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(Azido-κN){(*E*)-2-[1-(pyridin-2-yl)ethylideneamino]phenolato-$\kappa^3 N,N',O$}-copper(II)

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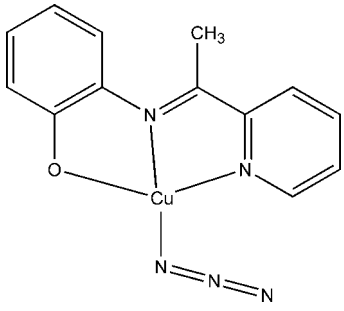
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.092; data-to-parameter ratio = 13.0.

In the title complex, $[Cu(C_{13}H_{11}N_2O)(N_3)]$, the Cu^{II} cation is four-coordinated by an N_2O donor set of the tridentate Schiff base ligand and by the terminal N atom of the azide anion, forming a slightly distorted square-planar configuration.

Related literature

For related structures, see: Talukder *et al.* (2004); Sun (2008); Wang *et al.* (2012); Yu (2012). For the synthesis, see: Shita *et al.* (2009).



Experimental

Crystal data

$[Cu(C_{13}H_{11}N_2O)(N_3)]$
M_r = 316.81
Monoclinic, $P2_1/c$
a = 6.5881 (3) Å
b = 10.1576 (3) Å
c = 18.3884 (7) Å
β = 92.810 (3)°
V = 1229.06 (8) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 2.54 mm^{-1}
T = 120 K
0.57 × 0.31 × 0.04 mm

Data collection

Agilent Xcalibur Gemini ultra diffractometer with Eos detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
T_{min} = 0.709, T_{max} = 1.000
4723 measured reflections
2357 independent reflections
2180 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.092
S = 1.05
2357 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Data collection: *CrysAlis PRO* (Agilent, 2011); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 2012) and *WinGX32* (Farrugia, 2012); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LR2111).

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supporting information

Acta Cryst. (2013). E**69**, m468 [doi:10.1107/S1600536813019570]

(Azido-κN){(*E*)-2-[1-(pyridin-2-yl)ethylideneamino]phenolato-$\kappa^3 N,N',O$}copper(II)

Amitabha Datta, Jack K. Clegg, Jui-Hsien Huang and Shiann-Cherng Sheu

S1. Comment

In the title complex, $[Cu(C_{13}H_{11}N_2O)(N_3)]$, the Cu^{II} ion exhibits a slightly distorted square-planar coordination environment defined by the deprotonated tridentate Schiff base ligand that coordinates *via* the phenolate O, imine N and pyridyl N atoms and the N atom of azide ion (Fig. 1). The bond angles around Cu^{II} ion are slightly distorted from those of regular square-planar and range from 81.53 (7) to 175.89 (7)°. The $[CuN_3O]$ square plane and the aryl and pyridyl rings in the Schiff base are almost coplanar. The dihedral angles among these three planes are 3.55 (6)°, 4.70 (5)° and 4.99 (7)°. The bond distances to the central copper [Cu—N_{py} = 1.9775 (16) Å, Cu—N_{imine} = 1.9682 (16) Å, Cu—N_{azide} = 1.9470 (18) Å, Cu—$O_{phenolic}$ = 1.9133 (14) Å] (Table 1) are similar to those in complexes $[Cu(C_{14}H_{13}N_2O_2)(N_3)]$ and $[Cu(C_{13}H_{10}ClN_2O)Cl]$ (Sun, 2008; Wang *et al.*, 2012). The bond distances and bond angles in azide ion bound to Cu^{II} ion are similar to those in complexes $[Cu(C_{14}H_{13}N_2O_2)(N_3)]$ and $[Cu(C_{16}H_{23}N_2O)(N_3)]$ (Sun, 2008; Talukder *et al.*, 2004). The N3—N4 bond [1.201 (3) Å] in the complex is longer than the N4—N5 bond [1.157 (3) Å]. The NNN moiety is nearly linear and shows a bent coordination mode with the Cu^{II} ion [(N3—N4—N5/Cu1—N3—N4 = 177.4 (2)/117.06 (15)°].

