Tetragonal
$P4_12_12$
$a = 6.7940$ (5) Å
$c = 11.9557$ (11) Å
$V = 551.86$ (8) Å^3
$Z = 4$
$D_x = 2.306$ Mg m^{-3}
$D_m = 2.2$ Mg m^{-3}
D_m measured by pycnometry

Cell parameters from 616 reflections
$\theta = 1.65$–$26.05°$
$\mu = 3.892$ mm^{-1}
$T = 293$ (2) K
Bipyramidal
0.50 × 0.45 × 0.40 mm
Dark blue

Data collection

Stoe Imaging Plate Diffractometer System (IPDS)
Rotation scans
Absorption correction: numerical (*FACEIT, X-SHAPE* and *X-RED*; Stoe & Cie, 1995)
$T_{\min} = 0.163$, $T_{\max} = 0.211$

1070 measured reflections
358 independent reflections (plus 183 Friedel-related reflections)
522 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\max} = 25.99°$
$h = -5 \rightarrow 5$
$k = 0 \rightarrow 8$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.018$
$wR(F^2) = 0.048$
$S = 1.095$
541 reflections
48 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0330P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.362$ e Å^{-3}
$\Delta\rho_{\min} = -0.259$ e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.067 (4)
Scattering factors from *International Tables for Crystallography* (Vol. C)
Absolute structure: Flack (1983)
Flack parameter = −0.02 (3)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU^{ij}a^ia^j\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{cq}
Cu1	0.23094 (4)	0.23094 (4)	0	0.01612 (18)
O1	0.4727 (2)	0.2598 (3)	0.08576 (13)	0.0224 (4)
O2	0.2995 (3)	0.4824 (3)	0.17479 (13)	0.0244 (4)
C1	0.4541 (3)	0.3782 (3)	0.16531 (18)	0.0170 (5)
C2	0.8164 (4)	0.3071 (4)	0.2131 (2)	0.0264 (6)
C3	0.6144 (4)	0.3856 (4)	1/4	0.0171 (6)

Table 2. *Selected geometric parameters (Å, °)*

Cu1—O2$^{\rm i}$	1.9243 (17)	Cu1—O1	1.9464 (16)
O2$^{\rm i}$—Cu1—O2$^{\rm ii}$	91.71 (11)	O1—Cu1—O1$^{\rm iii}$	96.18 (11)

Symmetry codes: (i) $y - \frac{1}{2}, \frac{1}{2} - x, z - \frac{1}{4}$; (ii) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{4} - z$; (iii) $y, x, -z$.

Two enantiomorphic space groups, $P4_12_12$ or $P4_32_12$, are possible for the investigated crystal. In order to determine the correct absolute structure, refinement was performed in both space groups. The Flack parameter (Flack, 1983) is helpful in solving the problem. It is almost zero [−0.02 (3)] for $P4_12_12$ and 1.00 (5) for $P4_32_12$, so it turns out that the first space group is correct.

Data collection: *IPDS Software* (Stoe & Cie, 1995). Cell refinement: *IPDS Software*. Data reduction: *IPDS Software*. Program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*). Molecular graphics: *PLATON*92 (Spek, 1992). Software used to prepare material for publication: *PLATON*92.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1210). Services for accessing these data are described at the back of the journal.

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Tetraaqua(1,10-phenanthroline-*N*,*N*)-nickel(II) dinitrate monohydrate

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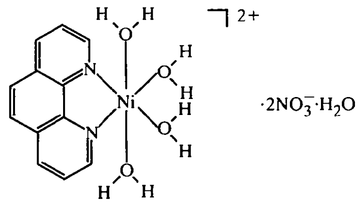
Abstract

The title compound, [Ni($C_{12}H_8N_2$)(H_2O)$_4$](NO_3)$_2$·H_2O, consists of tetraaqua(1,10-phenanthroline)nickel(II) cations, nitrate anions and water molecules. The Ni$^{\rm II}$ ion is coordinated in a distorted octahedral arrangement by two water molecules and two tertiary N atoms from one phenanthroline ligand in equatorial positions, and by two O atoms of water in axial positions. The Ni—O bond lengths range from 2.053 (2) to 2.105 (2) Å and the two Ni—N bond lengths are 2.068 (2) and 2.070 (2) Å,

while the N1—Ni—N2 chelate angle is 80.83 (8)°. The complex forms a three-dimensional hydrogen-bonded framework.

