metal-organic compounds

catena-Poly[[[2-[(2-methylaminoethyl-imino)methyl]-4-nitrophenolato]-copper(II)]-μ-thiocyanato]

Ping Zhang

Department of Chemistry, Xianyang Normal University, Xianyang 712000, People's Republic of China
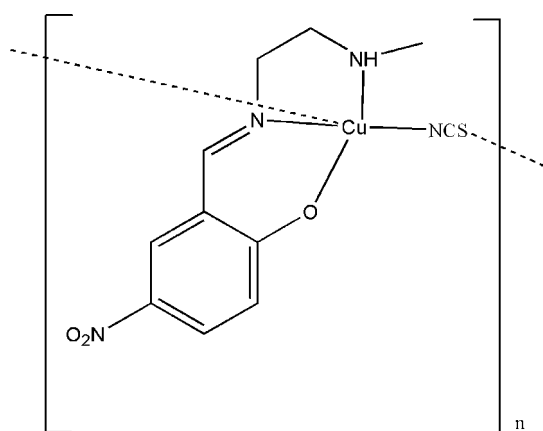
Correspondence e-mail: zhangping491@163.com

The title compound, $[\mathrm{Cu}(\mathrm{C_{10}H_{12}N_3O_3})(\mathrm{NCS})]$, is a thiocyanate-bridged polynuclear copper(II) complex. The $\mathrm{Cu^{II}}$ atom is pentacoordinated by two N atoms and one O atom of the Schiff base ligand, 4-nitro-2-[(2-methylaminoethylimino)methyl]phenol (HNP), and by two N atoms from symmetry-related thiocyanate anions, forming a slightly distorted square-pyramidal coordination configuration. The Cu(NP) units are linked through the bridging thiocyanate anions, forming chains running along the c direction.

Related literature

For related literature, see: Zhang (2004).

Experimental

Crystal data

$[\mathrm{Cu}(\mathrm{C_{10}H_{12}N_3O_3})(\mathrm{NCS})]$	V = 1348.8 (3) Å^3
M_r = 343.85	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 10.6144 (12) Å	μ = 1.79 mm^{-1}
b = 11.2212 (13) Å	T = 298 (2) K
c = 11.7323 (14) Å	$0.12 \times 0.12 \times 0.09$ mm
β = 105.160 (2)°	

Data collection

Bruker SMART CCD area-detector diffractometer	11164 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3073 independent reflections
T_{min} = 0.814, T_{max} = 0.856	2257 reflections with $I > 2\sigma(I)$
	R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.097	
S = 1.01	
3073 reflections	$\Delta\rho_{max}$ = 0.56 e Å^{-3}
185 parameters	$\Delta\rho_{min}$ = −0.39 e Å^{-3}
1 restraint	

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2018).

References

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supplementary materials

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Comment

Recently, the author has reported the *cf.*ystal structure of a mononuclear copper(II) complex derived from the Schiff base ligand 1-[3-(cyclohexylamino)propyliminomethyl]-2-naphthol (Zhang, 2004). As an extension of this work, on the structural characterization of Schiff base copper compounds, we report here on the crystal structure of the new title polynuclear complex.

The title compound is a thiocyanato-bridged polynuclear copper(II) complex, as shown in Fig. 1. The Cu[II] atom is penta-coordinated by the NNO donor atoms of the Schiff base ligand, 4-nitro-2-[(2-methylaminoethylimino)methyl]phenol(HNP), and by two N atoms from two thiocyanate anions, so forming a slightly distorted square pyramidal coordination configuration. The Cu(NP) units are linked through the bridging thiocyanate anions, forming chains running along the *c* direction. The bond lengths and angles (Table 1) are within normal ranges and comparable to the values of the complex referred to above.

Experimental

N-Methyl-1,2-diaminoethane (0.1 mmol, 7.4 mg) and 5-nitrosalicylaldehyde (0.1 mmol, 16.7 mg) were dissolved in ethanol (10 cm^3). The mixture was stirred for 10 min to give a clear yellow solution. To the solution was added an aqueous solution (2 cm^3) of ammonium thiocyanate (0.1 mmol, 7.6 mg) and CuCl$_2$·2H$_2$O (0.1 mmol, 17.1 mg), with stirring. The mixture was stirred at room temperature for 1 h and then filtered. After keeping the brown filtrate in air for five days, blue block-shaped crystals were formed with high yield (87% based on 5-nitrosalicylaldehyde).

