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catena-Poly[[{2,4-dichloro-6-[2-(ethylamino)ethyliminomethyl]phenolato}copper(II)]-μ-azido]

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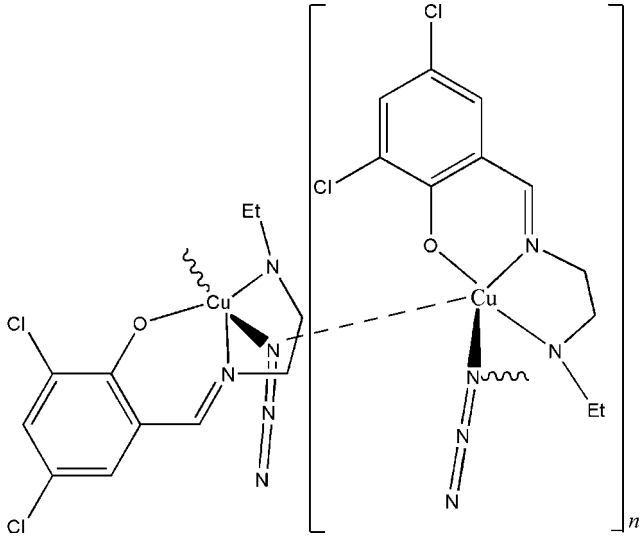
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.038; wR factor = 0.089; data-to-parameter ratio = 17.8.

The title compound, $[Cu(C_{11}H_{13}Cl_2N_2O)(N_3)]_n$, is an azide-bridged polymeric copper(II) complex. The Cu atom is pentacoordinated by one O and two N atoms of the Schiff base ligand and two bridging N atoms from two azide groups, forming a trigonal–bipyramidal geometry. The structure is further stabilized by an N—H···O hydrogen bond.

Related literature

For related literature, see: Diao (2007*a*, 2007*b*); El-Behairy et al. (1997); Escuer *et al.* (2000); Eshel *et al.* (2000); Jiang *et al.* (2005); Manhas *et al.* (2005).



Experimental

Crystal data

$[Cu(C_{11}H_{13}Cl_2N_2O)(N_3)]$
M_r = 365.70
Monoclinic, $P2_1/c$
a = 9.0767 (18) Å
b = 22.897 (4) Å
c = 7.2953 (14) Å
β = 96.448 (3)°
V = 1506.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.81 mm^{-1}
T = 298 (2) K
0.34 × 0.10 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.579, T_{max} = 0.884
12541 measured reflections
3287 independent reflections
2564 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.089
S = 1.02
3287 reflections
185 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.899 (10)	2.121 (15)	2.989 (3)	162 (3)

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

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: BT2379).

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supplementary materials

Acta Cryst. (2007). E**63**, m1816 [**doi:10.1107/S1600536807026591**]

catena-Poly[[{2,4-dichloro-6-[2-(ethylamino)ethyliminomethyl]phenolato}copper(II)]-μ-azido]

Y.-P. Diao, X.-H. Shu, B.-J. Zhang, Y.-H. Zhen and T.-G. Kang

Comment

Metal-organic compounds play an important role in the development of coordination chemistry related to magnetism and molecular architectures (Eshel *et al.*, 2000; Jiang *et al.*, 2005; Escuer *et al.*, 2000; El-Behairy *et al.*, 1997; Manhas *et al.*, 2005). We have recently reported a few transition metal complexes (Diao, 2007*a*,b). As an extension of the work on the crystal structures of such complexes, we report herein the crystal structure of the title complex.

The title compound is an azide-bridged polynuclear copper(II) complex. Each copper atom is pentacoordinated by one O and two N atoms of the Schiff base ligand and two bridging N atoms from two azide groups, forming a trigonal bipyramidal geometry. The structure is further stabilized by an N—H···O hydrogen bond.

