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Bis(μ-azido-κ^2N,N)bis({2-[3-(dimethylamino)propyliminomethyl]-4-nitrophenolato-κ^3O,N,N'}copper(II))

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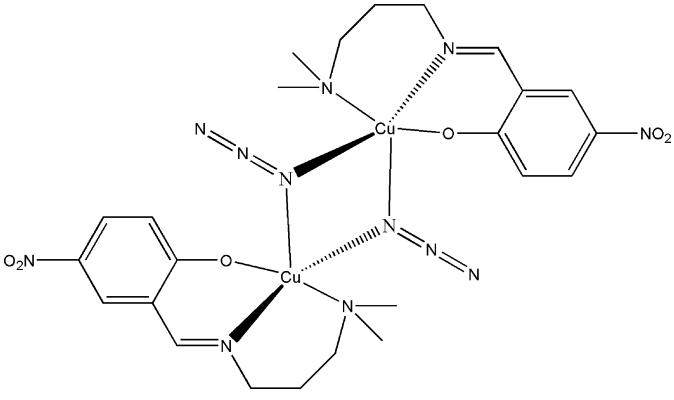
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.074; data-to-parameter ratio = 16.1.

The title complex, $[Cu_2(N_3)_2(C_{12}H_{16}N_3O_3)_2]$, is an azide-bridged dinuclear copper(II) complex. There is a crystallographic inversion centre at the mid-point of the Cu···Cu vector. Each Cu atom is coordinated by one O and two N atoms of one Schiff base ligand and two bridging N atoms from two azide ligands, forming a trigonal–bipyramidal geometry. The Cu···Cu distance is 3.327 (2) Å.

Related literature

For related literature, see: Cai *et al.* (2007); Li *et al.* (2007); Yu *et al.* (2006); Zhang *et al.* (2006); Zhao *et al.* (2003); Zhu *et al.* (2004).



Experimental

Crystal data

$[Cu_2(N_3)_2(C_{12}H_{16}N_3O_3)_2]$
M_r = 711.70
Monoclinic, $P2_1/n$
a = 8.7410 (17) Å
b = 14.846 (3) Å
c = 11.775 (2) Å
β = 102.43 (3)°
V = 1492.2 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.49 mm^{-1}
T = 293 (2) K
0.40 × 0.38 × 0.35 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.588, T_{max} = 0.624
12293 measured reflections
3239 independent reflections
2706 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.074
S = 1.04
3239 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.35 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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: BT2367).

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supplementary materials

Acta Cryst. (2007). E**63**, m1767 [**doi:10.1107/S1600536807023902**]

Bis(μ-azido-κ^2N,N)bis({2-[3-(dimethylamino)propyliminomethyl]-4-nitrophenolato-κ^3O,N,N'}copper(II))

H.-Y. Hou

Comment

Many azide-bridged polynuclear complexes have been reported previously (Zhao *et al.*, 2003; Cai *et al.*, 2007; Yu *et al.*, 2006; Zhang *et al.*, 2006; Zhu *et al.*, 2004; Li *et al.*, 2007). These complexes have interesting structures. We report herein the new copper(II) complex, (I), derived from the Schiff base ligand 4-nitro-2-[(3-dimethylaminopropylimino)methyl]phenol with azide bridges.

Complex (I) is an azide-bridged dinuclear copper(II) complex (Fig. 1). There is a crystallographic inversion centre at the midpoint of the two copper atoms in the complex. Each Cu atom is coordinated by one O and two N atoms of one Schiff base ligand and two briding N atoms from two azide ligands, forming a trigonal-bipyramidal geometry. The Cu···Cu distance is 3.327 (2) Å.

Experimental

5-Nitro-2-hydroxybenzaldehyde (0.2 mmol, 33.5 mg), *N*,*N*-dimethylpropane-1,3-diamine (0.2 mmol, 20.5 mg), NaN_3 (0.2 mmol, 6.5 mg), and $Cu(CH_3COO)_2{\cdot}H_2O$ (0.2 mmol, 40.0 mg) were mixed in a methanol solution. The mixture was stirred at 325 K for 30 min to give a transparent blue solution. Blue crystals were obtained by slow evaporation of the solution in air.

Refinement

H atoms were positioned geometrically and refined as riding atoms, with C–H distances in the range 0.93–0.97Å and U_{iso}(H) set at 1.2U_{eq}(C) and 1.5U_{eq}(methyl C).

