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

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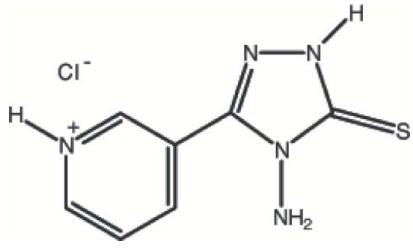
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.029; $wR$ factor = 0.089; data-to-parameter ratio = 16.4.

In the title compound, $C_7H_8N_5S^+\cdot Cl^-$, 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

$C_7H_8N_5S^+\cdot Cl^-$
$M_r$ = 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) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.59 mm$^{-1}$
$T$ = 293 (2) K
0.20 × 0.15 × 0.11 mm

### Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
6085 measured reflections
2301 independent reflections
2071 reflections with $I > 2\sigma(I)$
$R_{int}$ = 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
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.25 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N3—H3$A$···Cl1 | 0.86 | 2.21 | 3.0715 (13) | 175 |
| N5—H5$A$···Cl1$^{i}$ | 0.86 | 2.51 | 3.1740 (14) | 135 |
| N5—H5$A$···Cl1$^{ii}$ | 0.86 | 2.55 | 3.1999 (15) | 133 |
| N1—H1$A$···S1$^{iii}$ | 0.87 (2) | 2.74 (2) | 3.5381 (19) | 153.4 (18) |
| N1—H1$B$···Cl1$^{iv}$ | 0.88 (2) | 2.51 (2) | 3.300 (2) | 149.5 (18) |
| N1—H1$B$···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, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL/PC* (Sheldrick, 1997*b*); 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).

## References

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# supporting information

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## 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 $U_{iso}$=1.2$U_{eq}$ of the parent atoms.

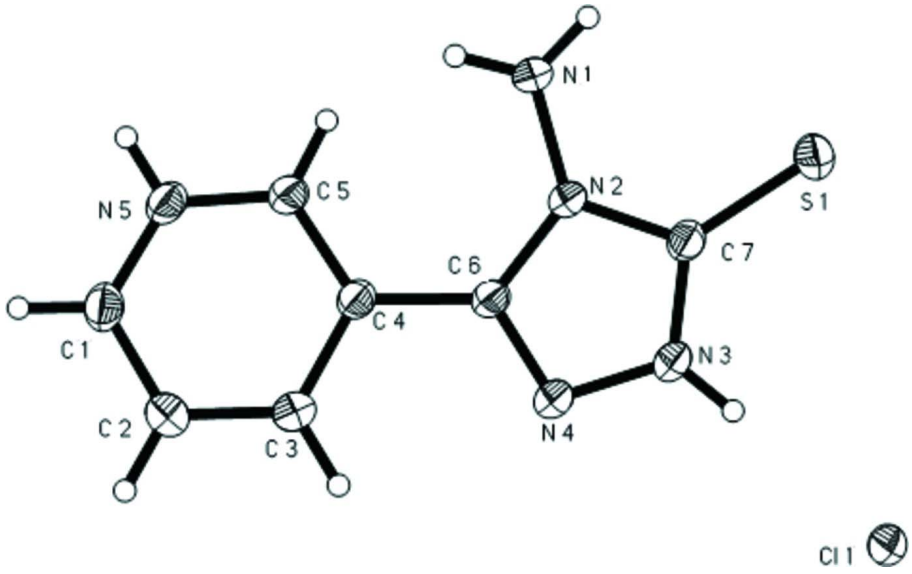


**Figure 1**

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-1*H*-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
$\beta = 114.90\ (2)°$
$V = 953.5\ (4)$ Å$^3$
$Z = 4$
$F(000) = 472$
$D_x = 1.600$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 4$–$14°$
$\mu = 0.59$ mm$^{-1}$
$T = 293$ K
Black, yellow
0.20 × 0.15 × 0.11 mm

*Data collection*

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
6085 measured reflections
2301 independent reflections
2071 reflections with $I > 2\sigma(I)$
$R_{int} = 0.017$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.5°$
$h = -6\rightarrow9$
$k = -17\rightarrow16$
$l = -14\rightarrow14$
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
$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 0.3755P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å$^{-3}$
$\Delta\rho_{min} = -0.25$ e Å$^{-3}$
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N3—H3*A*···Cl1 | 0.86 | 2.21 | 3.0715 (13) | 175 |
| N5—H5*A*···Cl1$^{i}$ | 0.86 | 2.51 | 3.1740 (14) | 135 |
| N5—H5*A*···Cl1$^{ii}$ | 0.86 | 2.55 | 3.1999 (15) | 133 |
| N1—H1*A*···S1$^{iii}$ | 0.87 (2) | 2.74 (2) | 3.5381 (19) | 153.4 (18) |
| N1—H1*B*···Cl1$^{iv}$ | 0.88 (2) | 2.51 (2) | 3.300 (2) | 149.5 (18) |
| N1—H1*B*···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.