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Acetonitrile{2-[3-(dimethylamino)-propyliminomethyl]-4-nitrophenolato-κ^3N,N',O}(thiocyanato-κN)copper(II)

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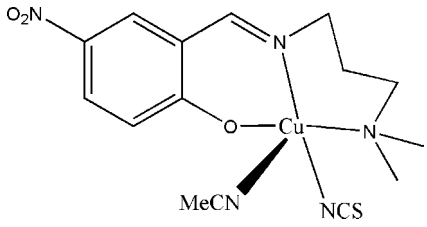
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.037; wR factor = 0.098; data-to-parameter ratio = 18.3.

In the title mononuclear copper(II) complex, [Cu($C_{12}H_{16}N_3O_3$)(NCS)(C_2H_3N)], the Cu^{II} atom is five-coordinated in a square-pyramidal geometry, with one O and two N atoms of the Schiff-base ligand and one N atom of the thiocyanate ligand defining the basal plane. The apical position is occupied by the N atom of the acetonitrile molecule. The structure is stabilized by intramolecular C—H···N and intermolecular C—H···O hydrogen-bonding interactions.

Related literature

For related literature, see: Ye & You (2007); Hu *et al.* (2005).



Experimental

Crystal data

[Cu($C_{12}H_{16}N_3O_3$)(NCS)(C_2H_3N)]
M_r = 412.95
Monoclinic, $P2_1/n$
a = 12.507 (2) Å
b = 11.552 (1) Å
c = 12.787 (2) Å
β = 93.543 (1)°
V = 1844.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.32 mm^{-1}
T = 298 (2) K
0.23 × 0.21 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.745, T_{max} = 0.770
15401 measured reflections
4186 independent reflections
3143 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.098
S = 1.02
4186 reflections
229 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.950 (2)	Cu1—N2	2.093 (2)
Cu1—N4	1.970 (2)	Cu1—N3	2.299 (2)
Cu1—N1	1.981 (2)		
O1—Cu1—N4	84.49 (9)	N1—Cu1—N2	93.04 (7)
O1—Cu1—N1	89.22 (7)	O1—Cu1—N3	98.37 (8)
N4—Cu1—N1	162.26 (9)	N4—Cu1—N3	101.30 (10)
O1—Cu1—N2	168.09 (8)	N1—Cu1—N3	96.01 (8)
N4—Cu1—N2	89.91 (9)	N2—Cu1—N3	93.03 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12B···N4	0.96	2.43	2.969 (3)	115
C10—H10B···O3^{i}	0.97	2.48	3.082 (3)	120

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2144).

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supplementary materials

Acta Cryst. (2007). E**63**, m1837 [**doi:10.1107/S1600536807025342**]

Acetonitrile{2-[3-(dimethylamino)propyliminomethyl]-4-nitrophenolato-κ^3N,N',O}(thiocyanato-κN)copper(II)

L.-J. Ye and Z. . You

Comment

Recently, we have reported a thiocyanate coordinated zinc(II) complex (Ye & You, 2007). As an extension of the work on the crystal structures of such complexes, we report herein the crystal structure of the title compound, (I).

The Cu^{II} atom in (I) is five-coordinated in a square-pyramidal geometry, with one O and two N atoms of the Schiff base ligand and one N atom of the thiocyanate ligand defining the basal plane, and the N atom of the acetonitrile group occupying the apical position (Fig. 1). Selected bond distances and angles within the coordination sphere of the metal are given in Table 1. The molecular and crystal structures are stabilized by C—H···N and C—H···O hydrogen bonds (Table 2).

Experimental

5-Nitrosalicylaldehyde (0.1 mmol, 16.5 mg), *N*,*N*-dimethylpropane-1,3-diamine (0.1 mmol, 10.2 mg), ammonium thiocyanate (0.1 mmol, 7.6 mg), and copper acetate monohydrate (0.1 mmol, 20.0 mg) were dissolved in an acetonitrile solution (10 ml). The mixture was stirred at room temperature for 10 min, giving a clear blue solution. Crystals of the title compound were formed by slow evaporation of the solvent over a week at room temperature.

