

2301 independent reflections

2071 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.017$

3 standard reflections

every 100 reflections

intensity decay: none

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.029$ $wR(F^2) = 0.089$ $S = 0.87$

2301 reflections

140 parameters

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 0.33$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.25$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D—H\cdots A$	$D—H$	$H\cdots A$	$D\cdots A$	$D—H\cdots A$
$\text{N3}—\text{H3A}\cdots\text{Cl1}^i$	0.86	2.21	3.0715 (13)	175
$\text{N5}—\text{H5A}\cdots\text{Cl1}^i$	0.86	2.51	3.1740 (14)	135
$\text{N5}—\text{H5A}\cdots\text{Cl1}^{ii}$	0.86	2.55	3.1999 (15)	133
$\text{N1}—\text{H1A}\cdots\text{S1}^{iii}$	0.87 (2)	2.74 (2)	3.5381 (19)	153.4 (18)
$\text{N1}—\text{H1B}\cdots\text{Cl1}^{iv}$	0.88 (2)	2.51 (2)	3.300 (2)	149.5 (18)
$\text{N1}—\text{H1B}\cdots\text{N4}^{ii}$	0.88 (2)	2.69 (2)	3.1199 (19)	111.7 (16)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL/PC* (Sheldrick, 1997b); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2512).

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supporting information

Acta Cryst. (2008). E64, o138 [https://doi.org/10.1107/S1600536807062848]

3-(4-Amino-5-thioxo-4,5-dihydro-1*H*-1,2,4-triazol-3-yl)pyridinium chloride**Guang-Qiang Zhao, Zhi-Fang Pan and Xue-Xi Tang****S1. Comment**

Five- and six-membered heterocyclic compounds are important constituents that often exist in biologically active natural products and synthetic compounds of medicinal interest (Gilchrist,1998). The title compound (I), is known to coordinate metal centres in a variety of coordination modes involving all combination of the S and N atoms. So it was synthesized and we report here its crystal structure.

In the crystal structure of (I) (Fig. 1), the dihedral angle formed by the pyridine ring (C1—C5/N5) and the plane of the (N2—N4/C6/C7) ring was 10.0 (1)°. The C? S bond length of 1.666 (3)Å is in agreement with that observed before (Jian *et al.*, 2007). In the crystal structure, there are N—H···S and N—H···N and N—H···Cl hydrogen-bond interactions to stabilize the molecular packing (table 2).

S2. Experimental

A mixture of nicotinic acid hydrazide (0.02 mol), carbon disulfide (0.02 mol) and potassium hydroxide (0.02 mol) was stirred with ethanol (50 ml) at 293 K for 5 h, the yellow precipitate was formed, upon collection by filtration,the deposit was washed with ethanol and dried for one day in air.Then dissolved in water (100 ml), hydrazine hydrate was added at 353 K with stirring.then afford the title compound (2.4 g, yield 62%). Single crystals suitable for X-ray measurements were obtained by recrystallization from 10% HCl liquor at room temperature.

S3. Refinement

The H atoms of the amine group and H5B bonded to C5 were found from difference Fourier map and refined freely. The other H atoms were fixed geometrically and allowed to ride on their parent atoms, with N—H and C—H distances of 0.86 and 0.93 Å, respectively, and with $U_{\text{iso}}=1.2U_{\text{eq}}$ of the parent atoms.