Figures

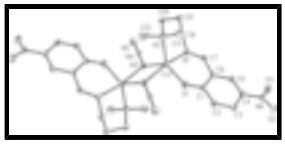

Fig. 1. The molecular structure of (I), with anisotropic displacement ellipsoids drawn at the 30% probability level.

Bis(μ-azido-κ^2N,N)bis({2- [3-(dimethylamino)propyliminomethyl]-4-nitrophenolato- κ^3O,N,N'}copper(II))

Crystal data

$[Cu_2(N_3)_2(C_{12}H_{16}N_3O_3)_2]$	F_{000} = 732
M_r = 711.70	D_x = 1.584 Mg m^{-3}

Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.7410\ (17)$ Å	Cell parameters from 3793 reflections
$b = 14.846\ (3)$ Å	$\theta = 2.2–27.3°$
$c = 11.775\ (2)$ Å	$\mu = 1.49$ mm^{-1}
$\beta = 102.43\ (3)°$	$T = 293\ (2)$ K
$V = 1492.2\ (5)$ Å^3	Block, blue
$Z = 2$	0.40 × 0.38 × 0.35 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3239 independent reflections
Radiation source: fine-focus sealed tube	2706 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.036$
$T = 293(2)$ K	$\theta_{max} = 27.0°$
ω scans	$\theta_{min} = 2.2°$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -11\rightarrow11$
$T_{min} = 0.588$, $T_{max} = 0.624$	$k = -18\rightarrow18$
12293 measured reflections	$l = -15\rightarrow15$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H-atom parameters constrained
$wR(F^2) = 0.074$	$w = 1/[\sigma^2(F_o^2) + (0.0349P)^2 + 0.1139P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
3239 reflections	$\Delta\rho_{max} = 0.35$ e Å^{-3}
201 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	0.94961 (3)	0.106755 (14)	0.014558 (19)	0.02791 (9)
O1	1.14184 (17)	0.17565 (9)	0.07071 (14)	0.0480 (4)
O2	1.42356 (19)	0.55892 (11)	0.08461 (15)	0.0562 (5)
O3	1.2338 (2)	0.57421 (11)	−0.06657 (16)	0.0573 (4)
N1	0.85117 (18)	0.21807 (10)	−0.07976 (14)	0.0312 (4)
N2	0.7638 (2)	0.10303 (11)	0.10393 (15)	0.0374 (4)
N3	0.90672 (19)	0.00703 (11)	−0.10862 (15)	0.0371 (4)
N4	0.7826 (2)	−0.00746 (11)	−0.17324 (15)	0.0359 (4)
N5	0.6652 (2)	−0.02203 (14)	−0.23562 (18)	0.0557 (6)
N6	1.3106 (2)	0.52861 (12)	0.01293 (17)	0.0416 (4)
C1	1.1769 (2)	0.25787 (13)	0.05229 (17)	0.0328 (4)
C2	1.3234 (2)	0.29231 (14)	0.11226 (18)	0.0371 (5)
H2	1.3917	0.2542	0.1616	0.044*
C3	1.3673 (2)	0.37865 (14)	0.10029 (19)	0.0388 (5)
H3	1.4635	0.3996	0.1417	0.047*
C4	1.2658 (2)	0.43592 (13)	0.02469 (18)	0.0343 (5)
C5	1.1238 (2)	0.40603 (12)	−0.03678 (18)	0.0333 (5)
H5	1.0589	0.4448	−0.0875	0.040*
C6	1.0759 (2)	0.31767 (13)	−0.02370 (16)	0.0295 (4)
C7	0.9186 (2)	0.29377 (13)	−0.08454 (17)	0.0314 (4)
H7	0.8614	0.3379	−0.1315	0.038*
C8	0.6851 (2)	0.21132 (14)	−0.13842 (18)	0.0376 (5)
H8A	0.6699	0.1597	−0.1902	0.045*
H8B	0.6540	0.2650	−0.1846	0.045*
C9	0.5848 (2)	0.20112 (15)	−0.0484 (2)	0.0428 (5)
H9A	0.6061	0.2516	0.0049	0.051*
H9B	0.4755	0.2048	−0.0880	0.051*
C10	0.6085 (2)	0.11484 (14)	0.0218 (2)	0.0401 (5)
H10A	0.5274	0.1113	0.0664	0.048*
H10B	0.5931	0.0645	−0.0320	0.048*
C11	0.7889 (3)	0.17461 (19)	0.1933 (2)	0.0604 (7)
H11A	0.7017	0.1761	0.2309	0.091*
H11B	0.7979	0.2318	0.1571	0.091*
H11C	0.8834	0.1625	0.2499	0.091*
C12	0.7584 (3)	0.01507 (18)	0.1626 (2)	0.0590 (7)
H12A	0.8537	0.0065	0.2197	0.089*
H12B	0.7470	−0.0325	0.1061	0.089*
H12C	0.6710	0.0142	0.1999	0.089*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.02889 (14)	0.01925 (13)	0.03410 (15)	−0.00229 (9)	0.00353 (10)	−0.00002 (9)
O1	0.0389 (8)	0.0283 (8)	0.0686 (11)	−0.0054 (6)	−0.0064 (7)	0.0129 (7)

