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Di-μ-thiocyanato-κ^4N:N-bis({2-[2-(methylamino)ethyliminomethyl]-phenolato}copper(II))

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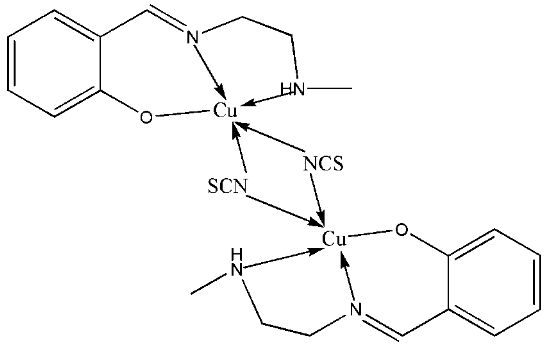
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.009 Å; R factor = 0.064; wR factor = 0.160; data-to-parameter ratio = 15.4.

The asymmetric unit of the title compound, $[Cu_2(NCS)_2(C_{10}H_{13}N_2O)_2]$, contains one-half of the centrosymmetric dinuclear complex, which exhibits a Cu$\cdots$Cu separation of 3.255 (2) Å. In the complex, each Cu^{II} ion is pentacoordinated in a square-pyramidal geometry. The basal plane is formed by one phenol O [Cu—O = 1.910 (4) Å], one imine N [Cu—N = 1.922 (5) Å] and one amine N [Cu—N = 2.041 (5) Å] atoms from the Schiff base ligand, and by the N atom [Cu—N = 1.940 (6) Å] from a thiocyanate ligand. The apical position is occupied by the N atom [Cu—N = 2.768 (6) Å] of another thiocyanate ligand. There are intramolecular N—H$\cdots$O hydrogen bonds linking the two ligands.

Related literature

For related structures, see: Diao *et al.* (2007); Diao & Li (2007); Wei (2005*a*,*b*); Wei & Wang (2006).



Experimental

Crystal data

$[Cu_2(NCS)_2(C_{10}H_{13}N_2O)_2]$
M_r = 597.69
Monoclinic, $P2_1/n$
a = 8.3305 (19) Å
b = 16.982 (4) Å
c = 9.137 (2) Å
β = 106.562 (3)°
V = 1239.0 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.92 mm^{-1}
T = 298 (2) K
0.20 × 0.07 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.700, T_{max} = 0.894
9340 measured reflections
2431 independent reflections
1510 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.160
S = 1.06
2431 reflections
158 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$\cdots$O1^i	0.90 (4)	2.17 (3)	2.993 (6)	153 (6)

Symmetry code: (i) $-x+2, -y, -z+1$.

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2307).

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supplementary materials

Acta Cryst. (2007). E**63**, m2629 [**doi:10.1107/S1600536807047022**]

Di-μ-thiocyanato-κ^4*N*:*N*-bis({2-[2-(methylamino)ethyliminomethyl]phenolato}copper(II))

Y.-J. Wei, F.-W. Wang and **Q.-Y. Zhu**

Comment

In continuation of our study of copper complexes with Shiff base ligands (Wei, 2005*a*,b; Wei & Wang, 2006), we report here the crystal structure of the title compound− a new dinuclear copper(II) complex (Fig. 1).

Each Cu atom in the complex is pentacoordinate in a square pyramidal geometry, with one phenolic O, one imine N, and one amine N atoms of one Schiff base ligand, and with one N atom of a thiocyanate ligand defining the basal plane, and with one N atom of another thiocyanate ligand occupying the apical position. The thiocyanate ligands adopt end-on coordination mode, with the Cu···Cu distance of 3.255 (2) Å. The coordinate bond lengths and angles are comparable with those observed in other Schiff base copper(II) complexes (Diao, Shu *et al.*, 2007; Diao & Li, 2007).

Experimental

Salicyaldehyde (1.0 mmol, 122.1 mg), *N*-methylethane-1,2-diamine (1.0 mmol, 74.0 mg), NH_4NCS (1.0 mmol, 7.6 mg) and $Cu(CH_3COO)_2\cdot H_2O$ (1.0 mmol, 199.1 mg) were dissolved in a methanol solution (150 ml). The mixture was refluxed at 340 K for about 1 h to give a clear blue solution. After keeping the cooled resulting solution in dark for five days, blue needle-shaped crystals were formed.

Refinement

Atom H2 attached to N2 was located from a difference Fourier map and refined isotropically, with N–H distance restrained to 0.90 (3) Å. Other H atoms were placed geometrically, with C–H distances in the range 0.93–0.97 Å, and with U_{iso}(H) set to $1.2U_{eq}$(C) and $1.5U_{eq}$(methyl C).

