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Azido{2,4-dibromo-6-[2-(diethylamino)-ethyliminomethyl]phenolato}copper(II)

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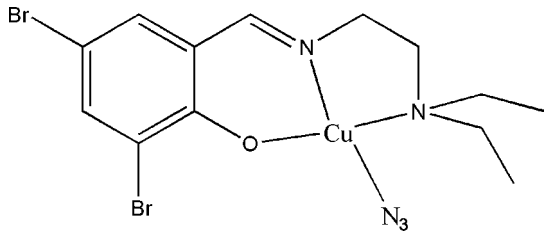
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.041; wR factor = 0.101; data-to-parameter ratio = 18.8.

In the title mononuclear copper(II) complex, $[Cu(C_{13}H_{17}Br_2N_2O)(N_3)]$, the Cu^{II} atom is four-coordinated by the phenolate O, imine N and amine N atoms of the Schiff base ligand, and by the terminal N atom of the azide ligand, forming a square-planar geometry.

Related literature

For related literature, see: Adsule *et al.* (2006); Butcher *et al.* (2003); Diao (2007*a*,*b*); Diao, Huang *et al.* (2007); Diao, Shu *et al.* (2007); Gray *et al.* (1986); Hatfield & Bunger (1969); Hebbachi & Benali-Cherif (2005); Ichikawa *et al.* (1970); Zhu *et al.* (2006).



Experimental

Crystal data

$[Cu(C_{13}H_{17}Br_2N_2O)(N_3)]$
M_r = 482.68
Monoclinic, $P2_1/c$
a = 10.996 (2) Å
b = 11.635 (2) Å
c = 13.330 (3) Å
β = 100.74 (3)°
V = 1675.5 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.08 mm^{-1}
T = 298 (2) K
0.27 × 0.25 × 0.24 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.291, T_{max} = 0.323
(expected range = 0.209–0.232)
13870 measured reflections
3780 independent reflections
2586 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.101
S = 0.99
3780 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.88 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.892 (3)	Cu1—N2	2.066 (4)
Cu1—N1	1.934 (3)	Br1—C3	1.891 (4)
Cu1—N3	1.940 (4)	Br2—C5	1.898 (4)
O1—Cu1—N1	93.78 (14)	O1—Cu1—N2	170.90 (14)
O1—Cu1—N3	93.04 (15)	N1—Cu1—N2	84.26 (15)
N1—Cu1—N3	164.42 (16)	N3—Cu1—N2	91.08 (16)

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: AT2360).

References

Adsule, S., Barve, V., Chen, D., Ahmed, F., Dou, Q. P., Padhye, S. & Sarkar, F. H. (2006). *J. Med. Chem.* **49**, 7242–7246.
Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.01). *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Butcher, R. J., Mockler, G. M. & McKern, O. (2003). *Acta Cryst.* E**59**, m61–m63.
Diao, Y.-P. (2007*a*). *Acta Cryst.* E**63**, m1081–m1083.
Diao, Y.-P. (2007*b*). *Acta Cryst.* E**63**, m1453–m1454.
Diao, Y.-P., Huang, S.-S., Zhang, H.-L., Deng, S. & Liu, K.-X. (2007). *Acta Cryst.* E**63**, m1694.
Diao, Y.-P., Shu, X.-H., Zhang, B.-J., Zhen, Y.-H. & Kang, T.-G. (2007). *Acta Cryst.* E**63**, m1816.
Gray, G. M., Zell, A. L. & Einspahr, H. (1986). *Inorg. Chem.* **25**, 2923–2925.
Hatfield, W. E. & Bunger, F. L. (1969). *Inorg. Chem.* **8**, 1194–1195.
Hebbachi, R. & Benali-Cherif, N. (2005). *Acta Cryst.* E**61**, m1188–m1190.
Ichikawa, T., Maeda, S., Araki, Y. & Ishido, Y. (1970). *J. Am. Chem. Soc.* **92**, 5514–5516.
Zhu, Q.-Y., Wei, Y.-J. & Wang, F.-W. (2006). *Acta Cryst.* E**62**, m983–m985.