Comment

In the course of our work on the synthesis of Ni complexes containing N-donor ligands, the title salt, (I), was obtained. This compound has been prepared previously by another route (Walmsley *et al.*, 1989); the structure was determined, but not fully reported. It consists of $[Ni(C_{12}H_8N_2)(H_2O)_4]^{2+}$ cations, NO_3^- anions and water molecules. The coordination polyhedron around the metal atom is distorted octahedral. The two ligand N atoms and two aqua O atoms define the basal plane and the remaining two O atoms are in axial positions (Fig. 1). The molecules are stacked in layers at $x = \frac{1}{2}$ parallel to (100). Hydrogen bonding (Table 2) links the cation to nitrate anions and water molecules to give a three-dimensional framework. The $[Ni(C_{12}H_8N_2)(H_2O)_4]^{2+}$ octahedron is distorted (Table 1). The angles at nickel are similar to those found in other complexes (Werner *et al.*, 1996; Mukherjee *et al.*, 1995). Furthermore, the N1—Ni—N2 chelate angle in (I) [80.83 (8)°] is consistent with that in $[Ni(C_2H_5OCS_2)_2(C_{11}H_6N_2O)]$ [81.97 (7)°], $[Ni(EtXA)(2,2'\text{-dipyridylamine})]\cdot C_6H_6$ [87.61 (9)°; EtXA is ethyl xanthate] and *cis*-[Ni(EtXA)$_2$(2,9-dimethyl-1,10-phenanthroline)] [78.3 (1)°] (Xiong *et al.*, 1996; Gable *et al.*, 1985; Guo *et al.*, 1996). The Ni—O distances vary from 2.053 (2) to 2.105 (2) Å. These distances are in good agreement with those of other nickel complexes (Cisarova & Zachova, 1996; Tahirov *et al.*, 1994; Berraho *et al.*, 1992; Cotton *et al.*, 1993; Lumme *et al.*, 1996). The values for the Ni—N bond lengths, 2.068 (2) and 2.070 (2) Å, lie in the normal range for nickel(II) complexes (Li *et al.*, 1996; Cisarova & Zachova, 1996; Lumme *et al.*, 1996; Fun *et al.*, 1996; Decurtins *et al.*, 1996; Guo *et al.*, 1996; Werner *et al.*, 1996; Mukherjee *et al.*, 1995). The *o*-phenanthroline rings are approximately planar and lie essentially in the basal coordination plane. The dihedral angle between the two rings is 1.6 (1)°. The bond lengths in the *o*-phenanthroline ligand and nitrate anions are normal. Two NO_3^- ions are located beside the cationic group and are connected to the complex and a water molecule by hydrogen bonds. The related compound $[Ni(bipy)(H_2O)_3(NO_3)]NO_3$ (bipy is 2,2'-bipyridyl) has one nitrate anion coordinated to Ni as a monodentate ligand (Walmsley *et al.*, 1989). The crystal structure contains narrow channels parallel to the *b* axis, with a diameter of only 2.4 Å (*PLATON*; van der Sluis & Spek, 1990).



(I)

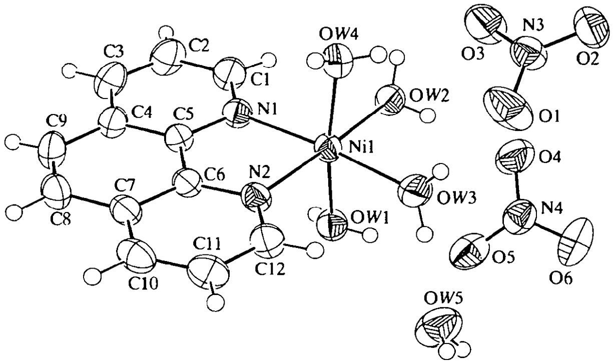


Fig. 1. The structure of (I) with 50% probability ellipsoids. H atoms are shown as spheres of arbitrary radii.

Experimental

The title compound was synthesized by mixing nickel nitrate hexahydrate (1.46 g, 5 mmol), 1,10-phenanthroline hydrate (1.0 g, 5 mmol) and phosphoric acid (0.98 g, 10 mmol) in an ethanol–water solution. The resulting solution was refluxed, after which most of the solvent was removed. After cooling to room temperature, the solution was filtered and the filtrate allowed to evaporate for several weeks, whereupon light-blue crystals of the title compound were obtained.