Experimental

3,5-Dichlorosalicylaldehyde (0.1 mmol, 19.0 mg), *N*-ethylethane-1,2-diamine (0.1 mmol, 8.8 mg), sodium azide (0.1 mmol, 6.5 mg), and copper acetate (0.1 mmol, 20.0 mg) were dissolved in a methanol solution (20 ml). The mixture was stirred for half an hour at room temperature, giving a blue solution. After allowing the solution to stand in air for a week, blue needle-like crystals were formed.

Refinement

The amino H atom was located in a difference Fourier map and refined isotropically, with the N–H distance restrained to 0.90 (1) Å. The remaining H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C–H = 0.93–0.97 Å, and with U_{iso}(H) set at 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl}).

Figures

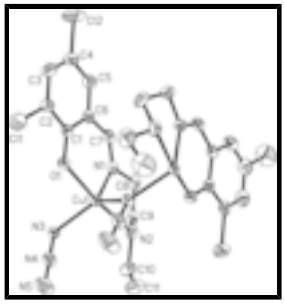

Fig. 1. Molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

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Crystal data

$[Cu(C_{11}H_{13}Cl_2N_2O)(N_3)]$	$F_{000} = 740$
$M_r = 365.70$	$D_x = 1.612$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.0767$ (18) Å	Cell parameters from 2549 reflections
$b = 22.897$ (4) Å	$\theta = 2.4$–$25.1°$
$c = 7.2953$ (14) Å	$\mu = 1.81$ mm^{-1}
$\beta = 96.448$ (3)°	$T = 298$ (2) K
$V = 1506.6$ (5) Å^3	Needle, blue
$Z = 4$	0.34 × 0.10 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer	3287 independent reflections
Radiation source: fine-focus sealed tube	2564 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.045$
$T = 298$(2) K	$\theta_{max} = 27.0°$
ω scans	$\theta_{min} = 1.8°$
Absorption correction: multi-scan (SADABS; Bruker, 2000)	$h = -11 \rightarrow 11$
$T_{min} = 0.579$, $T_{max} = 0.884$	$k = -27 \rightarrow 29$
12541 measured reflections	$l = -9 \rightarrow 9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.089$	$w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.4196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} < 0.001$
3287 reflections	$\Delta\rho_{max} = 0.33$ e Å^{-3}
185 parameters	$\Delta\rho_{min} = -0.39$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	0.09615 (4)	0.257917 (13)	0.30458 (4)	0.03101 (12)
Cl1	0.09232 (11)	0.04857 (4)	0.36375 (15)	0.0727 (3)
Cl2	0.65892 (15)	0.03398 (6)	0.2224 (2)	0.1229 (5)
O1	0.1433 (2)	0.17315 (8)	0.3618 (3)	0.0414 (5)
N1	0.3197 (3)	0.27140 (10)	0.3065 (3)	0.0410 (6)
N2	0.0927 (3)	0.35075 (11)	0.2526 (3)	0.0474 (6)
N3	−0.0139 (3)	0.26061 (10)	0.5403 (3)	0.0374 (5)
N4	−0.1446 (3)	0.26976 (12)	0.5340 (3)	0.0500 (6)
N5	−0.2703 (4)	0.27769 (19)	0.5282 (5)	0.0922 (12)