S2. Experimental

The tridentate Schiff base ligand was prepared according to literature procedure (Shita *et al.*, 2009). To a hot methanolic solution (20 ml) of $Cu(CCl_3COO)_2.6H_2O$ (0.484 g, 1.0 mmol), the ligand (1.0 mmol) was added, which produced immediately an intensely green solution. The solution was then heated to boiling and then, an aqueous solution (10 ml) of sodium azide (0.065 g, 1 mmol) was added dropwise slowly over 15 min in hot condition. After the completion of addition of sodium azide, the resulting solution was kept under boiling for another 10 min. On cooling and after slow evaporation of the solution, the dark-green plate-shaped single crystals of the complex were separated out in 3 d. The crystals were filtered off and washed with water and dried in air.

S3. Refinement

H atoms were placed at calculated positions (C—H = 0.95–0.98 Å) and were included in the refinement in the riding-model approximation, with *U*iso(H) = 1.2*U*eq(C) and 1.5 *U*eq(C)

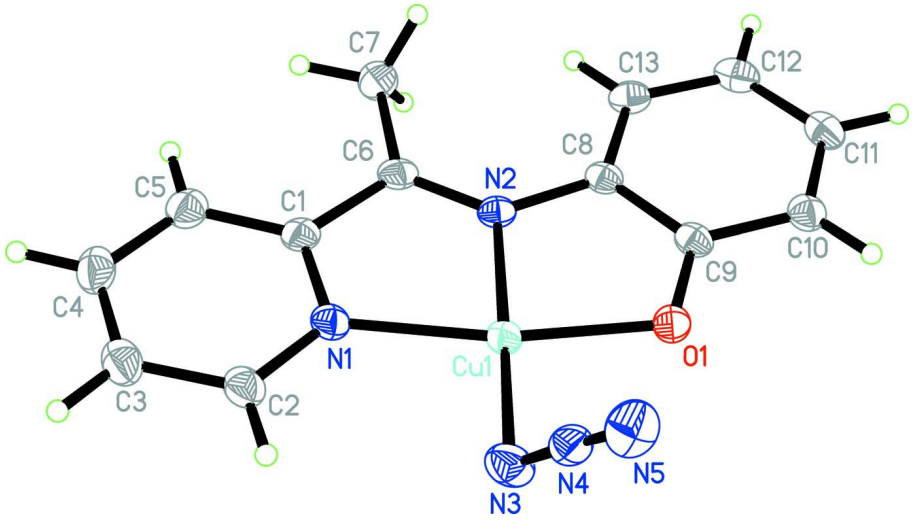

Figure 1

The molecular structure of the title complex, showing displacement ellipsoids at the 50% probability level.

(Azido-κN){(*E*)-2-[1-(pyridin-2-yl)ethylideneamino]phenolato-$\kappa^3 N,N',O$}copper(II)

Crystal data

$[Cu(C_{13}H_{11}CuN_2O)(N_3)]$
$M_r = 316.81$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.5881\ (3)$ Å
$b = 10.1576\ (3)$ Å
$c = 18.3884\ (7)$ Å
$\beta = 92.810\ (3)°$
$V = 1229.06\ (8)$ Å^3
$Z = 4$
$F(000) = 644$
$D_x = 1.712$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54184$ Å
Cell parameters from 2697 reflections
$\theta = 4.4–71.9°$
$\mu = 2.54$ mm^{-1}
$T = 120$ K
Plate, green
0.57 × 0.31 × 0.04 mm

Data collection

Agilent Xcalibur Gemini ultra diffractometer with Eos detector
Radiation source: Enhance Ultra (Cu) X-ray Source
Mirror monochromator
Detector resolution: 16.1183 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2011)
$T_{min} = 0.709$, $T_{max} = 1.000$
4723 measured reflections
2357 independent reflections
2180 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 72.0°$, $\theta_{min} = 4.8°$
$h = -8\rightarrow6$
$k = -11\rightarrow12$
$l = -21\rightarrow22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.092$
$S = 1.05$
2357 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.5226P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}