Refinement

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) = 1.2 or 1.5U_{eq}(C).

Figures

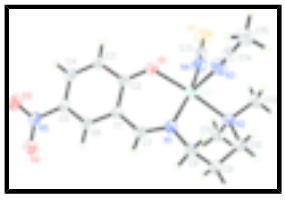


Fig. 1. The molecular structure of the title compound, shown with 30% probability displacement ellipsoids.

Acetonitrile{2-[3-(dimethylamino)propyliminomethyl]-4-nitrophenolato- κ^3N,N',O}(thiocyanato-κN)copper(II)

Crystal data

$[Cu(C_{12}H_{16}N_3O_3)(NCS)(C_2H_3N)]$	F_{000} = 852
M_r = 412.95	D_x = 1.487 Mg m^{-3}

Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 3875 reflections
$a = 12.507$ (2) Å	$\theta = 2.3$–24.5°
$b = 11.552$ (1) Å	$\mu = 1.32$ mm^{-1}
$c = 12.787$ (2) Å	$T = 298$ (2) K
$\beta = 93.543$ (1)°	Block, blue
$V = 1844.0$ (4) Å^3	0.23 × 0.21 × 0.20 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	4186 independent reflections
Radiation source: fine-focus sealed tube	3143 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.035$
$T = 298$(2) K	$\theta_{max} = 27.5^\circ$
ω scans	$\theta_{min} = 2.2^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -15 \rightarrow 16$
$T_{min} = 0.745$, $T_{max} = 0.770$	$k = -14 \rightarrow 14$
15401 measured reflections	$l = -16 \rightarrow 16$