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supplementary materials

Acta Cryst. (2007). E**63**, m2271 [**doi:10.1107/S1600536807037798**]

Azido{2,4-dibromo-6-[2-(diethylamino)ethyliminomethyl]phenolato}copper(II)

K. Li, H. Zhang, X.-N. Wang, J.-Y. Peng and Y.-P. Diao

Comment

Copper(II) complexes with Schiff base ligands have received much attention in recent years (Hatfield & Bunger, 1969; Gray *et al.*, 1986; Ichikawa *et al.*, 1970). Some of the complexes have been found to have pharmacological and antitumor properties (Adsule *et al.*, 2006). We have recently reported a few transition metal complexes (Diao, Huang *et al.*, 2007; Diao, Shu *et al.*, 2007; Diao, 2007*a*,*b*). In order to further develop the coordination chemistry of such copper complexes, we report herein the title new copper(II) compound.

The Cu^{II} atom in the mononuclear complex is four-coordinate in a square-planar geometry with one phenolate O, one imine N, and one amine N atoms of one Schiff base ligand and one terminal N atom of an azide ligand (Fig. 1). All the bond values (Table 1) subtended at the metal centres are comparable with the values observed in other Schiff base copper(II) complexes (Hebbachi & Benali-Cherif, 2005; Zhu *et al.*, 2006; Butcher *et al.*, 2003).

Experimental

3,5-Dibromosalicylaldehyde (0.1 mmol, 18.0 mg), *N*,*N*-diethylethane-1,2-diamine (0.1 mmol, 11.6 mg), sodium azide (0.1 mmol, 6.5 mg), and $Cu(CH_3COO)_2{\cdot}H_2O$ (0.1 mmol, 20.0 mg) were dissolved in a methanol solution (10 ml). The mixture was stirred at room temperature for 30 min to give a blue solution. After keeping the solution in air for 5 days, blue block-like crystals were formed.

Refinement

H atoms were placed in calculated 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

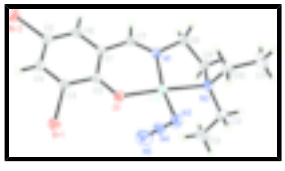


Fig. 1. The structure of the complex with 30% probability level.

Azido{2,4-dibromo-6-[2-(diethylamino)ethyliminomethyl]phenolato}copper(II)

