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(Nitrato-κO)(1,10-phenanthroline-$\kappa^2 N,N'$)-(picolinato *N*-oxide-$\kappa^2 O,O'$)copper(II)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.034
wR factor = 0.079
Data-to-parameter ratio = 14.2

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

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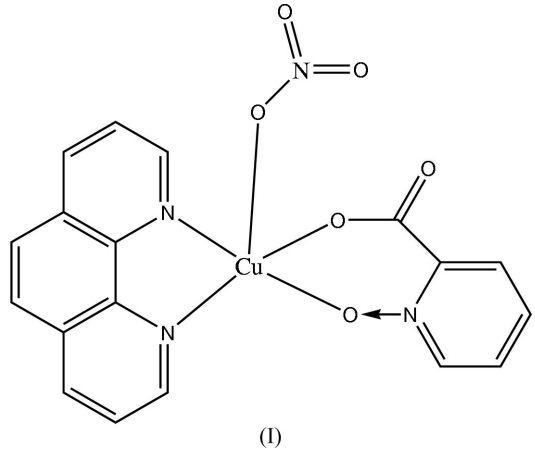
The coordination environment of copper(II) in the title compound, $[Cu(C_6H_4NO_3)(NO_3)(C_{12}H_8N_2)]$, is square pyramidal; the basal plane comprises the two N atoms of 1,10-phenanthroline and the two O atoms of picolinate *N*-oxide, with the apical position occupied by a nitrate O atom.

Comment

Picolinic acid *N*-oxide has a polar N→O group that provides a strongly basic O-atom site for coordination. Only a few complexes of this ligand have been crystallographically authenticated. In the binuclear compound La(6-mepic-NO)$_6$·6H$_2$O, two La^{III} atoms are bridged by two picolinate *N*-oxide groups (Yan *et al.*, 1995). In the Er^{III}–Na^{I} mixed-metal coordination polymer, the main feature is a polymeric chain of two zigzag chains (Mao *et al.*, 1998). The copper(II) nitrate derivative of the acid, (I), is a five-coordinate compound as the phenanthroline (phen) adduct. The Cu^{II} atom is coordinated by the two N atoms of a phen ligand and the two O atoms of a picolinate *N*-oxide anion, these atoms forming the basal plane; one O atom of the nitrate group occupies the apical position. The distances involving the Cu atom are comparable with those found in, for example, [Cu(phen)(ox)(H$_2$O)]·H$_2$O (Chen *et al.*, 2001) and [Cu(phen)(gly)(Cl)]·H$_2$O (Solans *et al.*, 1988). The Cu—O$_{nitrate}$ bond distance is significantly longer than the other Cu—O bond distances, since the O atom of the nitrate occupies the apical position. Both the heterocycle and the anion are planar. The Cu^{II} atom lies out of the basal plane by 0.1456 (5) Å in the direction of atom O4.



(I)

Experimental

To a solution of Cu(NO$_3$)$_2$·3H$_2$O (242 mg, 1 mmol) and 1,10-phenanthroline (180 mg, 1 mmol) in ethanol (20 ml) was added a solution of picolinic acid *N*-oxide (139 mg, 1 mmol) in tetrahydro-

furan (10 ml). The resulting solution was stirred for 4 h at room temperature. Crystals of (I) suitable for X-ray analysis were obtained after several days. Analysis found: C 48.56, H 2.57, N 12.86%; calculated for $C_{18}H_{12}CuN_4O_6$: C 48.71, H 2.73, N 12.62%.

Crystal data

$[Cu(C_6H_4NO_3)(NO_3)(C_{12}H_8N_2)]$	D_x = 1.797 Mg m^{-3}
M_r = 443.86	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 6799 reflections
a = 9.2861 (11) Å	θ = 3.1–27.4°
b = 9.5829 (12) Å	μ = 1.38 mm^{-1}
c = 18.438 (3) Å	T = 298 (2) K
β = 90.750 (4)°	Block, blue
V = 1640.7 (4) Å^3	0.38 × 0.24 × 0.16 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	3713 independent reflections
	2699 reflections with $I > 2\sigma(I)$
Oscillation scans	R_{int} = 0.033
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)	θ_{max} = 27.4°
	$h = -11 \rightarrow 12$
T_{min} = 0.678, T_{max} = 0.802	$k = -12 \rightarrow 12$
6799 measured reflections	$l = -23 \rightarrow 23$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$w = 1/[\sigma^2(F_o^2) + (0.0434P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.079	
S = 0.91	$(\Delta/\sigma)_{max}$ = 0.001
3713 reflections	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
262 parameters	$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.8962 (16)	Cu1—N2	2.0113 (19)
Cu1—O2	1.9018 (16)	Cu1—N3	2.0090 (19)
Cu1—O4	2.2839 (16)		
O1—Cu1—O2	93.78 (7)	O2—Cu1—N2	93.47 (7)
O1—Cu1—O4	104.56 (7)	O2—Cu1—N3	170.52 (7)
O1—Cu1—N2	165.37 (7)	O4—Cu1—N2	87.63 (7)
O1—Cu1—N3	88.42 (7)	O4—Cu1—N3	94.73 (7)
O2—Cu1—O4	93.64 (7)	N2—Cu1—N3	82.45 (7)

H atoms were placed at calculated positions and refined using a riding model, with C—H distances in the range 0.93–0.96 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$.

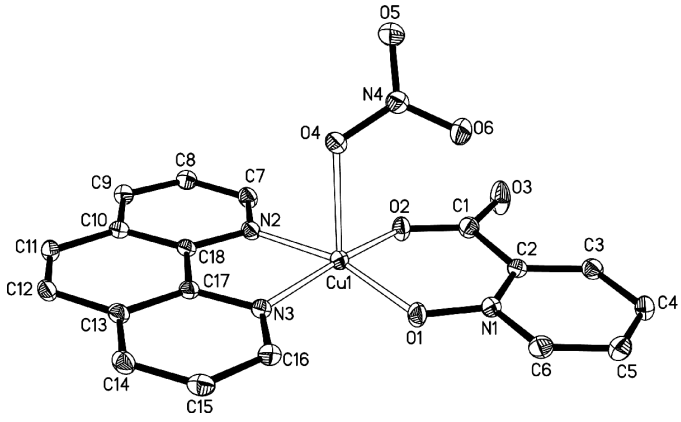


Figure 1
The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted.

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXTL* (Siemens, 1995).

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