C1	0.2593 (3)	0.14407 (12)	0.3292 (4)	0.0379 (6)
C2	0.2569 (4)	0.08268 (13)	0.3269 (4)	0.0486 (8)
C3	0.3771 (4)	0.04916 (15)	0.2956 (5)	0.0647 (10)
H3	0.3704	0.0086	0.2953	0.078*
C4	0.5069 (4)	0.07609 (17)	0.2650 (5)	0.0664 (10)
C5	0.5183 (4)	0.13493 (16)	0.2671 (4)	0.0568 (9)
H5	0.6078	0.1524	0.2481	0.068*
C6	0.3963 (3)	0.17011 (13)	0.2977 (4)	0.0431 (7)
C7	0.4186 (3)	0.23208 (14)	0.2918 (4)	0.0443 (7)
H7	0.5135	0.2450	0.2758	0.053*
C8	0.3617 (4)	0.33280 (14)	0.2894 (4)	0.0521 (8)
H8A	0.3820	0.3407	0.1640	0.062*
H8B	0.4510	0.3409	0.3715	0.062*
C9	0.2379 (4)	0.37133 (14)	0.3377 (4)	0.0575 (9)
H9A	0.2380	0.3722	0.4707	0.069*
H9B	0.2543	0.4108	0.2965	0.069*
C10	−0.0293 (5)	0.38750 (15)	0.3046 (6)	0.0739 (11)
H10A	−0.0044	0.4283	0.2895	0.089*
H10B	−0.0408	0.3811	0.4336	0.089*
C11	−0.1735 (5)	0.3744 (2)	0.1897 (7)	0.0940 (14)
H11A	−0.1638	0.3822	0.0624	0.141*
H11B	−0.2503	0.3985	0.2295	0.141*
H11C	−0.1984	0.3340	0.2039	0.141*
H2	0.096 (4)	0.3509 (15)	0.1300 (16)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0379 (2)	0.02888 (18)	0.02698 (18)	−0.00013 (14)	0.00675 (13)	−0.00008 (13)
Cl1	0.0856 (7)	0.0403 (5)	0.0954 (7)	−0.0123 (4)	0.0246 (6)	−0.0038 (4)
Cl2	0.0975 (9)	0.1071 (10)	0.1697 (14)	0.0592 (8)	0.0405 (9)	−0.0002 (9)
O1	0.0449 (11)	0.0359 (11)	0.0453 (12)	0.0020 (9)	0.0131 (9)	0.0008 (9)
N1	0.0477 (15)	0.0430 (14)	0.0318 (13)	−0.0116 (11)	0.0026 (11)	0.0025 (10)
N2	0.0741 (19)	0.0333 (13)	0.0351 (13)	0.0037 (12)	0.0076 (13)	0.0022 (11)
N3	0.0378 (13)	0.0441 (14)	0.0309 (12)	0.0041 (11)	0.0059 (10)	0.0006 (10)
N4	0.0478 (17)	0.0660 (18)	0.0370 (15)	0.0028 (13)	0.0084 (12)	−0.0010 (12)
N5	0.048 (2)	0.158 (4)	0.071 (2)	0.014 (2)	0.0125 (17)	0.010 (2)
C1	0.0446 (17)	0.0406 (16)	0.0283 (14)	0.0012 (13)	0.0028 (12)	0.0002 (11)
C2	0.062 (2)	0.0419 (18)	0.0428 (18)	0.0077 (15)	0.0109 (15)	−0.0007 (13)
C3	0.085 (3)	0.046 (2)	0.063 (2)	0.0216 (19)	0.009 (2)	0.0015 (17)
C4	0.068 (2)	0.063 (2)	0.070 (2)	0.0281 (19)	0.0153 (19)	0.0048 (19)
C5	0.0442 (19)	0.074 (3)	0.053 (2)	0.0082 (17)	0.0066 (15)	0.0076 (17)
C6	0.0472 (17)	0.0504 (18)	0.0317 (16)	0.0031 (14)	0.0039 (13)	0.0021 (13)
C7	0.0381 (17)	0.061 (2)	0.0341 (16)	−0.0048 (14)	0.0043 (13)	0.0023 (14)
C8	0.068 (2)	0.0487 (19)	0.0376 (17)	−0.0207 (16)	−0.0047 (15)	0.0057 (14)
C9	0.095 (3)	0.0362 (17)	0.0384 (17)	−0.0140 (17)	−0.0072 (17)	0.0004 (13)
C10	0.103 (3)	0.0378 (19)	0.085 (3)	0.016 (2)	0.029 (2)	0.0015 (18)
C11	0.087 (3)	0.078 (3)	0.120 (4)	0.031 (3)	0.024 (3)	0.020 (3)

Geometric parameters (Å, °)