Crystal data

$[Cu(C_{13}H_{17}Br_2N_2O)(N_3)]$ $F_{000} = 948$

M_r = 482.68	D_x = 1.913 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 3167 reflections
a = 10.996 (2) Å	θ = 2.3–24.3°
b = 11.635 (2) Å	μ = 6.08 mm^{-1}
c = 13.330 (3) Å	T = 298 (2) K
β = 100.74 (3)°	Block, blue
V = 1675.5 (6) Å^3	0.27 × 0.25 × 0.24 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3780 independent reflections
Radiation source: fine-focus sealed tube	2586 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.063
T = 298(2) K	θ_{max} = 27.5°
ω scans	θ_{min} = 1.9°
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −14→14
T_{min} = 0.291, T_{max} = 0.323	k = −14→14
13870 measured reflections	l = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.042	H-atom parameters constrained
$wR(F^2)$ = 0.101	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max} < 0.001$
3780 reflections	$\Delta\rho_{max}$ = 0.88 e Å^{-3}
201 parameters	$\Delta\rho_{min}$ = −0.58 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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *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	−0.04346 (5)	0.67672 (4)	0.11117 (4)	0.03252 (16)
Br1	0.28932 (4)	0.76477 (4)	0.39187 (3)	0.04188 (15)
Br2	0.49306 (5)	1.06280 (4)	0.13495 (4)	0.04974 (17)
O1	0.0942 (3)	0.7338 (2)	0.2045 (2)	0.0356 (7)
N1	−0.0162 (3)	0.7771 (3)	0.0018 (3)	0.0336 (8)
N2	−0.2078 (3)	0.6380 (3)	0.0141 (3)	0.0340 (8)
N3	−0.0613 (4)	0.5441 (3)	0.1955 (3)	0.0509 (11)
N4	0.0232 (4)	0.5031 (3)	0.2546 (3)	0.0394 (9)
N5	0.0983 (4)	0.4595 (4)	0.3123 (3)	0.0570 (12)
C1	0.1712 (4)	0.8663 (3)	0.0915 (3)	0.0309 (9)
C2	0.1760 (4)	0.8075 (3)	0.1863 (3)	0.0299 (9)
C3	0.2798 (4)	0.8336 (3)	0.2621 (3)	0.0297 (9)
C4	0.3719 (4)	0.9071 (3)	0.2469 (3)	0.0357 (10)
H4	0.4391	0.9211	0.2991	0.043*
C5	0.3644 (4)	0.9606 (4)	0.1531 (3)	0.0370 (10)
C6	0.2656 (4)	0.9426 (4)	0.0766 (3)	0.0357 (10)
H6	0.2605	0.9805	0.0146	0.043*
C7	0.0728 (4)	0.8491 (3)	0.0063 (3)	0.0326 (10)
H7	0.0735	0.8943	−0.0512	0.039*
C8	−0.1089 (4)	0.7677 (4)	−0.0921 (3)	0.0439 (12)
H8A	−0.0850	0.7092	−0.1366	0.053*
H8B	−0.1177	0.8403	−0.1284	0.053*
C9	−0.2284 (4)	0.7352 (4)	−0.0596 (4)	0.0431 (12)
H9A	−0.2606	0.8009	−0.0280	0.052*
H9B	−0.2891	0.7132	−0.1190	0.052*
C10	−0.1847 (4)	0.5282 (4)	−0.0367 (4)	0.0438 (12)
H10A	−0.1657	0.4695	0.0156	0.053*
H10B	−0.1117	0.5381	−0.0669	0.053*
C11	−0.2884 (5)	0.4838 (5)	−0.1187 (4)	0.0622 (15)
H11A	−0.3620	0.4743	−0.0906	0.093*
H11B	−0.2649	0.4111	−0.1433	0.093*
H11C	−0.3039	0.5378	−0.1740	0.093*
C12	−0.3136 (4)	0.6258 (4)	0.0673 (4)	0.0450 (12)
H12A	−0.3044	0.5545	0.1056	0.054*
H12B	−0.3891	0.6204	0.0166	0.054*
C13	−0.3267 (6)	0.7227 (5)	0.1388 (4)	0.0690 (17)
H13A	−0.2495	0.7342	0.1849	0.103*
H13B	−0.3900	0.7041	0.1770	0.103*
H13C	−0.3490	0.7917	0.1003	0.103*

Atomic displacement parameters (Å^2)

