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Redetermination of *catena*-poly-[[nitrato(1,10-phenanthroline)-copper(II)]-μ-nitrato]

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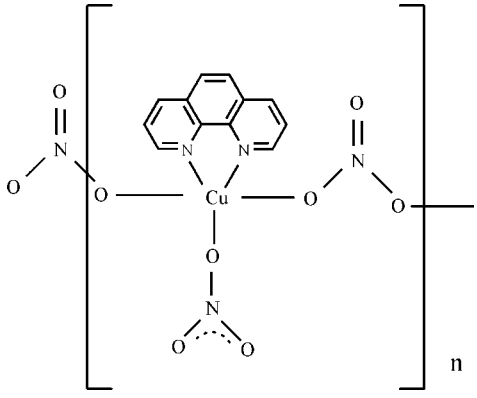
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.023; wR factor = 0.064; data-to-parameter ratio = 12.5.

The structure of the title copper coordination polymer, $[Cu(NO_3)_3(C_{10}H_8N_2)]_n$, previously reported by McFadden & McPhail [*J. Chem. Soc. Dalton Trans.* (1975), pp. 1993–1998], has been redetermined, leading to a more precise result. The crystal structure consists of a zigzag chain formed by the polymer $[Cu(NO_3)_3(phen)]_n$ (phen is 1,10-phenanthroline). The Cu^{II} atom has a slightly distorted square-pyramidal coordination environment consisting of two N atoms of the phen ligand, two O atoms of different nitrate ligands, and one O atom of a nitrate anion. The compound forms a one-dimensional chain using nitrate as an end-to-end bridging ligand.

Related literature

For related literature, see: McFadden & McPhail (1975).



Experimental

Crystal data

$[Cu(NO_3)_3(C_{10}H_8N_2)]$
M_r = 367.77
Monoclinic, $P2_1/n$
a = 8.7832 (15) Å
b = 9.1136 (16) Å
c = 17.173 (3) Å
β = 101.686 (2)°
V = 1346.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.66 mm^{-1}
T = 293 (2) K
0.40 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.607, T_{max} = 0.707
8361 measured reflections
2595 independent reflections
2071 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.064
S = 1.06
2595 reflections
208 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2223).

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  doi:10.1107/S1600536807037154

supplementary materials

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Redetermination of *catena*-poly[[nitrato(1,10-phenanthroline)copper(II)]-μ-nitrato]

X.-C. Zhou and Y.-Q. Liu

Comment

Each Cu^{II}ion is five-coordinated and possess a slightly distorted square-pyramidal coordination geometry, defined by two N atoms from one 1,10-phenanthroline ligand, two O atoms of two different bridging nitrate acid anion, and one O atom of a symetry related mono-dentate nitrate anion (Fig. 1). The compound forms a one-dimensional chain using nitrate anion as an end-to-end bridging ligand. The Cu—N bond lengths is almost equal, with Cu1—N1 being 1.998 (2) Å and Cu1—N2 being 2.004 (2) Å, respectively. The bond length between Cu1 and O atoms which set at the peak of the square-pyramidal are longer than between the O atoms which locates at the plane of square-pyramidal, the shorter bond lengths of Cu1—O1 and Cu1—O6 being 1.9556 (17) Å and 1.9979 (18) Å, the longer Cu—O bond length is 2.3410 (18) Å for Cu1—O4.

The packing is further stabilized by π_π stacking interactions involving the phenanthroline ring systems, the distance between the closest centroids being 3.66\%A with an interplanar distance of 3.60\%A.

Experimental

$Cu(NO_3)_2{\cdot}3H_2O$ (0.0242 g, 0.1 mmol) was dissolved in a small test tube (5 ml e thanol),then 1,10-phenanthroline (0.0199 g, 1 mmol) was added, NaOH (0.006 g, 0.15 mmol) was then added to the above solution. The whole mixture was sealed in a close vessel with 30 ml e ther. 4 days later, blue crystals of (I) were obtained.

Refinement

All H were fixed geometrically and treated as riding on their parent atoms with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C).

Figures

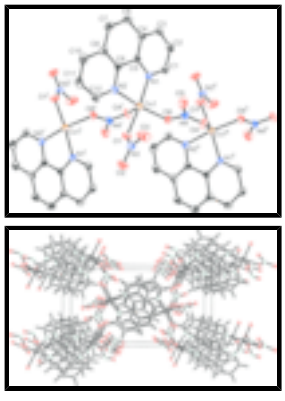


Fig. 1. View of complex (I) with the atom-labelling scheme showing the formation of the infinite chain. Ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry codes: (i) 3/2 − *x*, 1/2 + *y*, 3/2 − *z*; (ii) 3/2 − *x*, *y* − 1/2, 3/2 − *z*].

