metal-organic compounds

catena-Poly[[(1,10-phenanthroline-$\kappa^2 N,N'$)copper(I)]-μ-thiocyanato-$\kappa^2 N$:S]

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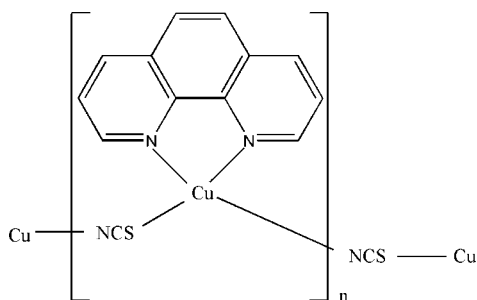Correspondence e-mail: honglizhang1968@yahoo.cn

In the title complex, [Cu(NCS)(C$_{12}$H$_8$N$_2$)]$_n$, the Cu^I ion is in a distorted tetrahedral CuN$_3$S coordination geometry. The thiocyanate ligand acts as bridging ligand, forming chains along [100]. A crystallographic mirror plane runs through the Cu^I ion, the thiocyanate ligand and the middle of the phenanthroline ligand.

Related literature

For related structures, see: Shi *et al.* (2006); Tadashi *et al.* (1990).

Experimental

Crystal data

[Cu(NCS)(C$_{12}$H$_8$N$_2$)]	V = 1234.4 (4) Å^3
M_r = 301.82	Z = 4
Orthorhombic, $Pnma$	Mo $K\alpha$ radiation
a = 7.9744 (15) Å	μ = 1.92 mm^{-1}
b = 11.948 (2) Å	T = 298 K
c = 12.956 (2) Å	0.23 × 0.15 × 0.15 mm

Data collection

Bruker SMART APEX CCD diffractometer	6226 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1421 independent reflections
T_{min} = 0.667, T_{max} = 0.762	1146 reflections with $I > 2\sigma(I)$
	R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028	89 parameters
$wR(F^2)$ = 0.080	H-atom parameters constrained
S = 1.03	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
1421 reflections	$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5123).

References

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Tadashi, T., Naofumi, W., Michio, N., Yoneichiro, M., Mitsuo, M., Shigeru, O. & Yoshihiko, S. (1990). *Bull. Chem. Soc. Jpn*, **63**, 364–369.

supporting information

catena-Poly[[(1,10-phenanthroline-*κ²N,N′*)copper(I)]-*μ*-thiocyanato-*κ²N:S*]

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S1. Comment

1,10-phenanthroline and thiocyanate anions play an important role in modern coordination chemistry and many complexes have been published with them as ligands (e.g. Shi *et al.*, 2006; Tadashi *et al.* (1990). We originally tried to prepare a new divalent Cu(II) complex with these two ligands, but the title monovalent Cu(I) complex was fortuitously obtained. Herein we report its crystal structure.

  Fig. 1 shows part of the title complex. The Cu^I ion is coordinated by three N atoms and one S atom, and is in a distorted tetrahedral coordination environment. The thiocyanate ligand acts as bridging forming a 1-D chain with a Cu···Cu distance of 5.9960 (9) Å.

S2. Experimental

A 5 ml H_2O solution of $Cu(ClO_4)_2 6H_2O$ (0.2000 g, 0.54 mmol) was added to a 10 ml methanol solution of 1,10-phenanthroline (0.1070 g, 0.54 mmol), and the mixture was stirred for a few minutes, then a 5 ml H_2O solution of NaNCS (0.0875 g, 1.08 mmol) was added dropwise and the mixture was stirred for a few minutes and then placed in a Teflon-lined autoclave and heated at 433K for 144 h at autogenous pressure. After the contents of the autoclave were cooled to room temperature, the red single crystals were obtained.