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-atom parameters constrained
$wR(F^2) = 0.098$	$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.3686P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
4186 reflections	$\Delta\rho_{max} = 0.31$ e Å^{-3}
229 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	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	0.21596 (2)	0.93882 (2)	0.25457 (2)	0.04336 (11)
S1	0.26057 (7)	0.83713 (8)	−0.09663 (6)	0.0811 (3)
O1	0.36548 (13)	0.89111 (16)	0.27255 (15)	0.0596 (5)
O2	0.68408 (15)	1.2638 (2)	0.4508 (2)	0.0856 (7)
O3	0.79311 (14)	1.1489 (2)	0.38151 (18)	0.0819 (7)
N1	0.23620 (14)	1.03387 (16)	0.38324 (14)	0.0414 (4)
N2	0.06710 (15)	1.01357 (17)	0.21204 (15)	0.0452 (5)
N3	0.14008 (19)	0.7828 (2)	0.3335 (2)	0.0679 (6)
N4	0.22514 (18)	0.8813 (2)	0.11039 (18)	0.0656 (6)
N5	0.70235 (15)	1.1749 (2)	0.40349 (17)	0.0544 (5)
C1	0.42878 (17)	1.05305 (18)	0.37335 (17)	0.0387 (5)
C2	0.44268 (19)	0.95528 (19)	0.30748 (19)	0.0448 (5)
C3	0.5498 (2)	0.9310 (2)	0.2819 (2)	0.0561 (7)
H3	0.5630	0.8650	0.2431	0.067*
C4	0.63293 (19)	1.0008 (2)	0.31240 (19)	0.0537 (6)
H4	0.7016	0.9838	0.2929	0.064*
C5	0.61515 (17)	1.0982 (2)	0.37302 (18)	0.0437 (5)
C6	0.51490 (17)	1.1218 (2)	0.40523 (17)	0.0414 (5)
H6	0.5049	1.1847	0.4490	0.050*
C7	0.32677 (18)	1.07729 (19)	0.41476 (18)	0.0408 (5)
H7	0.3266	1.1297	0.4699	0.049*
C8	0.14448 (19)	1.0635 (2)	0.44447 (19)	0.0508 (6)
H8A	0.1690	1.1113	0.5035	0.061*
H8B	0.1146	0.9930	0.4719	0.061*
C9	0.05886 (19)	1.1266 (2)	0.3807 (2)	0.0532 (6)
H9A	0.0908	1.1918	0.3463	0.064*
H9B	0.0072	1.1570	0.4271	0.064*
C10	0.00110 (19)	1.0514 (2)	0.2987 (2)	0.0545 (6)
H10A	−0.0255	0.9831	0.3328	0.065*
H10B	−0.0604	1.0937	0.2687	0.065*
C11	0.0905 (2)	1.1145 (2)	0.1461 (2)	0.0619 (7)
H11A	0.0247	1.1522	0.1234	0.093*
H11B	0.1265	1.0890	0.0860	0.093*
H11C	0.1356	1.1678	0.1860	0.093*
C12	−0.0019 (2)	0.9324 (2)	0.1481 (2)	0.0624 (7)
H12A	−0.0136	0.8636	0.1879	0.094*
H12B	0.0328	0.9124	0.0856	0.094*
H12C	−0.0694	0.9687	0.1294	0.094*
C13	0.24112 (19)	0.8638 (2)	0.0250 (2)	0.0520 (6)
C14	0.1136 (2)	0.7100 (2)	0.3835 (2)	0.0528 (6)