Crystal data

$[Ni(C_{12}H_8N_2)(H_2O)_4]$-$(NO_3)_2\cdot H_2O$
$M_r = 453.01$
Monoclinic
$P2_1/c$
$a = 8.783$ (1) Å
$b = 13.629$ (2) Å
$c = 15.411$ (2) Å
$\beta = 94.71$ (1)°
$V = 1838.6$ (5) Å^3
$Z = 4$
$D_x = 1.637$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10.77$–15.82°
$\mu = 1.122$ mm^{-1}
$T = 293$ (2) K
Cubic
0.37 × 0.22 × 0.22 mm
Blue

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical ψ scan (North *et al.*, 1968)
$T_{min} = 0.718$, $T_{max} = 0.781$
3740 measured reflections
3602 independent reflections

2852 reflections with $I > 2\sigma(I)$
$R_{int} = 0.011$
$\theta_{max} = 25.96$°
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 18$
2 standard reflections
frequency: 120 min
intensity decay: 0.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.088$
$S = 1.053$
3602 reflections
326 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0343P)^2 + 1.0044P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.283$ e Å^{-3}
$\Delta\rho_{\min} = -0.242$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0009 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Ni1—O*W*3	2.053 (2)	Ni1—N2	2.070 (2)
Ni1—O*W*2	2.059 (2)	Ni1—O*W*4	2.103 (2)
Ni1—N1	2.068 (2)	Ni1—O*W*1	2.105 (2)
O*W*3—Ni1—O*W*2	90.11 (9)	N1—Ni1—O*W*4	92.12 (9)
O*W*3—Ni1—N1	173.99 (9)	N2—Ni1—O*W*4	92.81 (9)
O*W*2—Ni1—N1	95.81 (8)	O*W*3—Ni1—O*W*1	87.3 (1)
O*W*3—Ni1—N2	93.25 (9)	O*W*2—Ni1—O*W*1	85.04 (9)
O*W*2—Ni1—N2	176.58 (8)	N1—Ni1—O*W*1	92.19 (9)
N1—Ni1—N2	80.83 (8)	N2—Ni1—O*W*1	94.48 (8)
O*W*3—Ni1—O*W*4	89.1 (1)	O*W*4—Ni1—O*W*1	172.04 (9)
O*W*2—Ni1—O*W*4	87.9 (1)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O*W*1—H1*W*1···O5	0.80 (3)	2.03 (3)	2.822 (3)	171 (3)
O*W*1—H2*W*1···O2^{i}	0.76 (4)	2.15 (4)	2.896 (3)	166 (4)
O*W*2—H1*W*2···O4	0.82 (3)	1.96 (4)	2.785 (3)	177 (3)
O*W*2—H2*W*2···O3^{ii}	0.88 (4)	1.95 (4)	2.799 (3)	160 (4)
O*W*3—H1*W*3···O1	0.76 (4)	2.00 (4)	2.764 (3)	173 (4)
O*W*3—H2*W*3···O*W*5	0.87 (4)	1.82 (4)	2.684 (4)	176 (4)
O*W*4—H1*W*4···O6^{iii}	0.84 (4)	2.01 (4)	2.847 (4)	175 (3)
O*W*4—H2*W*4···O3	0.78 (3)	2.08 (3)	2.833 (3)	164 (3)
O*W*5—H1*W*5···O4^{iv}	0.86 (4)	1.97 (4)	2.830 (4)	179 (3)
O*W*5—H2*W*5···O*W*4^{v}	0.81 (5)	2.28 (5)	3.062 (4)	161 (4)

Symmetry codes: (i) $x-1, y, z$; (ii) $1-x, -y, -z$; (iii) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (iv) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (v) $x, \frac{1}{2}-y, \frac{1}{2}+z$.

Data collection: *CAD*-4 *EXPRESS* (Duisenberg, 1992; Macíček & Yordanov, 1992; Enraf–Nonius, 1994). Cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ZORTEP* (Zsolnai, 1994). Software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1300). Services for accessing these data are described at the back of the journal.

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A selenium-bridged mixed-metal cluster, (CO)$_5$(PPh$_3$)$_2$FePt$_2$(μ_3-Se)

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Abstract

Stirring at room temperature of [(CO)$_6$Fe$_2${μ-SeC(H)=C(CH$_2$OH)Se}] with Pt(PPh$_3$)$_4$ or Pt(C$_2$H$_4$)(PPh$_3$)$_2$ in dichloromethane affords the μ_3-Se-bridged mixed-metal cluster pentacarbonyl-1κ^3C,2κC,3κC-μ_3-selenido-1:2:3κ^3*Se*-bis(triphenylphosphine)-2κP,3κP-irondiplatinum(2 *Fe—Pt*), [FePt$_2$(μ_3-Se)(C$_{18}$H$_{15}$P)$_2$(CO)$_5$]. The structure can be described as an FePt$_2$Se butterfly; each Pt atom is coordinated by one CO and one PPh$_3$ group,

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