S3. Refinement

All H atoms were placed in calculated positions and refined as riding with C—H = 0.93 Å, $U_{iso} = 1.2U_{eq}(C)$.
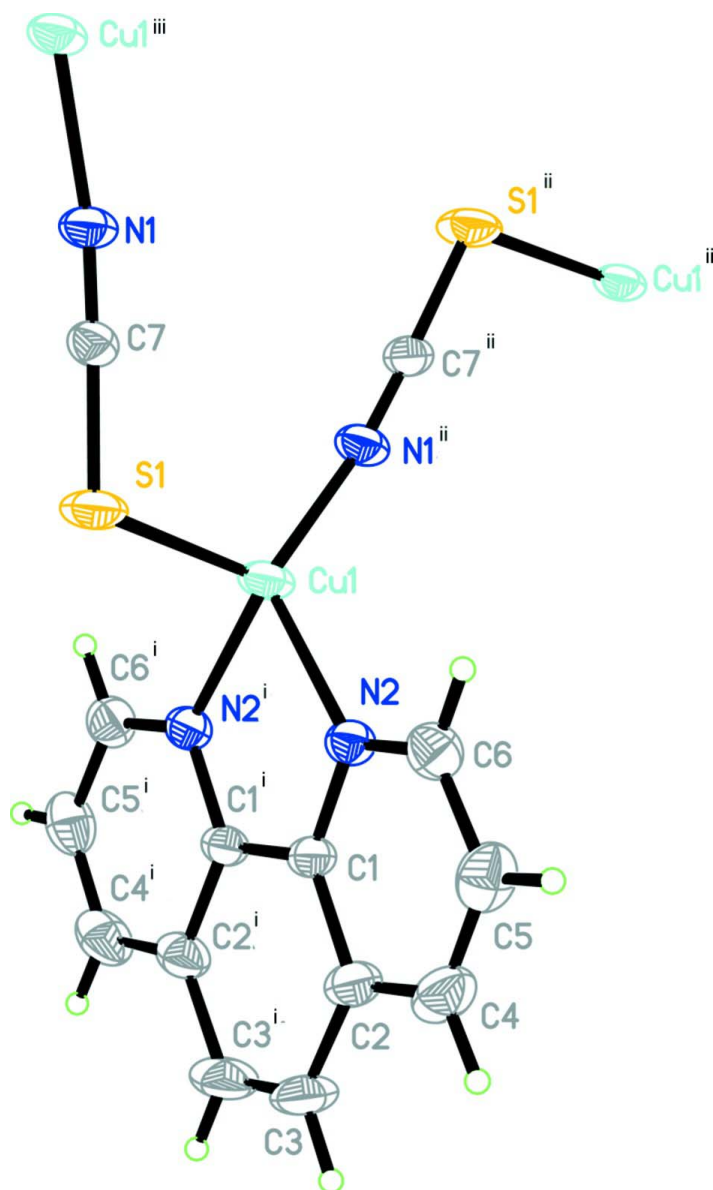
Figure 1

Part of the 1-D chain of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. [symmetry codes: (i): x, $-y + 3/2$, z; (ii): $x - 1/2$, y, $-z + 3/2$; (iii): $x + 1/2$, y, $-z + 3/2$]

***catena*-Poly[[(1,10-phenanthroline-*κ²N,N′*)copper(I)]- *μ*-thiocyanato-*κ²N:S*]**

Crystal data

[Cu(NCS)(C$_{12}$H$_8$N$_2$)]	$F(000) = 608$
$M_r = 301.82$	$D_x = 1.624$ Mg m^{-3}
Orthorhombic, *Pnma*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2n	Cell parameters from 2278 reflections
$a = 7.9744$ (15) Å	$\theta = 2.3–27.9°$
$b = 11.948$ (2) Å	$\mu = 1.92$ mm^{-1}
$c = 12.956$ (2) Å	$T = 298$ K
$V = 1234.4$ (4) Å^3	Block, red
$Z = 4$	$0.23 \times 0.15 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD diffractometer	6226 measured reflections
Radiation source: fine-focus sealed tube	1421 independent reflections
Graphite monochromator	1146 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.028$
Absorption correction: multi-scan ($SADABS$; Sheldrick, 1996)	$\theta_{max} = 27.0°$, $\theta_{min} = 2.3°$
	$h = -9 \rightarrow 10$
	$k = -15 \rightarrow 15$
$T_{min} = 0.667$, $T_{max} = 0.762$	$l = -5 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.028$	H-atom parameters constrained
$wR(F^2) = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
1421 reflections	$(\Delta/\sigma)_{max} = 0.001$
89 parameters	$\Delta\rho_{max} = 0.29$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.28$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: $SHELXTL$ (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0039 (10)

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.13936 (17)	0.68993 (15)	1.13076 (12)	0.0406 (4)
C2	0.1011 (2)	0.63175 (18)	1.22243 (13)	0.0543 (5)
C3	0.0628 (3)	0.69444 (18)	1.31338 (13)	0.0712 (6)
H3	0.0372	0.6565	1.3740	0.085*
C4	0.1051 (2)	0.51479 (18)	1.21873 (16)	0.0669 (6)
H4	0.0792	0.4731	1.2772	0.080*
C5	0.1468 (2)	0.46262 (19)	1.12958 (17)	0.0648 (6)
H5	0.1491	0.3849	1.1262	0.078*
C6	0.1862 (2)	0.52657 (16)	1.04275 (16)	0.0528 (5)
H6	0.2175	0.4895	0.9826	0.063*
C7	0.5834 (3)	0.7500	0.79202 (17)	0.0436 (5)
Cu1	0.23713 (4)	0.7500	0.92283 (2)	0.04865 (16)
N1	0.6286 (2)	0.7500	0.70801 (14)	0.0487 (5)
N2	0.18111 (17)	0.63783 (12)	1.04164 (11)	0.0412 (3)
S1	0.52660 (9)	0.7500	0.91372 (4)	0.0702 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0342 (8)	0.0577 (10)	0.0300 (8)	−0.0010 (7)	0.0005 (6)	0.0033 (7)
C2	0.0467 (10)	0.0768 (13)	0.0392 (9)	−0.0047 (9)	0.0023 (8)	0.0131 (9)
C3	0.0708 (12)	0.1093 (18)	0.0335 (9)	−0.0073 (11)	0.0146 (9)	0.0111 (9)
C4	0.0664 (14)	0.0748 (15)	0.0593 (13)	−0.0081 (10)	−0.0013 (10)	0.0283 (11)
C5	0.0638 (13)	0.0510 (11)	0.0796 (16)	−0.0007 (10)	−0.0080 (12)	0.0169 (11)
C6	0.0548 (11)	0.0519 (11)	0.0517 (11)	0.0028 (9)	−0.0052 (9)	−0.0009 (9)
C7	0.0417 (12)	0.0567 (15)	0.0323 (12)	0.000	−0.0043 (10)	0.000
Cu1	0.0589 (3)	0.0631 (3)	0.0240 (2)	0.000	0.00043 (12)	0.000
N1	0.0563 (13)	0.0619 (13)	0.0278 (9)	0.000	0.0038 (9)	0.000
N2	0.0423 (7)	0.0469 (8)	0.0345 (7)	0.0000 (6)	−0.0012 (6)	0.0021 (6)
S1	0.0499 (4)	0.1360 (8)	0.0245 (3)	0.000	0.0024 (3)	0.000