O2	0.0549 (10)	0.0413 (10)	0.0700 (12)	−0.0213 (8)	0.0078 (9)	−0.0117 (8)
O3	0.0655 (11)	0.0321 (8)	0.0720 (12)	−0.0070 (8)	0.0100 (9)	0.0082 (9)
N1	0.0303 (9)	0.0265 (9)	0.0352 (9)	−0.0001 (7)	0.0035 (7)	0.0009 (7)
N2	0.0352 (9)	0.0385 (10)	0.0377 (10)	−0.0022 (7)	0.0059 (8)	0.0036 (8)
N3	0.0367 (9)	0.0283 (9)	0.0412 (10)	0.0004 (8)	−0.0031 (8)	−0.0044 (7)
N4	0.0454 (10)	0.0247 (9)	0.0349 (10)	0.0028 (8)	0.0028 (8)	−0.0036 (7)
N5	0.0479 (11)	0.0508 (12)	0.0565 (13)	0.0010 (10)	−0.0150 (10)	−0.0132 (10)
N6	0.0439 (10)	0.0312 (10)	0.0536 (12)	−0.0073 (8)	0.0188 (9)	−0.0064 (9)
C1	0.0366 (11)	0.0255 (10)	0.0366 (11)	−0.0018 (8)	0.0086 (9)	0.0002 (8)
C2	0.0310 (11)	0.0369 (12)	0.0414 (12)	0.0006 (9)	0.0034 (9)	0.0050 (9)
C3	0.0310 (11)	0.0420 (13)	0.0428 (12)	−0.0065 (9)	0.0064 (9)	−0.0032 (10)
C4	0.0380 (11)	0.0272 (10)	0.0407 (12)	−0.0075 (9)	0.0153 (9)	−0.0035 (9)
C5	0.0376 (11)	0.0267 (10)	0.0365 (11)	0.0007 (8)	0.0099 (9)	0.0016 (8)
C6	0.0302 (10)	0.0260 (10)	0.0330 (11)	−0.0019 (8)	0.0081 (8)	−0.0006 (8)
C7	0.0356 (11)	0.0260 (10)	0.0316 (10)	0.0006 (8)	0.0053 (8)	0.0036 (8)
C8	0.0338 (11)	0.0327 (11)	0.0421 (12)	−0.0024 (9)	−0.0010 (9)	0.0047 (9)
C9	0.0306 (11)	0.0411 (13)	0.0546 (14)	0.0049 (9)	0.0042 (10)	0.0054 (10)
C10	0.0309 (11)	0.0422 (13)	0.0468 (13)	−0.0029 (9)	0.0072 (9)	0.0018 (10)
C11	0.0541 (15)	0.0777 (19)	0.0489 (15)	−0.0052 (13)	0.0101 (12)	−0.0190 (14)
C12	0.0505 (14)	0.0621 (17)	0.0653 (17)	−0.0008 (12)	0.0145 (13)	0.0314 (14)

Geometric parameters (Å, °)