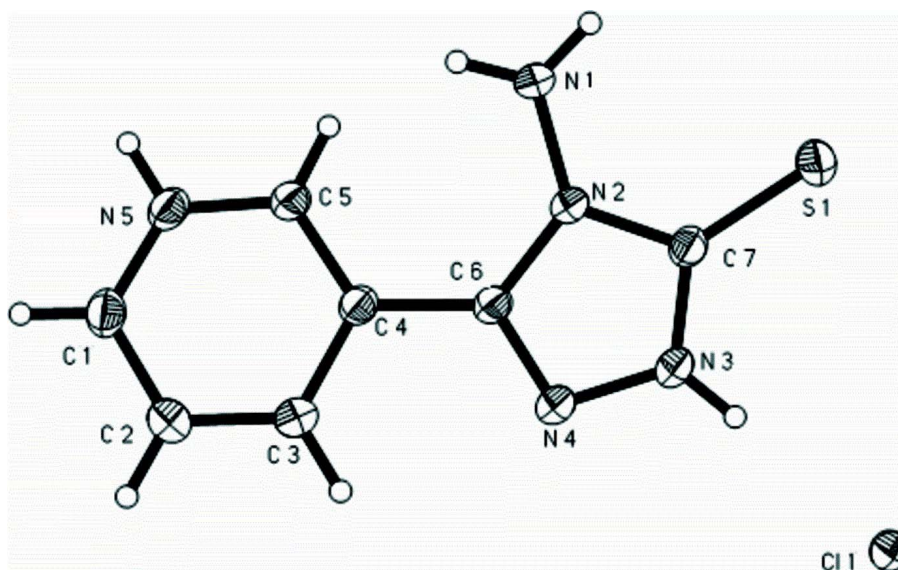


Figure 1

The molecular structure and atom-labeling scheme for (I), with displacement ellipsoids drawn at the 30% probability level.

3-(4-Amino-5-thioxo-4,5-dihydro-1H-1,2,4-triazol-3-yl)pyridinium chloride

Crystal data

$C_7H_8N_5S^+ \cdot Cl^-$

$M_r = 229.69$

Monoclinic, $P2_1/c$

$a = 7.2290$ (14) Å

$b = 12.922$ (3) Å

$c = 11.253$ (4) Å

$\beta = 114.90$ (2)°

$V = 953.5$ (4) Å³

$Z = 4$

$F(000) = 472$

$D_x = 1.600$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 4\text{--}14^\circ$

$\mu = 0.59$ mm⁻¹

$T = 293$ K

Black, yellow

$0.20 \times 0.15 \times 0.11$ mm

Data collection

Enraf–Nonius CAD-4

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

6085 measured reflections

2301 independent reflections

2071 reflections with $I > 2\sigma(I)$

$R_{int} = 0.017$

$\theta_{max} = 28.3^\circ$, $\theta_{min} = 2.5^\circ$

$h = -6 \rightarrow 9$

$k = -17 \rightarrow 16$

$l = -14 \rightarrow 14$

3 standard reflections every 100 reflections

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.089$

$S = 0.87$

2301 reflections

140 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 atoms treated by a mixture of independent

and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 0.3755P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.33 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.24 \text{ e } \text{\AA}^{-3}$$

Extinction correction: *SHELXL*,

$$F_c^* = kFc[1 + 0.001x\text{Fc}^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Extinction coefficient: 0.017 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > \sigma(F^2)$ 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 ($\text{\AA}^2$)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	0.21801 (6)	−0.06699 (3)	−0.00811 (4)	0.04580 (14)
S1	0.27272 (7)	−0.39138 (3)	−0.07565 (4)	0.04812 (14)
N1	0.5768 (2)	−0.45060 (10)	−0.20638 (17)	0.0452 (3)
N2	0.52602 (18)	−0.34784 (9)	−0.19273 (11)	0.0338 (3)
N3	0.41277 (19)	−0.21399 (9)	−0.13746 (12)	0.0389 (3)
H3A	0.3507	−0.1739	−0.1051	0.047*
N4	0.5307 (2)	−0.17818 (9)	−0.19636 (12)	0.0388 (3)
N5	0.9097 (2)	−0.33194 (10)	−0.40767 (12)	0.0410 (3)
H5A	0.9391	−0.3863	−0.4404	0.049*
C1	0.9917 (2)	−0.24258 (13)	−0.41851 (16)	0.0437 (3)
H1C	1.0785	−0.2393	−0.4603	0.052*
C2	0.9465 (3)	−0.15530 (13)	−0.36718 (18)	0.0497 (4)
H2A	1.0010	−0.0918	−0.3747	0.060*
C3	0.8196 (2)	−0.16212 (12)	−0.30438 (16)	0.0431 (3)
H3B	0.7896	−0.1032	−0.2685	0.052*
C4	0.7363 (2)	−0.25703 (10)	−0.29446 (13)	0.0332 (3)
C5	0.7842 (2)	−0.34241 (11)	−0.34905 (14)	0.0386 (3)
C6	0.5992 (2)	−0.26159 (10)	−0.22922 (13)	0.0326 (3)
C7	0.4025 (2)	−0.31668 (11)	−0.13467 (13)	0.0346 (3)
H5B	0.738 (3)	−0.4045 (15)	−0.3468 (18)	0.045 (5)*
H1A	0.569 (3)	−0.4851 (18)	−0.142 (2)	0.061 (6)*
H1B	0.476 (3)	−0.4713 (16)	−0.279 (2)	0.052 (6)*

