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Bis[4-amino-3-methyl-1*H*-1,2,4-triazole-5(4*H*)-thione]dichlorocopper(II) dihydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.025
wR factor = 0.067
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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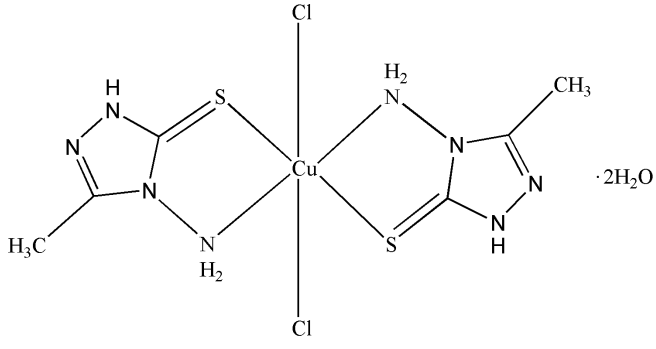
In the title compound, $[Cu(amt)_2Cl_2]\cdot 2H_2O$, where amt is 4-amino-1,2,4-triazole-5-thione ($C_3H_6N_4S$), the Cu^{II} atom is surrounded by two N atoms, two S atoms from two amt ligands, and two Cl ions, forming a distorted $CuCl_2N_2S_2$ octahedron. The copper complex, with the Cu^{II} cation located on a center of inversion, and solvent water molecules are connected together by five kinds of intermolecular hydrogen bonds, leading to the formation of a three-dimensional network structure.

Comment

1,2,4-Triazole and its derivatives display a broad range of biological activity, finding application as antitumor, antibacterial, antifungal and antiviral agents (Jantova *et al.*, 1998; Holla *et al.*, 1996). Although complexes of these ligands have been reported for a variety of transition metals, relatively few crystal structures of such complexes have been published (Jha *et al.*, 1994; Dubey *et al.*, 1994). Because of the potential applications of these compounds, we focused our attention on synthesizing them, using 1,2,4-triazole derivatives. We present here the structure of the title compound, $[Cu(amt)_2Cl_2]\cdot 2H_2O$, (I).



(I)

In (I), two amt ligands coordinate to the Cu^{II} atom through two amino N atoms and two thiocarbonyl S atoms. Each Cu^{II} atom, on an inversion center, is at the center of a distorted octahedron, with a distinct Jahn–Teller effect (Fig. 1). The equatorial plane consists of two amino N atoms and two S atoms from two amt ligands. The two axial positions are filled by two Cl anions. The Cu atom is located on the plane C1/C2/C3/N1/N2/N3/N4/S1; the r.m.s. deviation from the plane is 0.0277 Å. In the crystal structure, there are five kinds of intermolecular hydrogen bonds with H···A distances in the 1.86–2.54 Å range, linking the mononuclear units into a three-dimensional network structure (Fig. 2 and Table 2).

Experimental

The title compound was synthesized by a hydrothermal method from a mixture of 4-amino-1,2,4-triazole-5-thione (2 mmol, 0.38 g), $CuCl_2{\cdot}2H_2O$ (1 mmol, 0.17 g) and water (20 ml) in a 30 ml Teflon-lined stainless steel reactor. The solution was heated to 415 K for four days. After the reaction system was slowly cooled to room temperature, blue block-shaped crystals were collected and washed with distilled water.