Special details

Experimental. CrysAlisPro, Agilent Technologies, Version 1.171.35.21 (release 20-01-2012 CrysAlis171 .NET) (compiled Jan 23 2012,18:06:46). Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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}
C11	0.1572 (3)	0.7721 (2)	0.10880 (11)	0.0224 (4)
H11	0.1311	0.8364	0.1447	0.027*
C12	0.1511 (3)	0.80806 (19)	0.03531 (13)	0.0238 (4)
H12	0.1217	0.8965	0.0216	0.029*
C13	0.1878 (3)	0.7152 (2)	−0.01764 (11)	0.0214 (4)
H13	0.1842	0.7401	−0.0675	0.026*
N3	0.3338 (3)	0.14937 (18)	0.06852 (10)	0.0267 (4)
N4	0.1971 (3)	0.12200 (16)	0.10667 (9)	0.0225 (4)
N5	0.0684 (3)	0.09080 (19)	0.14363 (10)	0.0300 (4)
C1	0.3018 (3)	0.3279 (2)	−0.13806 (11)	0.0190 (4)
C2	0.3721 (3)	0.1131 (2)	−0.10036 (11)	0.0233 (4)
H2	0.3975	0.0518	−0.0620	0.028*
C3	0.3734 (3)	0.0698 (2)	−0.17231 (12)	0.0270 (4)
H3	0.3992	−0.0201	−0.1829	0.032*
C4	0.3368 (3)	0.1587 (2)	−0.22775 (12)	0.0281 (5)
H4	0.3346	0.1309	−0.2771	0.034*
C5	0.3030 (3)	0.2905 (2)	−0.21045 (11)	0.0250 (4)
H5	0.2809	0.3539	−0.2480	0.030*
C6	0.2590 (3)	0.4639 (2)	−0.11276 (10)	0.0190 (4)
C7	0.2071 (3)	0.5713 (2)	−0.16602 (11)	0.0269 (4)
H7A	0.0753	0.6095	−0.1553	0.040*
H7B	0.2001	0.5351	−0.2155	0.040*
H7C	0.3120	0.6397	−0.1622	0.040*
C8	0.2306 (3)	0.58395 (19)	0.00240 (10)	0.0176 (4)
C9	0.2387 (3)	0.54692 (19)	0.07755 (10)	0.0181 (4)
C10	0.2008 (3)	0.6441 (2)	0.12986 (11)	0.0208 (4)
H10	0.2053	0.6214	0.1800	0.025*
N1	0.3362 (2)	0.23872 (17)	−0.08411 (9)	0.0193 (3)
N2	0.2667 (2)	0.47613 (16)	−0.04273 (9)	0.0172 (3)