C15	0.0798 (2)	0.6164 (3)	0.4485 (2)	0.0713 (8)
H15A	0.1241	0.6145	0.5125	0.107*
H15B	0.0862	0.5444	0.4120	0.107*
H15C	0.0065	0.6281	0.4641	0.107*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.04219 (18)	0.04213 (18)	0.04608 (18)	−0.00245 (12)	0.00523 (12)	−0.00212 (12)
S1	0.1006 (6)	0.0958 (6)	0.0481 (4)	0.0151 (5)	0.0143 (4)	0.0046 (4)
O1	0.0443 (10)	0.0475 (10)	0.0868 (13)	0.0035 (8)	0.0027 (9)	−0.0201 (9)
O2	0.0455 (11)	0.0814 (15)	0.128 (2)	−0.0016 (10)	−0.0067 (11)	−0.0378 (14)
O3	0.0350 (10)	0.1144 (18)	0.0968 (16)	0.0000 (11)	0.0085 (10)	−0.0257 (14)
N1	0.0392 (10)	0.0449 (11)	0.0406 (10)	0.0004 (8)	0.0073 (8)	0.0032 (8)
N2	0.0432 (11)	0.0459 (11)	0.0465 (11)	−0.0023 (9)	0.0029 (9)	0.0004 (9)
N3	0.0707 (15)	0.0561 (14)	0.0764 (16)	−0.0128 (12)	0.0011 (12)	0.0113 (13)
N4	0.0653 (15)	0.0780 (16)	0.0540 (14)	0.0053 (13)	0.0064 (11)	−0.0135 (12)
N5	0.0352 (11)	0.0694 (15)	0.0578 (13)	0.0048 (10)	−0.0037 (9)	−0.0001 (11)
C1	0.0388 (12)	0.0389 (12)	0.0383 (11)	0.0053 (9)	0.0010 (9)	0.0066 (9)
C2	0.0447 (13)	0.0403 (13)	0.0493 (13)	0.0073 (10)	0.0010 (10)	0.0019 (10)
C3	0.0479 (14)	0.0573 (16)	0.0632 (17)	0.0129 (12)	0.0046 (12)	−0.0134 (13)
C4	0.0382 (13)	0.0689 (17)	0.0543 (15)	0.0120 (12)	0.0053 (11)	0.0003 (13)
C5	0.0350 (12)	0.0511 (13)	0.0446 (13)	0.0048 (10)	−0.0016 (10)	0.0060 (11)
C6	0.0385 (12)	0.0442 (13)	0.0410 (12)	0.0080 (10)	−0.0009 (9)	0.0018 (10)
C7	0.0413 (12)	0.0425 (13)	0.0386 (12)	0.0038 (10)	0.0026 (9)	0.0014 (9)
C8	0.0416 (13)	0.0644 (16)	0.0475 (13)	−0.0065 (12)	0.0121 (11)	−0.0042 (12)
C9	0.0407 (13)	0.0600 (16)	0.0603 (15)	0.0010 (12)	0.0144 (11)	−0.0095 (13)
C10	0.0370 (12)	0.0663 (17)	0.0607 (16)	−0.0002 (11)	0.0069 (11)	−0.0048 (13)
C11	0.0709 (18)	0.0526 (16)	0.0627 (17)	0.0043 (14)	0.0085 (14)	0.0101 (13)
C12	0.0526 (15)	0.0692 (18)	0.0637 (17)	−0.0074 (13)	−0.0101 (13)	−0.0108 (14)
C13	0.0489 (14)	0.0508 (15)	0.0563 (16)	0.0029 (11)	0.0036 (12)	0.0008 (12)
C14	0.0508 (14)	0.0462 (14)	0.0613 (16)	−0.0044 (12)	0.0015 (12)	−0.0010 (13)
C15	0.079 (2)	0.0554 (17)	0.080 (2)	−0.0118 (16)	0.0143 (16)	0.0121 (15)