Atomic displacement parameters ($\text{\AA}^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0618 (3)	0.0326 (2)	0.0622 (3)	0.00315 (15)	0.0448 (2)	0.00064 (14)
S1	0.0556 (3)	0.0435 (2)	0.0606 (3)	0.00530 (16)	0.0394 (2)	0.00822 (16)
N1	0.0599 (8)	0.0267 (6)	0.0637 (9)	0.0062 (6)	0.0405 (8)	0.0028 (6)
N2	0.0410 (6)	0.0273 (5)	0.0391 (6)	0.0041 (4)	0.0227 (5)	−0.0006 (4)
N3	0.0475 (7)	0.0340 (6)	0.0448 (6)	0.0052 (5)	0.0289 (6)	−0.0025 (5)

N4	0.0492 (7)	0.0314 (6)	0.0448 (7)	0.0030 (5)	0.0285 (6)	−0.0026 (5)
N5	0.0513 (7)	0.0378 (6)	0.0430 (7)	0.0076 (5)	0.0288 (6)	−0.0008 (5)
C1	0.0441 (8)	0.0484 (8)	0.0478 (8)	0.0044 (6)	0.0284 (7)	0.0042 (6)
C2	0.0540 (9)	0.0391 (8)	0.0680 (10)	−0.0043 (7)	0.0372 (8)	0.0002 (7)
C3	0.0486 (8)	0.0328 (7)	0.0561 (9)	−0.0003 (6)	0.0300 (7)	−0.0056 (6)
C4	0.0365 (6)	0.0324 (6)	0.0334 (6)	0.0031 (5)	0.0173 (5)	−0.0008 (5)
C5	0.0513 (8)	0.0306 (7)	0.0423 (7)	0.0020 (6)	0.0280 (6)	−0.0007 (5)
C6	0.0382 (7)	0.0294 (6)	0.0329 (6)	0.0021 (5)	0.0176 (5)	−0.0016 (5)
C7	0.0379 (7)	0.0353 (7)	0.0342 (6)	0.0054 (5)	0.0185 (5)	0.0009 (5)

Geometric parameters (Å, °)