Figures

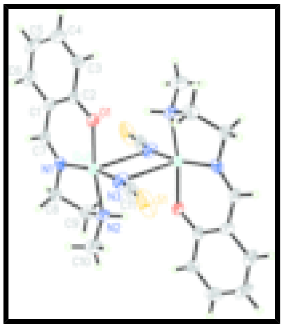

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids at the 30% probability level. Unlabelled atoms are related with labelled ones by symmetry code ($-x + 2$, $-y$, $-z + 1$).

Di-μ-thiocyanato-κ^4*N*:*N*-bis({2-[2- (methylamino)ethyliminomethyl]phenolato}copper(II))

Crystal data

$[Cu_2(NCS)_2(C_{10}H_{13}N_2O)_2]$

$M_r = 597.69$

Monoclinic, $P2_1/n$

$a = 8.3305$ (19) Å

$b = 16.982$ (4) Å

$c = 9.137$ (2) Å

$\beta = 106.562$ (3)°

$V = 1239.0$ (5) Å^3

$Z = 2$

$F_{000} = 612$

$D_x = 1.602$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 944 reflections

$\theta = 2.3$–24.9°

$\mu = 1.92$ mm^{-1}

$T = 298$ (2) K

Needle, blue

0.20 × 0.07 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2)$ K

ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.700$, $T_{\max} = 0.894$

9340 measured reflections

2431 independent reflections

1510 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.087$

$\theta_{\max} = 26.0$°

$\theta_{\min} = 2.4$°

$h = -10 \rightarrow 10$

$k = -20 \rightarrow 20$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.160$

$S = 1.06$

2431 reflections

158 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.4135P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.45$ e Å^{-3}

$\Delta\rho_{\min} = -0.67$ e Å^{-3}

Extinction correction: none

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	0.96892 (9)	0.04411 (4)	0.33613 (8)	0.0410 (3)
S1	1.4907 (3)	0.09746 (15)	0.6792 (3)	0.1066 (10)
O1	1.0498 (5)	−0.0513 (2)	0.2719 (4)	0.0443 (10)
N1	0.7859 (6)	0.0530 (3)	0.1529 (5)	0.0398 (12)
N2	0.8447 (7)	0.1282 (3)	0.4207 (5)	0.0463 (13)
N3	1.1753 (7)	0.0566 (3)	0.4992 (6)	0.0558 (15)
C1	0.8475 (8)	−0.0616 (3)	0.0253 (7)	0.0412 (14)
C2	0.9862 (7)	−0.0881 (3)	0.1430 (6)	0.0372 (14)
C3	1.0614 (9)	−0.1608 (4)	0.1219 (8)	0.0530 (17)
H3	1.1495	−0.1808	0.2000	0.064*
C4	1.0052 (10)	−0.2015 (4)	−0.0123 (9)	0.066 (2)
H4	1.0556	−0.2491	−0.0237	0.079*
C5	0.8739 (10)	−0.1731 (5)	−0.1324 (9)	0.070 (2)
H5	0.8393	−0.2008	−0.2238	0.084*
C6	0.7964 (9)	−0.1042 (4)	−0.1144 (7)	0.0562 (18)
H6	0.7093	−0.0851	−0.1944	0.067*
C7	0.7571 (7)	0.0073 (3)	0.0369 (6)	0.0406 (14)
H7	0.6687	0.0207	−0.0475	0.049*
C8	0.6778 (8)	0.1198 (4)	0.1574 (7)	0.0503 (16)
H8A	0.5665	0.1106	0.0896	0.060*
H8B	0.7224	0.1672	0.1243	0.060*
C9	0.6698 (8)	0.1295 (4)	0.3187 (7)	0.0554 (17)
H9A	0.6163	0.1791	0.3295	0.066*
H9B	0.6054	0.0871	0.3451	0.066*
C10	0.9218 (9)	0.2060 (4)	0.4491 (8)	0.072 (2)
H10A	0.9340	0.2272	0.3554	0.108*
H10B	1.0299	0.2018	0.5224	0.108*
H10C	0.8521	0.2403	0.4881	0.108*
C11	1.3052 (9)	0.0744 (4)	0.5740 (7)	0.0510 (17)
H2	0.841 (9)	0.110 (4)	0.512 (4)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0387 (4)	0.0473 (5)	0.0355 (4)	0.0051 (4)	0.0082 (3)	−0.0031 (4)
S1	0.0748 (16)	0.1044 (19)	0.1027 (17)	−0.0400 (14)	−0.0359 (13)	0.0478 (15)
O1	0.050 (3)	0.049 (3)	0.033 (2)	0.011 (2)	0.0107 (19)	−0.0028 (19)
N1	0.034 (3)	0.045 (3)	0.041 (3)	0.008 (2)	0.011 (2)	0.010 (2)
N2	0.054 (3)	0.044 (3)	0.042 (3)	0.003 (3)	0.017 (3)	−0.010 (2)
N3	0.047 (3)	0.072 (4)	0.047 (3)	0.007 (3)	0.011 (3)	−0.007 (3)
C1	0.040 (4)	0.044 (4)	0.046 (4)	−0.014 (3)	0.021 (3)	−0.006 (3)
C2	0.041 (4)	0.045 (4)	0.032 (3)	−0.004 (3)	0.020 (3)	0.006 (3)
C3	0.069 (5)	0.042 (4)	0.057 (4)	0.006 (3)	0.034 (4)	0.003 (3)
C4	0.085 (6)	0.049 (4)	0.078 (5)	−0.008 (4)	0.048 (5)	−0.013 (4)
C5	0.087 (6)	0.076 (5)	0.064 (5)	−0.036 (5)	0.050 (5)	−0.037 (4)
C6	0.059 (4)	0.077 (5)	0.039 (4)	−0.017 (4)	0.024 (3)	−0.009 (4)
C7	0.042 (4)	0.050 (4)	0.026 (3)	−0.003 (3)	0.004 (3)	0.002 (3)
C8	0.048 (4)	0.056 (4)	0.044 (4)	0.010 (3)	0.009 (3)	0.006 (3)
C9	0.042 (4)	0.059 (4)	0.067 (5)	0.011 (3)	0.018 (3)	−0.003 (4)
C10	0.078 (6)	0.058 (5)	0.084 (5)	0.001 (4)	0.032 (4)	−0.016 (4)
C11	0.050 (4)	0.056 (4)	0.041 (4)	−0.001 (3)	0.005 (3)	0.008 (3)