O1	0.2789 (2)	0.42425 (13)	0.09719 (7)	0.0201 (3)
Cu1	0.31041 (4)	0.31370 (3)	0.014256 (14)	0.01721 (13)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0158 (9)	0.0196 (10)	0.0318 (11)	−0.0014 (7)	0.0007 (8)	−0.0067 (8)
C12	0.0179 (10)	0.0169 (10)	0.0362 (12)	−0.0003 (7)	−0.0015 (8)	−0.0001 (8)
C13	0.0173 (10)	0.0203 (10)	0.0262 (10)	−0.0010 (7)	−0.0023 (8)	0.0030 (8)
N3	0.0297 (10)	0.0213 (8)	0.0294 (9)	0.0053 (7)	0.0047 (8)	0.0048 (7)
N4	0.0305 (9)	0.0141 (8)	0.0221 (8)	0.0025 (7)	−0.0051 (7)	−0.0003 (6)
N5	0.0361 (10)	0.0259 (9)	0.0278 (9)	−0.0051 (8)	0.0021 (8)	0.0035 (8)
C1	0.0107 (9)	0.0248 (10)	0.0213 (9)	0.0000 (7)	0.0001 (7)	0.0001 (8)
C2	0.0186 (9)	0.0242 (10)	0.0271 (10)	0.0020 (8)	0.0013 (7)	−0.0035 (8)
C3	0.0209 (10)	0.0283 (11)	0.0320 (11)	−0.0001 (8)	0.0019 (8)	−0.0091 (9)
C4	0.0211 (10)	0.0381 (12)	0.0253 (10)	−0.0010 (9)	0.0020 (8)	−0.0096 (9)
C5	0.0190 (10)	0.0330 (11)	0.0230 (10)	0.0007 (8)	0.0010 (8)	−0.0004 (9)
C6	0.0117 (8)	0.0239 (10)	0.0215 (9)	0.0010 (7)	0.0015 (7)	0.0035 (8)
C7	0.0322 (11)	0.0283 (11)	0.0202 (9)	0.0067 (9)	0.0017 (8)	0.0039 (8)
C8	0.0117 (8)	0.0191 (9)	0.0220 (9)	−0.0007 (7)	0.0006 (7)	−0.0001 (7)
C9	0.0124 (8)	0.0184 (9)	0.0236 (9)	−0.0006 (7)	0.0017 (7)	−0.0008 (8)
C10	0.0157 (9)	0.0233 (10)	0.0233 (9)	−0.0006 (7)	0.0012 (7)	−0.0021 (8)
N1	0.0139 (7)	0.0221 (8)	0.0218 (8)	0.0013 (6)	0.0016 (6)	−0.0016 (7)
N2	0.0123 (7)	0.0193 (8)	0.0199 (7)	−0.0003 (6)	0.0000 (6)	0.0018 (6)
O1	0.0241 (7)	0.0180 (7)	0.0182 (6)	0.0025 (5)	0.0011 (5)	0.0012 (5)
Cu1	0.0182 (2)	0.01587 (19)	0.01753 (19)	0.00191 (10)	0.00069 (12)	0.00060 (10)

Geometric parameters (Å, º)

C11—C10	1.383 (3)	C3—H3	0.9500
C11—C12	1.399 (3)	C4—C5	1.396 (3)
C11—H11	0.9500	C4—H4	0.9500
C12—C13	1.385 (3)	C5—H5	0.9500
C12—H12	0.9500	C6—N2	1.292 (3)
C13—C8	1.408 (3)	C6—C7	1.495 (3)
C13—H13	0.9500	C7—H7A	0.9800
N3—N4	1.201 (3)	C7—H7B	0.9800
N3—Cu1	1.9470 (18)	C7—H7C	0.9800
N4—N5	1.157 (3)	C8—N2	1.402 (3)
C1—N1	1.354 (3)	C8—C9	1.431 (3)
C1—C5	1.385 (3)	C9—O1	1.320 (2)
C1—C6	1.489 (3)	C9—C10	1.409 (3)
C2—N1	1.334 (3)	C10—H10	0.9500
C2—C3	1.395 (3)	N1—Cu1	1.9775 (16)
C2—H2	0.9500	N2—Cu1	1.9682 (16)
C3—C4	1.375 (3)	O1—Cu1	1.9134 (14)
C10—C11—C12	120.79 (18)	C6—C7—H7A	109.5

C10—C11—H11	119.6	C6—C7—H7B	109.5
C12—C11—H11	119.6	H7A—C7—H7B	109.5
C13—C12—C11	120.23 (19)	C6—C7—H7C	109.5
C13—C12—H12	119.9	H7A—C7—H7C	109.5
C11—C12—H12	119.9	H7B—C7—H7C	109.5
C12—C13—C8	120.02 (19)	N2—C8—C13	128.50 (18)
C12—C13—H13	120.0	N2—C8—C9	111.54 (17)
C8—C13—H13	120.0	C13—C8—C9	119.96 (18)
N4—N3—Cu1	117.06 (14)	O1—C9—C10	120.94 (18)
N5—N4—N3	177.4 (2)	O1—C9—C8	120.65 (17)
N1—C1—C5	120.84 (19)	C10—C9—C8	118.41 (18)
N1—C1—C6	114.76 (17)	C11—C10—C9	120.59 (19)
C5—C1—C6	124.37 (19)	C11—C10—H10	119.7
N1—C2—C3	121.6 (2)	C9—C10—H10	119.7
N1—C2—H2	119.2	C2—N1—C1	120.03 (17)
C3—C2—H2	119.2	C2—N1—Cu1	126.68 (14)
C4—C3—C2	119.1 (2)	C1—N1—Cu1	113.19 (14)
C4—C3—H3	120.4	C6—N2—C8	131.76 (18)
C2—C3—H3	120.4	C6—N2—Cu1	116.57 (14)
C3—C4—C5	119.0 (2)	C8—N2—Cu1	111.37 (12)
C3—C4—H4	120.5	C9—O1—Cu1	111.35 (12)
C5—C4—H4	120.5	O1—Cu1—N3	95.95 (7)
C1—C5—C4	119.3 (2)	O1—Cu1—N2	85.04 (6)
C1—C5—H5	120.3	N3—Cu1—N2	175.89 (7)
C4—C5—H5	120.3	O1—Cu1—N1	166.56 (7)
N2—C6—C1	113.71 (17)	N3—Cu1—N1	97.49 (8)
N2—C6—C7	125.38 (19)	N2—Cu1—N1	81.53 (7)
C1—C6—C7	120.89 (17)		