Cu1—O1	1.9567 (14)	C3—C4	1.401 (3)
Cu1—N3	2.0498 (17)	C3—H3	0.9300
Cu1—N1	2.0720 (16)	C4—C5	1.369 (3)
Cu1—N2	2.1178 (18)	C5—C6	1.396 (3)
Cu1—N3^{i}	2.2492 (17)	C5—H5	0.9300
O1—C1	1.288 (2)	C6—C7	1.451 (2)
O2—N6	1.237 (2)	C7—H7	0.9300
O3—N6	1.231 (2)	C8—C9	1.522 (3)
N1—C7	1.276 (2)	C8—H8A	0.9700
N1—C8	1.470 (2)	C8—H8B	0.9700
N2—C11	1.478 (3)	C9—C10	1.514 (3)
N2—C12	1.483 (3)	C9—H9A	0.9700
N2—C10	1.498 (3)	C9—H9B	0.9700
N3—N4	1.203 (2)	C10—H10A	0.9700
N3—Cu1^{i}	2.2492 (17)	C10—H10B	0.9700
N4—N5	1.147 (2)	C11—H11A	0.9600
N6—C4	1.445 (3)	C11—H11B	0.9600
C1—C2	1.417 (3)	C11—H11C	0.9600
C1—C6	1.424 (3)	C12—H12A	0.9600
C2—C3	1.354 (3)	C12—H12B	0.9600
C2—H2	0.9300	C12—H12C	0.9600
O1—Cu1—N3	129.02 (7)	C4—C5—C6	120.39 (19)
O1—Cu1—N1	89.52 (6)	C4—C5—H5	119.8
N3—Cu1—N1	101.84 (7)	C6—C5—H5	119.8
O1—Cu1—N2	122.72 (7)	C5—C6—C1	119.65 (18)

N3—Cu1—N2	106.84 (7)	C5—C6—C7	116.77 (17)
N1—Cu1—N2	90.73 (6)	C1—C6—C7	123.47 (18)
O1—Cu1—N3^{i}	83.01 (6)	N1—C7—C6	126.24 (18)
N3—Cu1—N3^{i}	78.68 (7)	N1—C7—H7	116.9
N1—Cu1—N3^{i}	170.54 (6)	C6—C7—H7	116.9
N2—Cu1—N3^{i}	98.18 (6)	N1—C8—C9	109.78 (17)
C1—O1—Cu1	131.04 (13)	N1—C8—H8A	109.7
C7—N1—C8	117.25 (16)	C9—C8—H8A	109.7
C7—N1—Cu1	125.72 (14)	N1—C8—H8B	109.7
C8—N1—Cu1	116.84 (12)	C9—C8—H8B	109.7
C11—N2—C12	108.4 (2)	H8A—C8—H8B	108.2
C11—N2—C10	110.51 (17)	C10—C9—C8	115.64 (18)
C12—N2—C10	106.79 (16)	C10—C9—H9A	108.4
C11—N2—Cu1	108.82 (14)	C8—C9—H9A	108.4
C12—N2—Cu1	111.05 (14)	C10—C9—H9B	108.4
C10—N2—Cu1	111.20 (13)	C8—C9—H9B	108.4
N4—N3—Cu1	125.42 (14)	H9A—C9—H9B	107.4
N4—N3—Cu1^{i}	120.61 (13)	N2—C10—C9	116.75 (17)
Cu1—N3—Cu1^{i}	101.32 (7)	N2—C10—H10A	108.1
N5—N4—N3	179.1 (2)	C9—C10—H10A	108.1
O3—N6—O2	122.90 (19)	N2—C10—H10B	108.1
O3—N6—C4	118.82 (18)	C9—C10—H10B	108.1
O2—N6—C4	118.27 (19)	H10A—C10—H10B	107.3
O1—C1—C2	118.69 (18)	N2—C11—H11A	109.5
O1—C1—C6	123.94 (18)	N2—C11—H11B	109.5
C2—C1—C6	117.36 (18)	H11A—C11—H11B	109.5
C3—C2—C1	122.37 (19)	N2—C11—H11C	109.5
C3—C2—H2	118.8	H11A—C11—H11C	109.5
C1—C2—H2	118.8	H11B—C11—H11C	109.5
C2—C3—C4	119.07 (19)	N2—C12—H12A	109.5
C2—C3—H3	120.5	N2—C12—H12B	109.5
C4—C3—H3	120.5	H12A—C12—H12B	109.5
C5—C4—C3	121.14 (19)	N2—C12—H12C	109.5
C5—C4—N6	119.42 (19)	H12A—C12—H12C	109.5
C3—C4—N6	119.43 (19)	H12B—C12—H12C	109.5

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

Fig. 1