Crystal data

$[Cu(C_3H_6N_4S)_2Cl_2]{\cdot}2H_2O$
M_r = 430.83
Monoclinic, $P2_1/c$
a = 9.4614 (8) Å
b = 10.0012 (9) Å
c = 8.4042 (7) Å
β = 93.028 (2)°
V = 794.14 (12) Å^3
Z = 2
D_x = 1.802 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2880 reflections
θ = 2.5–25.2°
μ = 1.99 mm^{-1}
T = 298 (2) K
Block, blue
0.37 × 0.29 × 0.26 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan *SADABS*; Sheldrick, 1996
T_{min} = 0.506, T_{max} = 0.606
4082 measured reflections
1427 independent reflections
1351 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 25.2°
$h = -7 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -10 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.067
S = 1.12
1427 reflections
104 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0378P)^2 + 0.2591P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.42 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—N4	2.0519 (16)	Cu1—Cl1	2.9008 (5)
Cu1—S1	2.3223 (5)		
N4—Cu1—S1	89.03 (4)	S1—Cu1—Cl1	93.040 (17)
N4—Cu1—Cl1	92.93 (4)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	1.86	2.706 (2)	169
N4—H4A···Cl1^{ii}	0.90	2.54	3.3996 (17)	161
N4—H4B···Cl1^{iii}	0.90	2.43	3.2893 (16)	161
O1—H1A···N2	0.807 (15)	2.098 (15)	2.858 (2)	157 (2)
O1—H1B···Cl1^{iv}	0.834 (15)	2.321 (15)	3.1543 (17)	177 (2)

Symmetry codes: (i) $x, \frac{3}{2}-y, \frac{1}{2}+z$; (ii) $1-x, y-\frac{1}{2}, \frac{3}{2}-z$; (iii) $x, \frac{5}{2}-y, z-\frac{1}{2}$; (iv) $2-x, y-\frac{1}{2}, \frac{3}{2}-z$.

H atoms of the water molecule were located in a difference Fourier map and refined with O—H and H···H distances restrained to 0.82 (2) Å and 1.39 (1) Å, respectively, and with U_{iso} = 1.2U_{eq}(parent atom). The other H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^3—H = 0.96 Å with U_{iso} = 1.5U_{eq}(parent atom), N—H = 0.86 or 0.90 Å with U_{iso} = 1.2U_{eq}(parent atom).

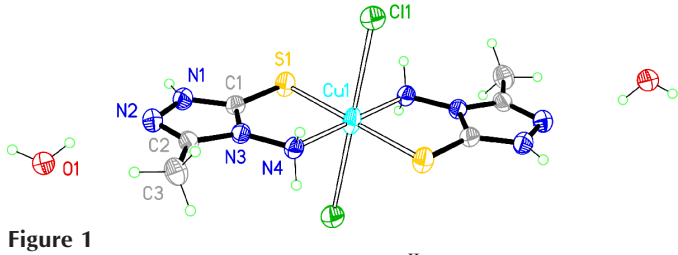


Figure 1
The coordination environment of the Cu^{II} ion in (I), with the atom numbering, showing displacement ellipsoids at the 50% probability level. Unlabeled atoms are related to labeled atoms by $1-x, 2-y, 2-z$.

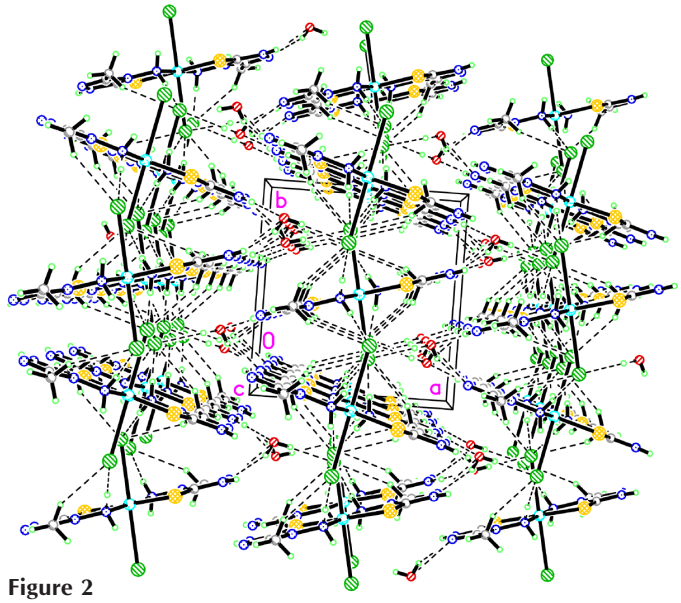


Figure 2
The three-dimensional network formed by hydrogen bonds (shown as dashed lines).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL*97 (Bruker, 2002).

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