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A copper(I) complex of 4-amino-3-thioxo-6-methyl-2,3,4,5-tetrahydro-1,2,4-triazin-5-one

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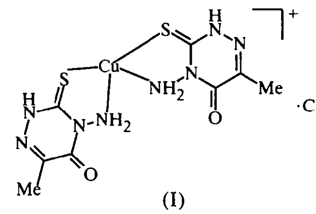
Abstract

In the crystal of the title compound, bis(4-amino-κ*N*-3-thioxo-κ*S*-6-methyl-2,3,4,5-tetrahydro-1,2,4-triazin-5-one)copper(I) chloride, [Cu($C_4H_6N_4O$)$_2$]Cl, the asymmetric unit contains one-half of the cation, with the other half related by a crystallographic twofold axis; the Cl and Cu atoms lie on the twofold axis. The Cu atom is in a distorted tetrahedral geometry. The inversion-related molecules are linked by N···O [2.805 (3) Å] short contacts to form an infinite zigzag chain along the *c* direction. The Cl atom is involved in both intra- and interchain N—H···Cl hydrogen bonds.

Comment

Many metal complexes of chelating agents derived from 4-amino-3-thioxo-6-methyl-2,3,4,5-tetrahydro-1,2,4-triazin-5-one have been studied (Iskander *et al.*, 1989; Sharda *et al.*, 1994; Dubey & Beena, 1991). Some complexes of triazine have been shown to possess fungicidal activity (Bala *et al.*, 1978). As part of our studies on the synthesis and characterization, as well as the potential fungicidal activities, of triazine derivatives, we report here the crystal structure of the title compound, (I).



(I)

The asymmetric unit of (I) contains one-half of the [Cu($C_4H_6N_4O$)$_2$]$^+$ cation, with the other half related by a crystallographic twofold axis; both the Cl^- anion and Cu atom lie on the twofold axis. The Cu atom is in a heavily distorted tetrahedral geometry, with the bond angles around it ranging from 84.75 (5) to 120.78 (6)°. The Cu1—S and Cu1—N bond lengths agree with reported values (Orpen *et al.*, 1989).

The inversion-related molecules are linked by N4···O1(1 − *x*, 1 − *y*, 1 − *z*) [2.805 (3) Å] short contacts to form an infinite zigzag ribbon-like structure along the *c* direction. Interestingly, the narrow N—H···O angles involving N4 and O1 (103 and 102°) decrease the involvement of any N—H···O hydrogen bonding in the chain. The Cl^- anion is located in the cavity of the chain and is linked to it through N4—H4*A*···Cl(1 − *x*, 1 − *y*, 1 − *z*) and N4—H4*B*···Cl1 hydrogen bonds (Table 3). Adjacent chains are interlinked by N1—H1···Cl1($x - \frac{1}{2}$, $y - \frac{1}{2}$, $\frac{1}{2} - z$) hydrogen bonds. Other short contacts observed in the

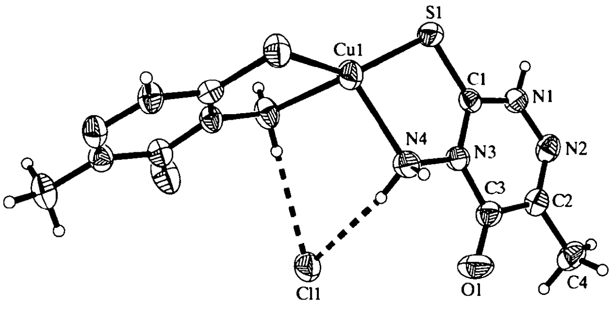


Fig. 1. The structure of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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structure are Cu1···C1 and Cu1···C1($1-x$, y, $\frac{1}{2}-z$) of 3.009 (2) Å, and Cu1···N3 and Cu1···N3($1-x$, y, $\frac{1}{2}-z$) of 3.016 (2) Å.

Experimental

The title complex was prepared from $CuCl_2 \cdot 2H_2O$ and a large excess of 4-amino-3-thioxo-6-methyl-2,3,4,5-tetrahydro-1,2,4-triazin-5-one as described by Iskander *et al.* (1989).

Crystal data

$[Cu(C_4H_6N_4O)_2]Cl$
$M_r = 415.37$
Orthorhombic
Pbcn
$a = 12.8678\,(5)$ Å
$b = 11.4996\,(4)$ Å
$c = 10.0894\,(3)$ Å
$V = 1492.97\,(9)$ Å^3
$Z = 4$
$D_x = 1.848$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 4351 reflections
$\theta = 3.12$–$27.50°$
$\mu = 1.941$ mm^{-1}
$T = 293\,(2)$ K
Plate
0.44 × 0.14 × 0.12 mm
Yellow

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan, empirical (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.48$, $T_{max} = 0.80$

9130 measured reflections
1709 independent reflections
1316 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.080$
$S = 1.04$
1709 reflections
101 parameters
H atoms constrained
$w = 1/[\sigma^2(F_o^2) + (0.0261P)^2 + 1.6821P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu1	1/2	0.25691 (4)	1/4	0.03871 (15)
S1	0.33435 (5)	0.19455 (6)	0.25541 (7)	0.03613 (17)
O1	0.34822 (14)	0.54724 (18)	0.5385 (2)	0.0500 (6)
N1	0.18029 (15)	0.30452 (18)	0.3715 (2)	0.0305 (5)
N2	0.12836 (16)	0.38666 (19)	0.4418 (2)	0.0329 (5)
N3	0.33943 (14)	0.38365 (17)	0.4130 (2)	0.0277 (4)
N4	0.44895 (15)	0.38210 (19)	0.3992 (2)	0.0358 (5)
C1	0.28311 (18)	0.2987 (2)	0.3509 (2)	0.0264 (5)
C2	0.18162 (18)	0.4675 (2)	0.4976 (2)	0.0307 (6)
C3	0.29512 (19)	0.4733 (2)	0.4879 (3)	0.0315 (6)
C4	0.1283 (2)	0.5580 (3)	0.5784 (3)	0.0428 (7)
Cl1	1/2	0.64603 (9)	1/4	0.0438 (2)

Table 2. *Selected geometric parameters (Å, °)*

Cu1—N4	2.184 (2)	Cu1—S1	2.2497 (6)
N4—Cu1—N4^i	97.5 (1)	N4—Cu1—S1	84.75 (5)
N4—Cu1—S1^i	120.78 (6)	S1^i—Cu1—S1	142.82 (4)

Symmetry code: (i) $1-x, y, \frac{1}{2}-z$.

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4B···Cl1	0.90	2.58	3.451 (2)	163
N1—H1···Cl1^i	0.86	2.37	3.195 (2)	162
N4—H4A···Cl1^{ii}	0.90	2.76	3.614 (2)	158
C4—H4C···N2^{iii}	0.96	2.45	3.370 (3)	160
N4—H4A···O1^{ii}	0.90	2.46	2.805 (3)	103
N4—H4B···O1^{ii}	0.90	2.48	2.805 (3)	102

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

After checking their presence in the difference map, all H atoms were fixed geometrically and allowed to ride on their attached atoms.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 1990).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1282). Services for accessing these data are described at the back of the journal.

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