Refinement

The N—H H-atom was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. The other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H distances in the range 0.93–0.97 Å, and with $U_{iso}(H) = 1.2U_{eq}(C)$ and $1.5U_{eq}$(methyl C).
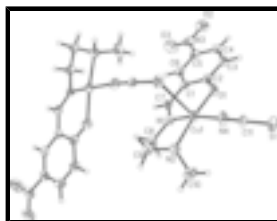
Figures

Fig. 1. The structure of the title complex, showing the atom-numbering scheme with displacement ellipsoids drawn at the 30% probability level.

supplementary materials

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Crystal data

$[Cu(C_{10}H_{12}N_3O_3)(NCS)]$	$F_{000} = 700$
$M_r = 343.85$	$D_x = 1.693$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2670 reflections
$a = 10.6144$ (12) Å	$\theta = 2.5–25.9°$
$b = 11.2212$ (13) Å	$\mu = 1.79$ mm^{-1}
$c = 11.7323$ (14) Å	$T = 298$ (2) K
$\beta = 105.160$ (2)°	Block, blue
$V = 1348.8$ (3) Å^3	$0.12 \times 0.12 \times 0.09$ mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3073 independent reflections
Radiation source: fine-focus sealed tube	2257 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
$T = 298(2)$ K	$\theta_{max} = 27.5°$
ω scans	$\theta_{min} = 2.0°$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -13\rightarrow13$
$T_{min} = 0.814$, $T_{max} = 0.856$	$k = -14\rightarrow14$
11164 measured reflections	$l = -15\rightarrow14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.037$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.097$	$w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.1747P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
3073 reflections	$\Delta\rho_{max} = 0.56$ e Å^{-3}
185 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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}
Cu1	0.36112 (3)	0.75795 (3)	0.17031 (3)	0.04190 (13)
S1	0.57494 (8)	0.78609 (8)	0.57534 (6)	0.0540 (2)
O1	0.3209 (2)	0.59558 (17)	0.20010 (15)	0.0497 (5)
O2	0.0453 (2)	0.1542 (2)	−0.0731 (2)	0.0775 (7)
O3	−0.0014 (3)	0.2839 (2)	−0.2116 (2)	0.0934 (9)
N1	0.2374 (2)	0.76051 (18)	0.01592 (19)	0.0381 (5)
N2	0.3757 (2)	0.9355 (2)	0.1417 (2)	0.0499 (6)
N3	0.0476 (3)	0.2569 (2)	−0.1091 (3)	0.0563 (7)
N4	0.4606 (3)	0.7728 (2)	0.3345 (2)	0.0584 (7)
C1	0.1869 (2)	0.5502 (2)	0.0052 (2)	0.0345 (5)
C2	0.2510 (2)	0.5216 (2)	0.1262 (2)	0.0394 (6)
C3	0.2321 (3)	0.4050 (3)	0.1644 (2)	0.0461 (7)
H3	0.2665	0.3859	0.2436	0.055*
C4	0.1658 (3)	0.3198 (3)	0.0900 (3)	0.0479 (7)
H4	0.1558	0.2437	0.1179	0.057*
C5	0.1131 (2)	0.3482 (2)	−0.0283 (2)	0.0415 (6)
C6	0.1200 (2)	0.4613 (2)	−0.0692 (2)	0.0388 (6)
H6	0.0795	0.4792	−0.1477	0.047*
C7	0.1822 (2)	0.6689 (2)	−0.0415 (2)	0.0386 (6)
H7	0.1354	0.6803	−0.1196	0.046*
C8	0.2156 (3)	0.8792 (2)	−0.0400 (2)	0.0472 (7)
H8A	0.1386	0.9158	−0.0251	0.057*
H8B	0.2024	0.8720	−0.1247	0.057*
C9	0.3341 (3)	0.9543 (3)	0.0121 (2)	0.0502 (7)
H9A	0.4043	0.9323	−0.0226	0.060*
H9B	0.3138	1.0378	−0.0050	0.060*
C10	0.2983 (4)	1.0049 (3)	0.2052 (3)	0.0852 (12)
H10A	0.3056	1.0882	0.1894	0.128*
H10B	0.3302	0.9907	0.2885	0.128*
H10C	0.2084	0.9811	0.1793	0.128*
C11	0.5081 (3)	0.7796 (2)	0.4342 (2)	0.0393 (6)
H2	0.4577 (16)	0.964 (3)	0.171 (3)	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0555 (2)	0.0438 (2)	0.02492 (17)	−0.00736 (15)	0.00780 (14)	−0.00031 (13)
S1	0.0494 (4)	0.0807 (6)	0.0288 (3)	−0.0122 (4)	0.0044 (3)	−0.0053 (3)
O1	0.0690 (13)	0.0490 (12)	0.0253 (9)	−0.0143 (10)	0.0019 (9)	0.0051 (8)
O2	0.0818 (17)	0.0491 (15)	0.102 (2)	−0.0157 (12)	0.0242 (15)	−0.0172 (14)
O3	0.118 (2)	0.0742 (18)	0.0644 (18)	−0.0119 (16)	−0.0183 (16)	−0.0234 (14)
N1	0.0433 (12)	0.0406 (13)	0.0305 (10)	0.0012 (9)	0.0101 (9)	0.0043 (9)
N2	0.0591 (16)	0.0448 (14)	0.0450 (13)	−0.0032 (11)	0.0118 (12)	−0.0030 (11)
N3	0.0482 (15)	0.0514 (18)	0.0671 (19)	−0.0020 (12)	0.0114 (14)	−0.0173 (14)
N4	0.0746 (18)	0.0677 (18)	0.0312 (12)	−0.0234 (14)	0.0108 (12)	−0.0012 (11)
C1	0.0330 (13)	0.0420 (15)	0.0291 (12)	0.0026 (10)	0.0090 (10)	0.0008 (10)
C2	0.0418 (15)	0.0458 (16)	0.0301 (13)	0.0004 (12)	0.0084 (11)	0.0034 (11)
C3	0.0516 (17)	0.0473 (17)	0.0360 (14)	0.0005 (13)	0.0053 (12)	0.0092 (12)
C4	0.0451 (16)	0.0401 (17)	0.0570 (18)	0.0025 (12)	0.0107 (14)	0.0084 (13)
C5	0.0321 (14)	0.0432 (16)	0.0491 (16)	0.0005 (11)	0.0103 (12)	−0.0106 (13)
C6	0.0326 (13)	0.0501 (16)	0.0328 (13)	0.0056 (11)	0.0068 (10)	−0.0021 (12)
C7	0.0388 (14)	0.0484 (17)	0.0274 (12)	0.0058 (12)	0.0063 (11)	0.0048 (11)
C8	0.0551 (18)	0.0446 (17)	0.0411 (15)	0.0043 (13)	0.0109 (13)	0.0113 (12)
C9	0.0621 (19)	0.0406 (16)	0.0507 (17)	0.0011 (13)	0.0195 (14)	0.0058 (13)
C10	0.121 (3)	0.073 (3)	0.060 (2)	0.021 (2)	0.023 (2)	−0.0222 (19)
C11	0.0426 (15)	0.0452 (16)	0.0315 (13)	−0.0097 (11)	0.0119 (11)	−0.0017 (11)