catena-poly[[nitrato(1,10-phenanthroline)copper(II)]-μ-nitrato]

Crystal data

$[Cu(NO_3)_3(C_{10}H_8N_2)]$	$Z = 4$
$M_r = 367.77$	$F_{000} = 740.0$
Monoclinic, $P2_1/n$	$D_x = 1.815$ Mg m^{-3}
Hall symbol: -P 2yn	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 8.7832$ (15) Å	$\mu = 1.66$ mm^{-1}
$b = 9.1136$ (16) Å	$T = 293$ (2) K
$c = 17.173$ (3) Å	Block, blue
$\beta = 101.686$ (2)º	0.40 × 0.25 × 0.20 mm
$V = 1346.2$ (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	2595 independent reflections
Radiation source: fine-focus sealed tube	2071 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.025$
$T = 293(2)$ K	$\theta_{max} = 26.0$º
φ and ω scans	$\theta_{min} = 2.4$º
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -10 \rightarrow 10$
$T_{min} = 0.607$, $T_{max} = 0.707$	$k = -11 \rightarrow 11$
8361 measured reflections	$l = -21 \rightarrow 21$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.024$	H-atom parameters constrained
$wR(F^2) = 0.064$	$w = 1[\sigma^2(F_o^2) + (0.0308P)^2 + 0.6118P]$ where $P = (F_o^2 + 2F_c^2)3$
$S = 1.06$	$(\Delta/\sigma)_{max} = 0.002$
2595 reflections	$\Delta\rho_{max} = 0.27$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.27$ 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	0.83503 (3)	0.42791 (3)	0.696821 (13)	0.03409 (9)
N1	0.70510 (18)	0.36714 (18)	0.59243 (9)	0.0336 (3)
N2	0.89172 (19)	0.59365 (17)	0.63144 (10)	0.0348 (4)
N3	1.0982 (2)	0.4773 (2)	0.81600 (10)	0.0411 (4)
N4	0.88410 (19)	0.12873 (19)	0.72182 (10)	0.0375 (4)
O1	0.95129 (17)	0.50491 (18)	0.79780 (8)	0.0459 (4)
O2	1.1715 (2)	0.5285 (2)	0.87827 (10)	0.0624 (5)
O3	1.1543 (2)	0.4000 (3)	0.77103 (11)	0.0764 (6)
O4	0.87904 (17)	0.00535 (16)	0.75191 (9)	0.0459 (4)
O5	0.9548 (2)	0.15217 (19)	0.66849 (11)	0.0620 (5)
O6	0.81196 (16)	0.23479 (15)	0.74821 (8)	0.0387 (3)
C1	0.6044 (2)	0.2574 (2)	0.57589 (12)	0.0409 (5)
H1	0.5843	0.1996	0.6173	0.049*
C2	0.5276 (3)	0.2260 (2)	0.49841 (13)	0.0447 (5)
H2	0.4563	0.1494	0.4889	0.054*
C3	0.5575 (2)	0.3078 (2)	0.43656 (12)	0.0415 (5)
H3	0.5078	0.2863	0.3847	0.050*
C4	0.6639 (2)	0.4248 (2)	0.45183 (11)	0.0356 (4)
C5	0.7326 (2)	0.4513 (2)	0.53139 (11)	0.0308 (4)
C6	0.7046 (3)	0.5186 (3)	0.39209 (12)	0.0439 (5)
H6	0.6634	0.5001	0.3387	0.053*
C7	0.8020 (3)	0.6335 (3)	0.41217 (12)	0.0443 (5)
H7	0.8270	0.6923	0.3723	0.053*
C8	0.8675 (2)	0.6664 (2)	0.49334 (12)	0.0372 (4)
C9	0.8347 (2)	0.5732 (2)	0.55247 (11)	0.0316 (4)
C10	0.9630 (3)	0.7881 (2)	0.51904 (14)	0.0469 (5)
H10	0.9897	0.8527	0.4821	0.056*
C11	1.0165 (3)	0.8110 (2)	0.59846 (14)	0.0492 (5)
H11	1.0774	0.8926	0.6159	0.059*
C12	0.9792 (2)	0.7110 (2)	0.65335 (13)	0.0430 (5)
H12	1.0171	0.7274	0.7072	0.052*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.04125 (15)	0.03153 (14)	0.02821 (13)	−0.00254 (10)	0.00398 (10)	−0.00099 (9)
N1	0.0400 (9)	0.0289 (8)	0.0312 (8)	−0.0026 (7)	0.0059 (7)	0.0014 (7)