Geometric parameters (Å, °)

Cu1—O1	1.950 (2)	C4—C5	1.391 (4)
Cu1—N4	1.970 (2)	C4—H4	0.9300
Cu1—N1	1.981 (2)	C5—C6	1.371 (3)
Cu1—N2	2.093 (2)	C6—H6	0.9300
Cu1—N3	2.299 (2)	C7—H7	0.9300
S1—C13	1.618 (3)	C8—C9	1.495 (3)
O1—C2	1.276 (3)	C8—H8A	0.9700
O2—N5	1.220 (3)	C8—H8B	0.9700
O3—N5	1.224 (3)	C9—C10	1.511 (3)
N1—C7	1.281 (3)	C9—H9A	0.9700
N1—C8	1.469 (3)	C9—H9B	0.9700
N2—C11	1.479 (3)	C10—H10A	0.9700
N2—C12	1.485 (3)	C10—H10B	0.9700
N2—C10	1.487 (3)	C11—H11A	0.9600
N3—C14	1.119 (3)	C11—H11B	0.9600
N4—C13	1.140 (3)	C11—H11C	0.9600
N5—C5	1.441 (3)	C12—H12A	0.9600

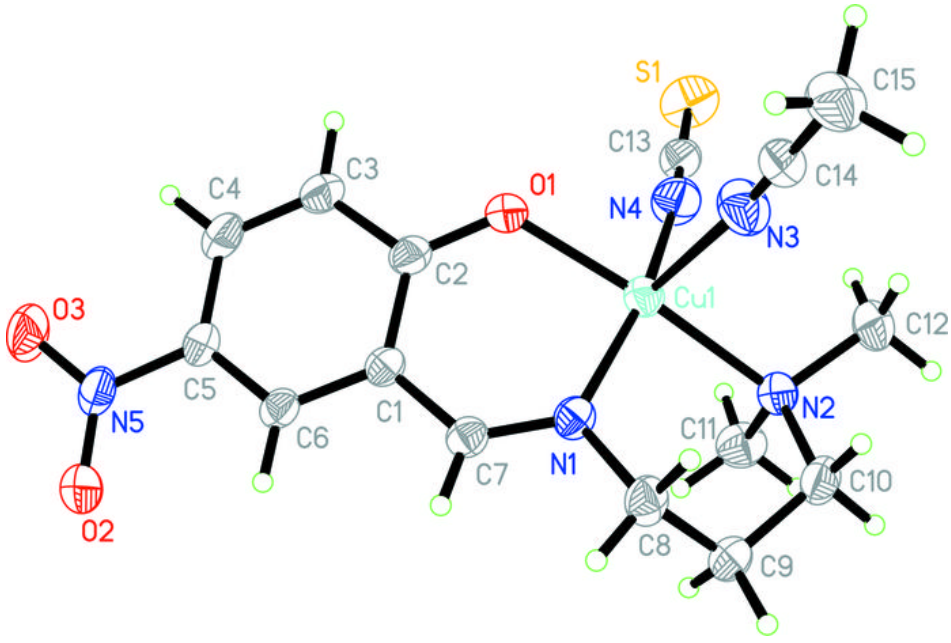
C1—C6	1.379 (3)	C12—H12B	0.9600
C1—C2	1.426 (3)	C12—H12C	0.9600
C1—C7	1.439 (3)	C14—C15	1.443 (4)
C2—C3	1.427 (3)	C15—H15A	0.9600
C3—C4	1.354 (4)	C15—H15B	0.9600
C3—H3	0.9300	C15—H15C	0.9600
O1—Cu1—N4	84.49 (9)	C1—C6—H6	119.7
O1—Cu1—N1	89.22 (7)	N1—C7—C1	126.5 (2)
N4—Cu1—N1	162.26 (9)	N1—C7—H7	116.7
O1—Cu1—N2	168.09 (8)	C1—C7—H7	116.7
N4—Cu1—N2	89.91 (9)	N1—C8—C9	112.3 (2)
N1—Cu1—N2	93.04 (7)	N1—C8—H8A	109.1
O1—Cu1—N3	98.37 (8)	C9—C8—H8A	109.1
N4—Cu1—N3	101.30 (10)	N1—C8—H8B	109.1
N1—Cu1—N3	96.01 (8)	C9—C8—H8B	109.1
N2—Cu1—N3	93.03 (8)	H8A—C8—H8B	107.9
C2—O1—Cu1	125.21 (15)	C8—C9—C10	113.2 (2)
C7—N1—C8	116.47 (19)	C8—C9—H9A	108.9
C7—N1—Cu1	122.92 (15)	C10—C9—H9A	108.9
C8—N1—Cu1	120.52 (15)	C8—C9—H9B	108.9
C11—N2—C12	108.2 (2)	C10—C9—H9B	108.9
C11—N2—C10	109.4 (2)	H9A—C9—H9B	107.8
C12—N2—C10	105.38 (19)	N2—C10—C9	115.0 (2)
C11—N2—Cu1	105.60 (15)	N2—C10—H10A	108.5
C12—N2—Cu1	111.08 (16)	C9—C10—H10A	108.5
C10—N2—Cu1	116.96 (15)	N2—C10—H10B	108.5
C14—N3—Cu1	170.0 (2)	C9—C10—H10B	108.5
C13—N4—Cu1	168.5 (3)	H10A—C10—H10B	107.5
O2—N5—O3	121.7 (2)	N2—C11—H11A	109.5
O2—N5—C5	119.4 (2)	N2—C11—H11B	109.5
O3—N5—C5	118.9 (2)	H11A—C11—H11B	109.5
C6—C1—C2	120.6 (2)	N2—C11—H11C	109.5
C6—C1—C7	118.2 (2)	H11A—C11—H11C	109.5
C2—C1—C7	121.0 (2)	H11B—C11—H11C	109.5
O1—C2—C1	123.3 (2)	N2—C12—H12A	109.5
O1—C2—C3	120.4 (2)	N2—C12—H12B	109.5
C1—C2—C3	116.2 (2)	H12A—C12—H12B	109.5
C4—C3—C2	122.1 (2)	N2—C12—H12C	109.5
C4—C3—H3	118.9	H12A—C12—H12C	109.5
C2—C3—H3	118.9	H12B—C12—H12C	109.5
C3—C4—C5	119.7 (2)	N4—C13—S1	178.4 (3)
C3—C4—H4	120.1	N3—C14—C15	179.7 (3)
C5—C4—H4	120.1	C14—C15—H15A	109.5
C6—C5—C4	120.6 (2)	C14—C15—H15B	109.5
C6—C5—N5	119.2 (2)	H15A—C15—H15B	109.5
C4—C5—N5	120.2 (2)	C14—C15—H15C	109.5
C5—C6—C1	120.6 (2)	H15A—C15—H15C	109.5
C5—C6—H6	119.7	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C12—H12B···N4	0.96	2.43	2.969 (3)	115
C10—H10B···O3^{i}	0.97	2.48	3.082 (3)	120

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1


S1
C15
C13
C14
C4
C3
O1
N4
N3
C2
Cu1
C12
O3
C5
C1
N2
N5
C6
C11
N1
C7
C10
C8
O2
C9