C10—C11—C12—C13	0.3 (3)	C7—C6—N2—C8	0.6 (3)
C11—C12—C13—C8	0.3 (3)	C1—C6—N2—Cu1	−4.7 (2)
Cu1—N3—N4—N5	166 (5)	C7—C6—N2—Cu1	173.67 (15)
N1—C2—C3—C4	0.0 (3)	C13—C8—N2—C6	−4.9 (3)
C2—C3—C4—C5	1.1 (3)	C9—C8—N2—C6	174.29 (18)
N1—C1—C5—C4	1.1 (3)	C13—C8—N2—Cu1	−178.26 (16)
C6—C1—C5—C4	−177.32 (19)	C9—C8—N2—Cu1	0.96 (18)
C3—C4—C5—C1	−1.6 (3)	C10—C9—O1—Cu1	177.20 (14)
N1—C1—C6—N2	1.7 (2)	C8—C9—O1—Cu1	−2.2 (2)
C5—C1—C6—N2	−179.81 (18)	C9—O1—Cu1—N3	−173.93 (13)
N1—C1—C6—C7	−176.72 (17)	C9—O1—Cu1—N2	2.06 (12)
C5—C1—C6—C7	1.7 (3)	C9—O1—Cu1—N1	5.2 (3)
C12—C13—C8—N2	178.35 (18)	N4—N3—Cu1—O1	56.36 (17)
C12—C13—C8—C9	−0.8 (3)	N4—N3—Cu1—N2	−47.4 (11)
N2—C8—C9—O1	0.8 (2)	N4—N3—Cu1—N1	−123.42 (16)
C13—C8—C9—O1	−179.90 (17)	C6—N2—Cu1—O1	−176.11 (14)
N2—C8—C9—C10	−178.59 (16)	C8—N2—Cu1—O1	−1.67 (12)
C13—C8—C9—C10	0.7 (3)	C6—N2—Cu1—N3	−72.0 (10)
C12—C11—C10—C9	−0.4 (3)	C8—N2—Cu1—N3	102.5 (10)

O1—C9—C10—C11	−179.47 (17)	C6—N2—Cu1—N1	4.61 (14)
C8—C9—C10—C11	−0.1 (3)	C8—N2—Cu1—N1	179.05 (13)
C3—C2—N1—C1	−0.6 (3)	C2—N1—Cu1—O1	177.1 (2)
C3—C2—N1—Cu1	175.50 (15)	C1—N1—Cu1—O1	−6.5 (4)
C5—C1—N1—C2	0.1 (3)	C2—N1—Cu1—N3	−3.81 (17)
C6—C1—N1—C2	178.62 (16)	C1—N1—Cu1—N3	172.56 (14)
C5—C1—N1—Cu1	−176.55 (15)	C2—N1—Cu1—N2	−179.78 (17)
C6—C1—N1—Cu1	2.0 (2)	C1—N1—Cu1—N2	−3.41 (13)
C1—C6—N2—C8	−177.73 (17)		