Geometric parameters (Å, º)

C1—N2	1.353 (2)	C5—H5	0.9300
C1—C2	1.410 (2)	C6—N2	1.330 (2)
C1—C1^i	1.435 (4)	C6—H6	0.9300
C2—C4	1.399 (3)	C7—N1	1.147 (3)
C2—C3	1.429 (2)	C7—S1	1.640 (2)
C3—C3^i	1.328 (4)	Cu1—N1^{ii}	1.9033 (19)
C3—H3	0.9300	Cu1—N2^i	2.0893 (14)
C4—C5	1.354 (3)	Cu1—N2	2.0893 (14)
C4—H4	0.9300	Cu1—S1	2.3113 (9)
C5—C6	1.396 (3)	N1—Cu1^{iii}	1.9033 (19)
N2—C1—C2	123.04 (17)	N2—C6—C5	123.3 (2)
N2—C1—C1^i	117.39 (9)	N2—C6—H6	118.4
C2—C1—C1^i	119.55 (11)	C5—C6—H6	118.4
C4—C2—C1	117.32 (19)	N1—C7—S1	177.7 (2)
C4—C2—C3	123.83 (19)	N1^{ii}—Cu1—N2^i	123.98 (6)
C1—C2—C3	118.84 (18)	N1^{ii}—Cu1—N2	123.98 (6)
C3^i—C3—C2	121.60 (11)	N2^i—Cu1—N2	79.80 (8)
C3^i—C3—H3	119.2	N1^{ii}—Cu1—S1	114.12 (6)
C2—C3—H3	119.2	N2^i—Cu1—S1	104.55 (4)
C5—C4—C2	119.65 (19)	N2—Cu1—S1	104.55 (4)
C5—C4—H4	120.2	C7—N1—Cu1^{iii}	171.3 (2)
C2—C4—H4	120.2	C6—N2—C1	117.26 (16)
C4—C5—C6	119.4 (2)	C6—N2—Cu1	129.99 (13)
C4—C5—H5	120.3	C1—N2—Cu1	112.71 (11)
C6—C5—H5	120.3	C7—S1—Cu1	108.96 (9)
N2—C1—C2—C4	−0.9 (2)	C1^i—C1—N2—C6	177.93 (12)
C1^i—C1—C2—C4	−179.14 (12)	C2—C1—N2—Cu1	−178.16 (12)
N2—C1—C2—C3	178.35 (16)	C1^i—C1—N2—Cu1	0.08 (11)
C1^i—C1—C2—C3	0.1 (2)	N1^{ii}—Cu1—N2—C6	58.10 (18)

C4—C2—C3—C3ⁱ	179.09 (13)	N2ⁱ—Cu1—N2—C6	−177.60 (14)
C1—C2—C3—C3ⁱ	−0.2 (2)	S1—Cu1—N2—C6	−75.07 (16)
C1—C2—C4—C5	0.9 (3)	N1ⁱⁱ—Cu1—N2—C1	−124.40 (10)
C3—C2—C4—C5	−178.38 (19)	N2ⁱ—Cu1—N2—C1	−0.09 (13)
C2—C4—C5—C6	0.4 (3)	S1—Cu1—N2—C1	102.44 (10)
C4—C5—C6—N2	−1.7 (3)	N1—C7—S1—Cu1	180.00 (2)
S1—C7—N1—Cu1ⁱⁱⁱ	0.00 (2)	N1ⁱⁱ—Cu1—S1—C7	0.0
C5—C6—N2—C1	1.7 (3)	N2ⁱ—Cu1—S1—C7	−138.50 (4)
C5—C6—N2—Cu1	179.08 (13)	N2—Cu1—S1—C7	138.50 (4)
C2—C1—N2—C6	−0.3 (2)		

Symmetry codes: (i) *x*, −*y*+3/2, *z*; (ii) *x*−1/2, *y*, −*z*+3/2; (iii) *x*+1/2, *y*, −*z*+3/2.