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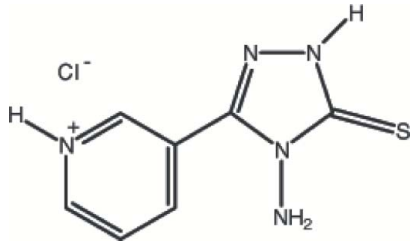
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.089; data-to-parameter ratio = 16.4.

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
β = 114.90 (2)°
V = 953.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 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—H3A···Cl1	0.86	2.21	3.0715 (13)	175
N5—H5A···Cl1^{i}	0.86	2.51	3.1740 (14)	135
N5—H5A···Cl1^{ii}	0.86	2.55	3.1999 (15)	133
N1—H1A···S1^{iii}	0.87 (2)	2.74 (2)	3.5381 (19)	153.4 (18)
N1—H1B···Cl1^{iv}	0.88 (2)	2.51 (2)	3.300 (2)	149.5 (18)
N1—H1B···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).

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supplementary materials

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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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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).

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.

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}=1.2U_{eq} of the parent atoms.

Figures

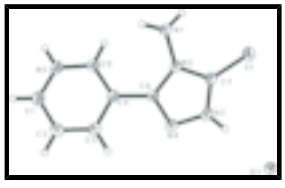

Fig. 1. The molecular structure and atom-labeling scheme for (I), with displacement ellipsoids drawn at the 30% probability level.

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

Crystal data

$C_7H_8N_5S^+\cdot Cl^-$	F_{000} = 472
M_r = 229.69	D_x = 1.600 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.2290 (14) Å	Cell parameters from 25 reflections
b = 12.922 (3) Å	θ = 4–14º
c = 11.253 (4) Å	μ = 0.59 mm^{-1}
β = 114.90 (2)º	T = 293 (2) K
V = 953.5 (4) Å^3	Black, yellow
Z = 4	0.20 × 0.15 × 0.11 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.017
Radiation source: fine-focus sealed tube	θ_{max} = 28.3º
Monochromator: graphite	θ_{min} = 2.5º
T = 293(2) K	h = −6→9
ω scans	k = −17→16
Absorption correction: none	l = −14→14
6085 measured reflections	3 standard reflections
2301 independent reflections	every 100 reflections
2071 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.029	$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 0.3755P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.089	$(\Delta/\sigma)_{max} < 0.001$
S = 0.87	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
2301 reflections	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
140 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.017 (2)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	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 (Å^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, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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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.

Fig. 1