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[*N*-(2-Oxidophenyl)salicylaldiminato-κ^3N,O,O']-(2,2′:6′,2″-terpyridine-κ^3N)copper(II)

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The title compound, [$Cu(C_{13}H_9NO_2)(C_{15}H_{11}N_3)$], was prepared by direct reaction of the ligands with copper(II) salts in methanol solution. The air-stable compound crystallizes in the mononclinic space group *C*2/*c*. Although the imine portion of the chelate is disordered over a twofold axis, the copper environment is well defined as a meridionally substituted, tetragonally distorted octahedron.

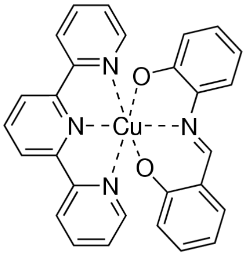
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
Disorder in main residue
R factor = 0.045
wR factor = 0.105
Data-to-parameter ratio = 11.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

In the course of preparing a series of complexes based on cyclotriveratrylene (Bohle & Stasko, 1998), a number of salicylaldimines were prepared as ancillary ligands (Bohle & Stasko, 2000). During these studies, the title compound, [Cu(terpy)(salicylaldimine)], (I), was prepared and crystallographically characterized. The title compound was synthesized as a possible model for copper(II) constellation enzymes (Fenton & Õkawa, 1993). Unfortunately, the low yield (<5%) of the synthesis precluded further work along these lines.



(I)

Compound (I) was found to have symmetry-related disorder. The molecule lies on a twofold axis, which bisects both ligands, resulting in disorder of the salicylaldimine chelate (Fig. 1). While the terpy ligand is also bisected, it has C_2 symmetry and its geometry is unaffected (Table 1). The lower symmetry of the imine causes the appearance of a slight compression in the N1—C2—C7 bond angle of the chelate ring to 100.4 (4)°. This is significantly smaller than the 115–120° range of angles (Elmali *et al.*, 1994) normally found for this chelate, but is an artifact resulting from the disorder. The geometric parameters observed are comparable to a chemically similar disordered structure, including the apparent distortion of the chelate framework, though the previously reported deviation is not as profound (Elmali *et al.*, 1993).

The copper coordination environment is tetragonally distorted, with distances similar to known terpy copper(II) (Cini & Pifferi, 1999) and square-planar salicylaldimine–

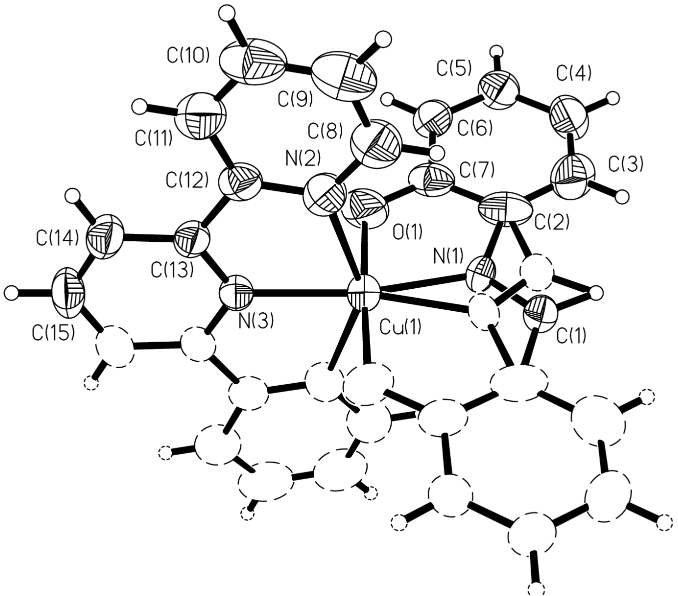


Figure 1

The title complex, with 50% probability ellipsoids, showing the disorder. The symmetry-generated part of the molecule is shown dashed.

copper(II) complexes (Elmali *et al.*, 1993; Wei *et al.*, 1964). The meridional O—N—O coordination of the salicylaldimine and the central N atom of the terpy ligand form a pseudo-square-planar environment with very similar bond lengths [1.952 (3)–2.093 (4) Å], while the remaining N atoms of the terpy ligand cap the system at much longer distances [2.339 (3) Å].

Experimental

Salicylaldimine (Elerman *et al.*, 1991) (50 mg, 0.2 mmol) was stirred with copper(II) nitrate (45 mg, 0.25 mmol) and terpyridine (55 mg, 0.23 mmol) in methanol (20 ml). To this solution was added potassium carbonate (0.3 g) and the mixture was left without stirring overnight. The resulting almost black crystals, suitable for crystallography, were collected by filtration and washed with water, methanol and methylene chloride, yielding 4 mg of the complex (3%). IR (KBr, cm^{-1}): 1614 (*s*), 1600 (*m*), 1581 (*s*), 1464 (*s*), 1440 (*m*), 1297 (*s*), 831 (*m*), 741 (*s*), 538 (*s*).

Crystal data

[Cu($C_{13}H_9NO_2$)($C_{15}H_{11}N_3$)]
M_r = 508.02
Monoclinic, *C*2/*c*
a = 17.132 (3) Å
b = 13.918 (3) Å
c = 9.793 (2) Å
β = 102.87 (3)°
V = 2276.5 (8) Å^3
Z = 4

D_x = 1.482 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 20 reflections
θ = 1.5–22.5°
μ = 0.99 mm^{-1}
T = 293 (2) K
Rhomb, dark green
0.5 × 0.3 × 0.3 mm

Data collection

Siemens *P*4 diffractometer
ω scans
Absorption correction: ψ scan (*XSCANS*; Siemens, 1994)
$T_{\min}$ = 0.666, $T_{\max}$ = 0.743
2417 measured reflections
1997 independent reflections
1395 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.035
$\theta_{\max}$ = 25.0°
$h = -20 \rightarrow 1$
$k = -1 \rightarrow 16$
$l = -11 \rightarrow 11$
3 standard reflections every 47 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.105
S = 1.01
1997 reflections
171 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.7221P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.28 e Å^{-3}
$\Delta\rho_{\min}$ = −0.34 e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Cu1—O1	1.952 (3)	O1—C7	1.305 (4)
Cu1—N1	1.987 (5)	N1—C1	1.283 (7)
Cu1—N3	2.093 (4)	N1—C2	1.493 (6)
Cu1—N2	2.339 (3)		
O1—Cu1—N1$^{\rm i}$	101.73 (16)	N3—Cu1—N2	74.02 (8)
O1—Cu1—N1	75.75 (16)	N2—Cu1—N2$^{\rm i}$	148.03 (16)
O1—Cu1—N3	91.26 (9)	C7—O1—Cu1	118.6 (3)
O1—Cu1—N2	90.03 (11)	C7—C2—N1	100.4 (4)
N1—Cu1—N2	106.2 (2)		

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

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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