Tris(phenanthroline-$\kappa^2 N,N'$)nickel(II) bis(malate) tetrahydrate

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The crystal structure of the title complex, [Ni($C_{12}H_8N_2$)$_3$]($C_4H_5O_5$)$_2 \cdot 4H_2O$, consists of Ni^{II} complex cations, malate anions and solvent water molecules. The Ni^{II} ion is located on a twofold axis and chelated by three phenanthroline (phen) with a distorted octahedral geometry.

Comment

π–π Stacking plays an important role in some biological processes. The title new Ni^{II} complex with 1,10-phenanthroline (phen) ligands, (I), is presented here.

(I)
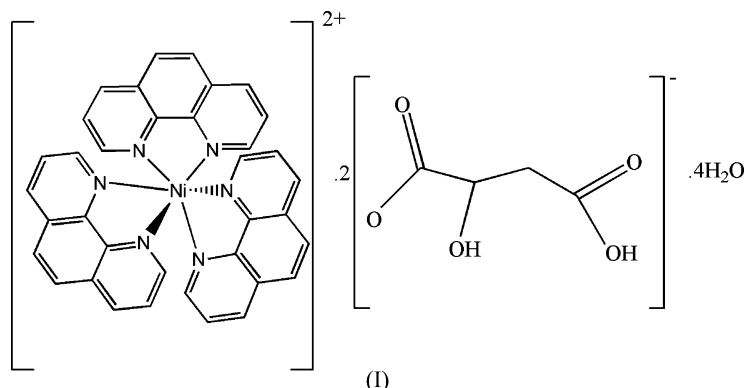
The crystal structure of (I) consists of Ni^{II} complex cations, malate monoanions and solvent water molecules (Fig. 1). The Ni^{II} ion is located on a twofold axis and is chelated by three phen ligands in a distorted octahedral geometry (Table 1). The malate monoanions link the Ni^{II} complex cations via weak C—H···O hydrogen bonds (Table 2). Solvent water molecules and malate anions are linked together by O—H···O hydrogen bonds, which stabilize the crystal structure of (I).

Experimental

An aqueous solution (5 ml) of NiCl$_2 \cdot 6H_2O$ (1 mmol) was mixed with an aqueous solution (5 ml) containing malic acid (2 mmol) and Na$_2$CO$_3$ (2 mmol). The solution was refluxed for 30 min, then phen (2 mmol) was added. The mixture was refluxed for a further 30 min and filtered. Green single crystals of (I) were obtained from the filtrate after 6 d.

Crystal data

[Cu($C_{12}H_8N_2$)$_3$]($C_4H_5O_5$)$_2 \cdot 4H_2O$

$M_r = 937.55$

Orthorhombic, $Pcca$

$a = 19.7761\ (9)$ Å

$b = 14.4074\ (5)$ Å

$c = 14.8877\ (6)$ Å

$V = 4241.8\ (3)$ Å^3

$Z = 4$

$D_x = 1.468$ Mg m^{-3}

Mo $K\alpha$ radiation

$\mu = 0.54$ mm^{-1}

$T = 273\ (2)$ K

Prism, green

$0.40 \times 0.20 \times 0.16$ mm

Data collection

Bruker SMART CCD area-detector diffractometer	18394 measured reflections
φ and ω scans	5194 independent reflections
Absorption correction: multi-scan	2617 reflections with $I > 2I$
(*SADABS*; Sheldrick, 1996)	$R_{int} = 0.061$
$T_{min} = 0.817$, $T_{max} = 0.918$	$\theta_{max} = 28.1°$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.078P)^2]$
$wR(F^2) = 0.141$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.88$	$(\Delta/\sigma)_{max} = 0.001$
5194 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
294 parameters	$\Delta\rho_{min} = -0.36$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—N1	2.107 (2)	Ni1—N3	2.080 (2)
Ni1—N2	2.095 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$A\cdots$O5^i	0.85	1.69	2.528 (3)	171
O3—H3$A\cdots$O5	0.92	1.93	2.583 (3)	126
O6—H6$A\cdots$O7	0.83	1.97	2.790 (9)	168
O6—H6$B\cdots$O4	0.86	1.81	2.673 (6)	176
C8—H8$\cdots$O5^{ii}	0.93	2.49	3.415 (4)	177
C13—H13$\cdots$O1^{iii}	0.93	2.50	3.208 (4)	133
C14—H14$\cdots$O4^{iv}	0.93	2.58	3.301 (4)	135
C18—H18$\cdots$O2^v	0.93	2.43	3.327 (4)	162

Symmetry codes: (i) $-x + \frac{3}{2}, y, z + \frac{1}{2}$; (ii) $-x + 1, -y + 2, -z + 1$; (iii) $-x + 1, y, -z + \frac{3}{2}$; (iv) $x - \frac{1}{2}, y, -z + 1$; (v) $x - \frac{1}{2}, -y + 1, -z + \frac{3}{2}$.

H atoms on O1, O3 and O6 were located in a difference Fourier map and H atoms on O7 were placed in calculated positions (O—H = 0.85 Å; Nardelli, 1999). H atoms on O atoms were refined as riding in as-found relative positions, with $U_{iso}(H) = 1.5U_{eq}(O)$. Other H atoms were placed in calculated positions, with C—H = 0.93 (aromatic), 0.97 (methylene) or 0.98 Å (methine), and refined in riding mode, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve
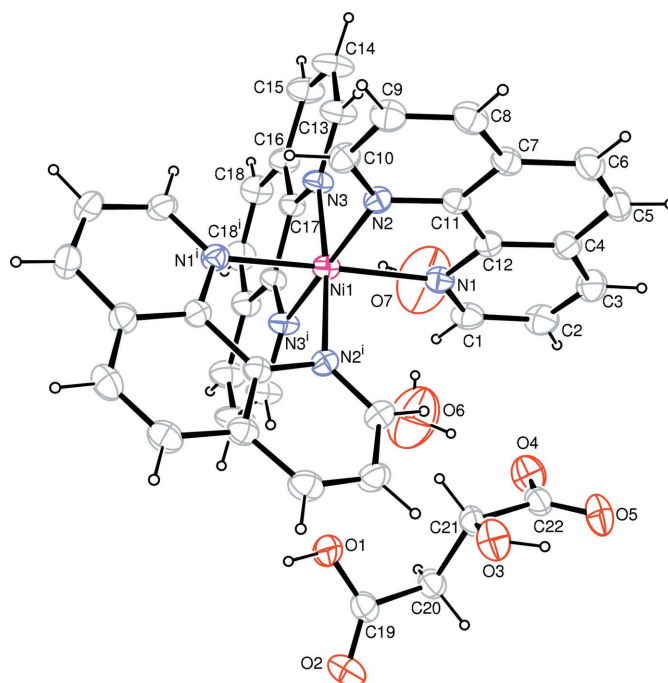

Figure 1
The molecular structure of (I), shown with 30% probability displacement ellipsoids (arbitrary spheres for H atoms) [symmetry code: (i) $1 - x, y, \frac{3}{2} - z$].

structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

References

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