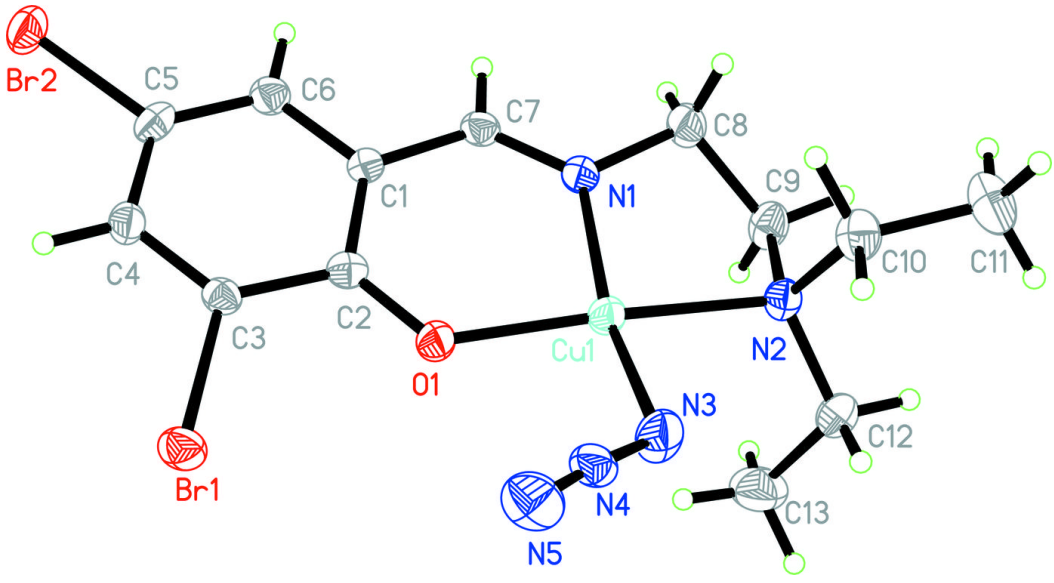
	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0324 (3)	0.0350 (3)	0.0295 (3)	−0.0053 (2)	0.0041 (2)	0.0043 (2)
Br1	0.0420 (3)	0.0507 (3)	0.0322 (3)	0.0050 (2)	0.0049 (2)	0.0075 (2)
Br2	0.0383 (3)	0.0558 (3)	0.0550 (3)	−0.0155 (2)	0.0085 (2)	0.0046 (2)
O1	0.0371 (18)	0.0375 (16)	0.0315 (16)	−0.0089 (14)	0.0046 (14)	0.0042 (13)
N1	0.034 (2)	0.038 (2)	0.0279 (19)	−0.0047 (17)	0.0050 (16)	0.0013 (15)
N2	0.031 (2)	0.0337 (19)	0.036 (2)	−0.0026 (16)	0.0028 (16)	0.0017 (16)
N3	0.055 (3)	0.046 (2)	0.048 (2)	−0.014 (2)	0.001 (2)	0.017 (2)
N4	0.054 (3)	0.034 (2)	0.030 (2)	−0.007 (2)	0.009 (2)	0.0006 (17)
N5	0.064 (3)	0.056 (3)	0.047 (3)	0.008 (2)	0.002 (2)	0.014 (2)
C1	0.031 (2)	0.033 (2)	0.029 (2)	−0.0037 (19)	0.0085 (19)	−0.0008 (18)
C2	0.034 (2)	0.026 (2)	0.032 (2)	0.0034 (19)	0.009 (2)	−0.0001 (17)
C3	0.030 (2)	0.032 (2)	0.027 (2)	0.0051 (18)	0.0057 (19)	−0.0020 (17)
C4	0.028 (3)	0.041 (2)	0.038 (3)	−0.001 (2)	0.004 (2)	−0.007 (2)
C5	0.032 (3)	0.035 (2)	0.048 (3)	−0.0048 (19)	0.015 (2)	0.001 (2)
C6	0.040 (3)	0.035 (2)	0.033 (2)	−0.003 (2)	0.008 (2)	0.0016 (19)
C7	0.039 (3)	0.033 (2)	0.026 (2)	−0.003 (2)	0.0077 (19)	0.0026 (17)
C8	0.042 (3)	0.054 (3)	0.032 (3)	−0.009 (2)	−0.003 (2)	0.008 (2)
C9	0.034 (3)	0.044 (3)	0.048 (3)	−0.002 (2)	−0.001 (2)	0.008 (2)
C10	0.035 (3)	0.044 (3)	0.050 (3)	0.005 (2)	0.001 (2)	0.000 (2)
C11	0.056 (4)	0.062 (3)	0.062 (4)	0.005 (3)	−0.005 (3)	−0.022 (3)
C12	0.032 (3)	0.055 (3)	0.049 (3)	−0.004 (2)	0.010 (2)	−0.002 (2)
C13	0.067 (4)	0.091 (4)	0.055 (4)	0.005 (3)	0.025 (3)	−0.008 (3)

Geometric parameters (Å, °)