Cu1—O1	2.0209 (19)	C3—C4	1.371 (5)
Cu1—N1	2.051 (2)	C3—H3	0.9300
Cu1—N3	2.085 (2)	C4—C5	1.351 (5)
Cu1—N3^{i}	2.112 (2)	C5—C6	1.408 (4)
Cu1—N2	2.159 (2)	C5—H5	0.9300
Cl1—C2	1.733 (3)	C6—C7	1.435 (4)
Cl2—C4	1.740 (3)	C7—H7	0.9300
O1—C1	1.290 (3)	C8—C9	1.502 (5)
N1—C7	1.284 (4)	C8—H8A	0.9700
N1—C8	1.466 (4)	C8—H8B	0.9700
N2—C9	1.469 (4)	C9—H9A	0.9700
N2—C10	1.474 (4)	C9—H9B	0.9700
N2—H2	0.899 (10)	C10—C11	1.503 (6)
N3—N4	1.201 (3)	C10—H10A	0.9700
N3—Cu1^{ii}	2.112 (2)	C10—H10B	0.9700
N4—N5	1.151 (4)	C11—H11A	0.9600
C1—C2	1.406 (4)	C11—H11B	0.9600
C1—C6	1.421 (4)	C11—H11C	0.9600
C2—C3	1.373 (4)		
O1—Cu1—N1	87.52 (9)	C3—C4—Cl2	119.6 (3)
O1—Cu1—N3	88.17 (8)	C4—C5—C6	120.8 (3)

N1—Cu1—N3	123.73 (9)	C4—C5—H5	119.6
O1—Cu1—N3^{i}	93.61 (8)	C6—C5—H5	119.6
N1—Cu1—N3^{i}	113.73 (9)	C5—C6—C1	120.3 (3)
N3—Cu1—N3^{i}	122.53 (11)	C5—C6—C7	116.4 (3)
O1—Cu1—N2	168.65 (9)	C1—C6—C7	123.3 (3)
N1—Cu1—N2	81.24 (10)	N1—C7—C6	126.0 (3)
N3—Cu1—N2	96.73 (9)	N1—C7—H7	117.0
N3^{i}—Cu1—N2	92.37 (9)	C6—C7—H7	117.0
C1—O1—Cu1	128.01 (17)	N1—C8—C9	109.5 (3)
C7—N1—C8	118.3 (3)	N1—C8—H8A	109.8
C7—N1—Cu1	126.5 (2)	C9—C8—H8A	109.8
C8—N1—Cu1	114.2 (2)	N1—C8—H8B	109.8
C9—N2—C10	111.8 (3)	C9—C8—H8B	109.8
C9—N2—Cu1	104.29 (18)	H8A—C8—H8B	108.2
C10—N2—Cu1	120.9 (2)	N2—C9—C8	111.8 (2)
C9—N2—H2	107 (2)	N2—C9—H9A	109.3
C10—N2—H2	111 (2)	C8—C9—H9A	109.3
Cu1—N2—H2	100 (2)	N2—C9—H9B	109.3
N4—N3—Cu1	122.42 (19)	C8—C9—H9B	109.3
N4—N3—Cu1^{ii}	115.75 (19)	H9A—C9—H9B	107.9
Cu1—N3—Cu1^{ii}	121.73 (11)	N2—C10—C11	112.0 (3)
N5—N4—N3	179.0 (4)	N2—C10—H10A	109.2
O1—C1—C2	120.4 (3)	C11—C10—H10A	109.2
O1—C1—C6	124.1 (3)	N2—C10—H10B	109.2
C2—C1—C6	115.5 (3)	C11—C10—H10B	109.2
C3—C2—C1	123.3 (3)	H10A—C10—H10B	107.9
C3—C2—Cl1	119.2 (3)	C10—C11—H11A	109.5
C1—C2—Cl1	117.4 (2)	C10—C11—H11B	109.5
C4—C3—C2	119.3 (3)	H11A—C11—H11B	109.5
C4—C3—H3	120.4	C10—C11—H11C	109.5
C2—C3—H3	120.4	H11A—C11—H11C	109.5
C5—C4—C3	120.8 (3)	H11B—C11—H11C	109.5
C5—C4—Cl2	119.6 (3)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.899 (10)	2.121 (15)	2.989 (3)	162 (3)

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

Fig. 1