Geometric parameters (Å, °)

Cu1—O1	1.9240 (19)	C1—C2	1.441 (3)
Cu1—N1	1.940 (2)	C2—C3	1.413 (4)
Cu1—N4	1.947 (3)	C3—C4	1.361 (4)
Cu1—N2	2.033 (3)	C3—H3	0.9300
S1—C11	1.623 (3)	C4—C5	1.392 (4)
O1—C2	1.287 (3)	C4—H4	0.9300
O2—N3	1.231 (3)	C5—C6	1.366 (4)
O3—N3	1.217 (4)	C6—H6	0.9300
N1—C7	1.284 (3)	C7—H7	0.9300
N1—C8	1.476 (3)	C8—C9	1.505 (4)
N2—C10	1.468 (4)	C8—H8A	0.9700
N2—C9	1.484 (3)	C8—H8B	0.9700
N2—H2	0.906 (10)	C9—H9A	0.9700
N3—C5	1.444 (3)	C9—H9B	0.9700
N4—C11	1.149 (4)	C10—H10A	0.9600
C1—C6	1.392 (3)	C10—H10B	0.9600
C1—C7	1.435 (4)	C10—H10C	0.9600
O1—Cu1—N1	93.18 (8)	C3—C4—H4	120.5
O1—Cu1—N4	89.43 (9)	C5—C4—H4	120.5
N1—Cu1—N4	169.22 (11)	C6—C5—C4	121.3 (3)
O1—Cu1—N2	171.14 (9)	C6—C5—N3	119.3 (3)

N1—Cu1—N2	83.89 (9)	C4—C5—N3	119.4 (3)
N4—Cu1—N2	91.96 (10)	C5—C6—C1	120.7 (2)
C2—O1—Cu1	127.24 (16)	C5—C6—H6	119.6
C7—N1—C8	119.5 (2)	C1—C6—H6	119.6
C7—N1—Cu1	125.64 (18)	N1—C7—C1	125.3 (2)
C8—N1—Cu1	114.68 (17)	N1—C7—H7	117.4
C10—N2—C9	112.9 (3)	C1—C7—H7	117.4
C10—N2—Cu1	111.0 (2)	N1—C8—C9	107.9 (2)
C9—N2—Cu1	107.05 (17)	N1—C8—H8A	110.1
C10—N2—H2	104 (2)	C9—C8—H8A	110.1
C9—N2—H2	109 (2)	N1—C8—H8B	110.1
Cu1—N2—H2	113 (2)	C9—C8—H8B	110.1
O3—N3—O2	122.0 (3)	H8A—C8—H8B	108.4
O3—N3—C5	118.7 (3)	N2—C9—C8	109.2 (2)
O2—N3—C5	119.3 (3)	N2—C9—H9A	109.8
C11—N4—Cu1	173.4 (3)	C8—C9—H9A	109.8
C6—C1—C7	117.6 (2)	N2—C9—H9B	109.8
C6—C1—C2	119.4 (2)	C8—C9—H9B	109.8
C7—C1—C2	122.9 (2)	H9A—C9—H9B	108.3
O1—C2—C3	119.2 (2)	N2—C10—H10A	109.5
O1—C2—C1	124.1 (2)	N2—C10—H10B	109.5
C3—C2—C1	116.7 (2)	H10A—C10—H10B	109.5
C4—C3—C2	122.7 (2)	N2—C10—H10C	109.5
C4—C3—H3	118.7	H10A—C10—H10C	109.5
C2—C3—H3	118.7	H10B—C10—H10C	109.5
C3—C4—C5	118.9 (3)	N4—C11—S1	178.8 (3)

Fig. 1