Cu1—O1	1.892 (3)	C5—C6	1.360 (6)
Cu1—N1	1.934 (3)	C6—H6	0.9300
Cu1—N3	1.940 (4)	C7—H7	0.9300
Cu1—N2	2.066 (4)	C8—C9	1.507 (6)
Br1—C3	1.891 (4)	C8—H8A	0.9700
Br2—C5	1.898 (4)	C8—H8B	0.9700
O1—C2	1.298 (5)	C9—H9A	0.9700
N1—C7	1.281 (5)	C9—H9B	0.9700
N1—C8	1.464 (5)	C10—C11	1.515 (6)
N2—C12	1.478 (5)	C10—H10A	0.9700
N2—C9	1.487 (6)	C10—H10B	0.9700
N2—C10	1.490 (6)	C11—H11A	0.9600
N3—N4	1.199 (5)	C11—H11B	0.9600
N4—N5	1.138 (5)	C11—H11C	0.9600
C1—C6	1.408 (6)	C12—C13	1.500 (7)
C1—C2	1.429 (5)	C12—H12A	0.9700
C1—C7	1.430 (6)	C12—H12B	0.9700
C2—C3	1.410 (6)	C13—H13A	0.9600
C3—C4	1.370 (6)	C13—H13B	0.9600
C4—C5	1.384 (6)	C13—H13C	0.9600

C4—H4	0.9300		
O1—Cu1—N1	93.78 (14)	C1—C7—H7	117.1
O1—Cu1—N3	93.04 (15)	N1—C8—C9	106.2 (4)
N1—Cu1—N3	164.42 (16)	N1—C8—H8A	110.5
O1—Cu1—N2	170.90 (14)	C9—C8—H8A	110.5
N1—Cu1—N2	84.26 (15)	N1—C8—H8B	110.5
N3—Cu1—N2	91.08 (16)	C9—C8—H8B	110.5
C2—O1—Cu1	127.3 (3)	H8A—C8—H8B	108.7
C7—N1—C8	119.5 (4)	N2—C9—C8	110.3 (4)
C7—N1—Cu1	125.8 (3)	N2—C9—H9A	109.6
C8—N1—Cu1	114.7 (3)	C8—C9—H9A	109.6
C12—N2—C9	110.7 (3)	N2—C9—H9B	109.6
C12—N2—C10	110.6 (3)	C8—C9—H9B	109.6
C9—N2—C10	111.7 (3)	H9A—C9—H9B	108.1
C12—N2—Cu1	113.3 (3)	N2—C10—C11	116.8 (4)
C9—N2—Cu1	104.8 (3)	N2—C10—H10A	108.1
C10—N2—Cu1	105.6 (3)	C11—C10—H10A	108.1
N4—N3—Cu1	123.2 (3)	N2—C10—H10B	108.1
N5—N4—N3	175.8 (5)	C11—C10—H10B	108.1
C6—C1—C2	121.3 (4)	H10A—C10—H10B	107.3
C6—C1—C7	116.4 (4)	C10—C11—H11A	109.5
C2—C1—C7	122.3 (4)	C10—C11—H11B	109.5
O1—C2—C3	120.3 (4)	H11A—C11—H11B	109.5
O1—C2—C1	124.7 (4)	C10—C11—H11C	109.5
C3—C2—C1	115.0 (4)	H11A—C11—H11C	109.5
C4—C3—C2	123.4 (4)	H11B—C11—H11C	109.5
C4—C3—Br1	118.9 (3)	N2—C12—C13	114.2 (4)
C2—C3—Br1	117.6 (3)	N2—C12—H12A	108.7
C3—C4—C5	119.5 (4)	C13—C12—H12A	108.7
C3—C4—H4	120.2	N2—C12—H12B	108.7
C5—C4—H4	120.2	C13—C12—H12B	108.7
C6—C5—C4	120.9 (4)	H12A—C12—H12B	107.6
C6—C5—Br2	120.7 (3)	C12—C13—H13A	109.5
C4—C5—Br2	118.4 (3)	C12—C13—H13B	109.5
C5—C6—C1	119.9 (4)	H13A—C13—H13B	109.5
C5—C6—H6	120.1	C12—C13—H13C	109.5
C1—C6—H6	120.1	H13A—C13—H13C	109.5
N1—C7—C1	125.9 (4)	H13B—C13—H13C	109.5
N1—C7—H7	117.1		

Fig. 1


Br2
C5
C6
C7
C8
C1
N1
C9
C4
C10
C11
C2
C3
Cu1
N2
O1
N3
C12
Br1
N4
C13
N5