N2	0.0376 (8)	0.0304 (8)	0.0365 (8)	−0.0013 (7)	0.0075 (7)	−0.0034 (7)
N3	0.0447 (10)	0.0394 (9)	0.0374 (9)	0.0016 (8)	0.0040 (8)	0.0007 (8)
N4	0.0375 (9)	0.0358 (9)	0.0382 (9)	−0.0020 (8)	0.0052 (7)	0.0025 (8)
O1	0.0427 (8)	0.0510 (9)	0.0400 (8)	0.0055 (7)	−0.0014 (6)	−0.0121 (7)
O2	0.0539 (10)	0.0664 (11)	0.0564 (10)	0.0050 (9)	−0.0136 (8)	−0.0195 (9)
O3	0.0592 (11)	0.1147 (17)	0.0551 (11)	0.0235 (11)	0.0110 (9)	−0.0262 (11)
O4	0.0466 (8)	0.0337 (8)	0.0578 (9)	0.0045 (7)	0.0120 (7)	0.0140 (7)
O5	0.0791 (12)	0.0495 (10)	0.0710 (11)	0.0040 (9)	0.0474 (10)	0.0071 (9)
O6	0.0498 (8)	0.0342 (7)	0.0333 (7)	0.0056 (6)	0.0110 (6)	0.0023 (6)
C1	0.0478 (11)	0.0329 (10)	0.0406 (11)	−0.0070 (9)	0.0059 (9)	0.0033 (9)
C2	0.0480 (12)	0.0345 (11)	0.0478 (12)	−0.0085 (9)	0.0006 (10)	−0.0035 (10)
C3	0.0483 (12)	0.0398 (11)	0.0336 (10)	0.0003 (10)	0.0016 (9)	−0.0060 (9)
C4	0.0406 (10)	0.0344 (10)	0.0320 (9)	0.0036 (9)	0.0078 (8)	−0.0020 (8)
C5	0.0345 (9)	0.0275 (9)	0.0311 (9)	0.0027 (8)	0.0085 (8)	0.0003 (7)
C6	0.0508 (12)	0.0518 (13)	0.0296 (10)	0.0008 (11)	0.0097 (9)	0.0013 (9)
C7	0.0515 (12)	0.0473 (12)	0.0374 (11)	0.0019 (11)	0.0168 (10)	0.0093 (10)
C8	0.0380 (10)	0.0352 (10)	0.0416 (10)	0.0028 (9)	0.0153 (9)	0.0033 (9)
C9	0.0333 (9)	0.0285 (9)	0.0342 (9)	0.0028 (8)	0.0097 (8)	−0.0014 (8)
C10	0.0501 (12)	0.0370 (12)	0.0572 (13)	−0.0063 (10)	0.0197 (11)	0.0067 (10)
C11	0.0481 (12)	0.0358 (12)	0.0638 (14)	−0.0119 (10)	0.0117 (11)	−0.0036 (11)
C12	0.0463 (11)	0.0367 (11)	0.0439 (11)	−0.0051 (9)	0.0042 (9)	−0.0064 (9)

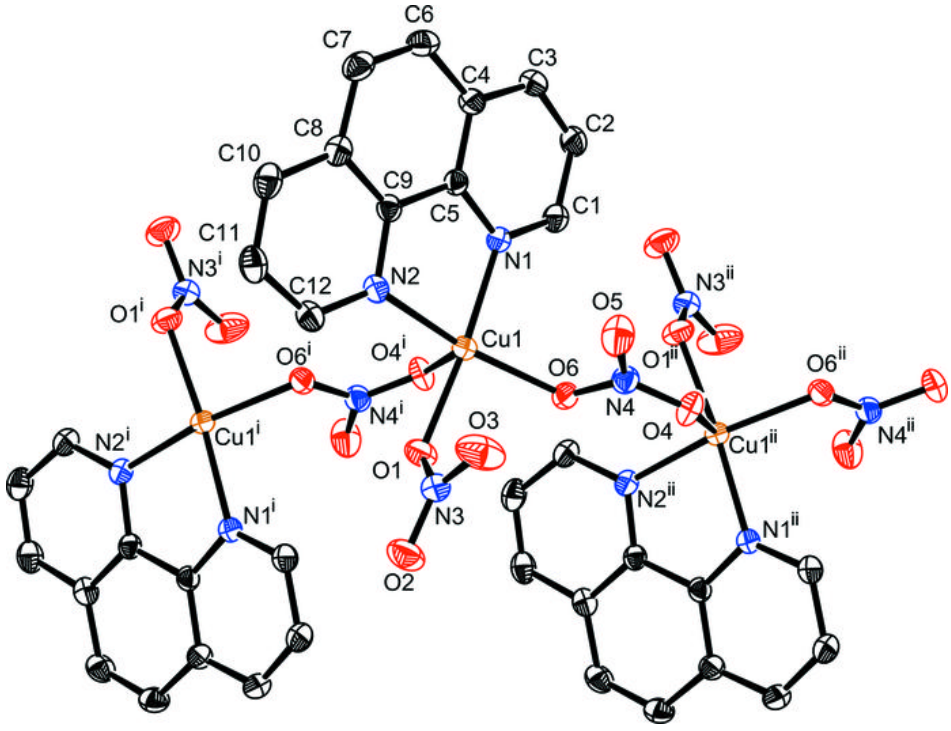
Geometric parameters (Å, °)