Geometric parameters (Å, °)

Cu1—O1	1.910 (4)	C3—C4	1.369 (9)
Cu1—N1	1.922 (5)	C3—H3	0.9300
Cu1—N3	1.940 (6)	C4—C5	1.396 (10)
Cu1—N2	2.041 (5)	C4—H4	0.9300
S1—C11	1.617 (7)	C5—C6	1.368 (10)
O1—C2	1.305 (6)	C5—H5	0.9300
N1—C7	1.280 (7)	C6—H6	0.9300
N1—C8	1.457 (7)	C7—H7	0.9300
N2—C10	1.459 (8)	C8—C9	1.503 (8)
N2—C9	1.490 (8)	C8—H8A	0.9700
N2—H2	0.90 (4)	C8—H8B	0.9700
N3—C11	1.144 (8)	C9—H9A	0.9700
C1—C2	1.410 (8)	C9—H9B	0.9700
C1—C7	1.411 (8)	C10—H10A	0.9600
C1—C6	1.423 (8)	C10—H10B	0.9600
C2—C3	1.422 (8)	C10—H10C	0.9600

O1—Cu1—N1	93.47 (18)	C6—C5—C4	119.5 (6)
O1—Cu1—N3	90.91 (19)	C6—C5—H5	120.3
N1—Cu1—N3	165.9 (2)	C4—C5—H5	120.3
O1—Cu1—N2	166.3 (2)	C5—C6—C1	120.8 (7)
N1—Cu1—N2	85.0 (2)	C5—C6—H6	119.6
N3—Cu1—N2	93.8 (2)	C1—C6—H6	119.6
C2—O1—Cu1	126.6 (4)	N1—C7—C1	125.9 (5)
C7—N1—C8	121.6 (5)	N1—C7—H7	117.0

C7—N1—Cu1	126.0 (4)	C1—C7—H7	117.0
C8—N1—Cu1	112.3 (4)	N1—C8—C9	108.3 (5)
C10—N2—C9	113.6 (5)	N1—C8—H8A	110.0
C10—N2—Cu1	117.1 (4)	C9—C8—H8A	110.0
C9—N2—Cu1	106.5 (4)	N1—C8—H8B	110.0
C10—N2—H2	106 (5)	C9—C8—H8B	110.0
C9—N2—H2	107 (5)	H8A—C8—H8B	108.4
Cu1—N2—H2	105 (5)	N2—C9—C8	107.7 (5)
C11—N3—Cu1	165.6 (6)	N2—C9—H9A	110.2
C2—C1—C7	123.1 (5)	C8—C9—H9A	110.2
C2—C1—C6	119.5 (6)	N2—C9—H9B	110.2
C7—C1—C6	117.4 (6)	C8—C9—H9B	110.2
O1—C2—C1	124.7 (5)	H9A—C9—H9B	108.5
O1—C2—C3	117.2 (5)	N2—C10—H10A	109.5
C1—C2—C3	118.1 (5)	N2—C10—H10B	109.5
C4—C3—C2	120.6 (6)	H10A—C10—H10B	109.5
C4—C3—H3	119.7	N2—C10—H10C	109.5
C2—C3—H3	119.7	H10A—C10—H10C	109.5
C3—C4—C5	121.4 (7)	H10B—C10—H10C	109.5
C3—C4—H4	119.3	N3—C11—S1	178.6 (7)
C5—C4—H4	119.3		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.90 (4)	2.17 (3)	2.993 (6)	153 (6)

Symmetry codes: (i) −*x*+2, −*y*, −*z*+1.

Fig. 1