S1—C7	1.6667 (15)	N5—C5	1.3339 (19)
N1—N2	1.4032 (16)	N5—H5A	0.8600
N1—H1A	0.87 (2)	C1—C2	1.368 (2)
N1—H1B	0.88 (2)	C1—H1C	0.9300
N2—C6	1.3686 (17)	C2—C3	1.376 (2)
N2—C7	1.3703 (17)	C2—H2A	0.9300
N3—C7	1.3302 (19)	C3—C4	1.391 (2)
N3—N4	1.3625 (17)	C3—H3B	0.9300
N3—H3A	0.8600	C4—C5	1.3762 (19)
N4—C6	1.3029 (17)	C4—C6	1.4622 (19)
N5—C1	1.327 (2)	C5—H5B	0.874 (19)
N2—N1—H1A	106.4 (15)	C1—C2—H2A	120.2
N2—N1—H1B	103.6 (14)	C3—C2—H2A	120.2
H1A—N1—H1B	107 (2)	C2—C3—C4	120.16 (14)
C6—N2—C7	108.38 (11)	C2—C3—H3B	119.9
C6—N2—N1	125.86 (12)	C4—C3—H3B	119.9
C7—N2—N1	125.69 (12)	C5—C4—C3	118.15 (13)
C7—N3—N4	113.76 (11)	C5—C4—C6	122.90 (13)
C7—N3—H3A	123.1	C3—C4—C6	118.94 (12)
N4—N3—H3A	123.1	N5—C5—C4	119.39 (14)
C6—N4—N3	104.32 (12)	N5—C5—H5B	117.2 (13)
C1—N5—C5	123.85 (13)	C4—C5—H5B	123.4 (13)
C1—N5—H5A	118.1	N4—C6—N2	110.35 (12)
C5—N5—H5A	118.1	N4—C6—C4	121.86 (12)
N5—C1—C2	118.87 (14)	N2—C6—C4	127.79 (12)
N5—C1—H1C	120.6	N3—C7—N2	103.17 (12)
C2—C1—H1C	120.6	N3—C7—S1	129.32 (11)
C1—C2—C3	119.57 (15)	N2—C7—S1	127.51 (11)
C7—N3—N4—C6	−1.15 (17)	C7—N2—C6—C4	180.00 (13)
C5—N5—C1—C2	−0.1 (2)	N1—N2—C6—C4	2.9 (2)
N5—C1—C2—C3	0.8 (3)	C5—C4—C6—N4	−169.13 (14)
C1—C2—C3—C4	−0.7 (3)	C3—C4—C6—N4	9.6 (2)
C2—C3—C4—C5	−0.1 (2)	C5—C4—C6—N2	11.6 (2)
C2—C3—C4—C6	−178.92 (15)	C3—C4—C6—N2	−169.64 (14)

C1—N5—C5—C4	−0.8 (2)	N4—N3—C7—N2	1.52 (16)
C3—C4—C5—N5	0.8 (2)	N4—N3—C7—S1	−178.52 (11)
C6—C4—C5—N5	179.58 (13)	C6—N2—C7—N3	−1.28 (15)
N3—N4—C6—N2	0.25 (15)	N1—N2—C7—N3	175.79 (14)
N3—N4—C6—C4	−179.13 (12)	C6—N2—C7—S1	178.76 (11)
C7—N2—C6—N4	0.67 (16)	N1—N2—C7—S1	−4.2 (2)
N1—N2—C6—N4	−176.40 (14)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H $\cdots$ <i>A</i>	<i>D</i> —H	H $\cdots$ <i>A</i>	<i>D</i> $\cdots$ <i>A</i>	<i>D</i> —H $\cdots$ <i>A</i>
N3—H3 <i>A</i> $\cdots$ Cl1	0.86	2.21	3.0715 (13)	175
N5—H5 <i>A</i> $\cdots$ Cl1 ⁱ	0.86	2.51	3.1740 (14)	135
N5—H5 <i>A</i> $\cdots$ Cl1 ⁱⁱ	0.86	2.55	3.1999 (15)	133
N1—H1 <i>A</i> $\cdots$ S1 ⁱⁱⁱ	0.87 (2)	2.74 (2)	3.5381 (19)	153.4 (18)
N1—H1 <i>B</i> $\cdots$ Cl1 ^{iv}	0.88 (2)	2.51 (2)	3.300 (2)	149.5 (18)
N1—H1 <i>B</i> $\cdots$ N4 ⁱⁱ	0.88 (2)	2.69 (2)	3.1199 (19)	111.7 (16)

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