Cu1—O1	1.9558 (14)	C2—C3	1.366 (3)
Cu1—O6	1.9975 (14)	C2—H2	0.9300
Cu1—N1	1.9985 (16)	C3—C4	1.407 (3)
Cu1—N2	2.0042 (17)	C3—H3	0.9300
Cu1—O4^{i}	2.3407 (15)	C4—C5	1.398 (3)
N1—C1	1.327 (3)	C4—C6	1.435 (3)
N1—C5	1.360 (2)	C5—C9	1.428 (3)
N2—C12	1.326 (3)	C6—C7	1.352 (3)
N2—C9	1.360 (2)	C6—H6	0.9300
N3—O3	1.221 (2)	C7—C8	1.428 (3)
N3—O2	1.224 (2)	C7—H7	0.9300
N3—O1	1.289 (2)	C8—C9	1.398 (3)
N4—O5	1.225 (2)	C8—C10	1.407 (3)
N4—O4	1.242 (2)	C10—C11	1.366 (3)
N4—O6	1.287 (2)	C10—H10	0.9300
O4—Cu1^{ii}	2.3407 (15)	C11—C12	1.397 (3)
C1—C2	1.394 (3)	C11—H11	0.9300
C1—H1	0.9300	C12—H12	0.9300
O1—Cu1—O6	90.34 (6)	C2—C3—C4	119.57 (18)
O1—Cu1—N1	174.57 (6)	C2—C3—H3	120.2
O6—Cu1—N1	93.53 (6)	C4—C3—H3	120.2
O1—Cu1—N2	94.70 (7)	C5—C4—C3	116.87 (18)
O6—Cu1—N2	166.06 (6)	C5—C4—C6	118.35 (18)
N1—Cu1—N2	82.41 (7)	C3—C4—C6	124.78 (19)

O1—Cu1—O4^{i}	82.72 (6)	N1—C5—C4	123.17 (17)
O6—Cu1—O4^{i}	86.47 (6)	N1—C5—C9	116.37 (17)
N1—Cu1—O4^{i}	93.71 (6)	C4—C5—C9	120.45 (17)
N2—Cu1—O4^{i}	107.04 (6)	C7—C6—C4	121.03 (19)
C1—N1—C5	118.36 (17)	C7—C6—H6	119.5
C1—N1—Cu1	129.46 (14)	C4—C6—H6	119.5
C5—N1—Cu1	112.15 (12)	C6—C7—C8	121.37 (19)
C12—N2—C9	117.93 (17)	C6—C7—H7	119.3
C12—N2—Cu1	130.12 (14)	C8—C7—H7	119.3
C9—N2—Cu1	111.86 (12)	C9—C8—C10	116.66 (19)
O3—N3—O2	124.37 (19)	C9—C8—C7	118.70 (19)
O3—N3—O1	118.15 (18)	C10—C8—C7	124.64 (19)
O2—N3—O1	117.47 (18)	N2—C9—C8	123.56 (18)
O5—N4—O4	122.36 (18)	N2—C9—C5	116.42 (17)
O5—N4—O6	119.50 (17)	C8—C9—C5	119.99 (18)
O4—N4—O6	118.14 (16)	C11—C10—C8	119.8 (2)
N3—O1—Cu1	117.52 (12)	C11—C10—H10	120.1
N4—O4—Cu1^{ii}	121.92 (12)	C8—C10—H10	120.1
N4—O6—Cu1	113.88 (11)	C10—C11—C12	119.6 (2)
N1—C1—C2	122.10 (19)	C10—C11—H11	120.2
N1—C1—H1	119.0	C12—C11—H11	120.2
C2—C1—H1	119.0	N2—C12—C11	122.4 (2)
C3—C2—C1	119.9 (2)	N2—C12—H12	118.8
C3—C2—H2	120.1	C11—C12—H12	118.8
C1—C2—H2	120.1		

Symmetry codes: (i) −*x*+3/2, *y*+1/2, −*z*+3/2; (ii) −*x*+3/2, *y*−1/2, −*z*+3/2.

Fig. 1


C6
C7
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C3
C8
C2
C10
C9
C5
C1
C11
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N1
C12
N2
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O5
N3ii
O6i
O4i
Cu1
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O1ii
O6ii
N4i
N4
O4
N2i
Cu1i
O3
Cu1ii
N4ii
O1
N2ii
N3
N1i
N1ii
O2

Fig. 2