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Key indicators

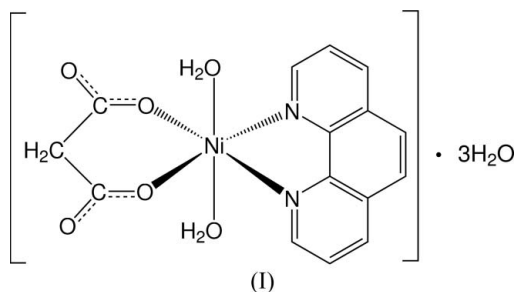
Single-crystal X-ray study
 $T = 295\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
Disorder in main residue
 R factor = 0.025
 wR factor = 0.076
Data-to-parameter ratio = 16.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.*trans*-Diaqua(malonato)(1,10-phenanthroline)-
nickel(II) trihydrate

The title complex, $[\text{Ni}(\text{C}_3\text{H}_2\text{O}_4)(\text{C}_{12}\text{H}_8\text{N}_2)(\text{H}_2\text{O})_2]\cdot 3\text{H}_2\text{O}$, displays a distorted octahedral NiN_2O_4 coordination geometry, formed by a malonate dianion, a 1,10-phenanthroline ligand and two *trans* water molecules. The Ni atom and one O atom, the latter from a solvent water molecule, are located on twofold axes of symmetry. The crystal structure is stabilized by extensive $\text{O}-\text{H}\cdots\text{O}$ hydrogen-bonding interactions.

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Comment

The malonate anion (mal) displays a variety of coordination modes owing to the presence of two carboxylate groups in the 1- and 3-positions (Catalina *et al.*, 2000*a,b*; Yolanda, Catalina *et al.*, 2001; Yolanda, Joaquín *et al.*, 2001; Yilmaz *et al.*, 2004). The analogous *cis*- Ni^{II} complex containing malonate and 1,10-phenanthroline (phen) has been reported (Wang *et al.*, 2004). In this paper, the title Ni^{II} complex, (I), with a *trans* configuration, has been prepared and its crystal structure determined.



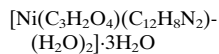
The crystal structure of (I) contains the $[\text{Ni}(\text{mal})(\text{phen})(\text{H}_2\text{O})_2]$ complex and uncoordinated water molecules, in a 1:3 ratio, as shown in Fig. 1. The Ni atom is located on a twofold axis, as is one of the water molecules, *viz.* that containing the O5 atom. The Ni^{II} centre is six-coordinated by two carboxylate O atoms from the malonate dianion, two N atoms of the phen ligand, and two O atoms of the coordinated water molecules. Both the dianion and neutral ligands chelate the Ni centre; the water molecules occupy *trans* positions in the distorted octahedral geometry. The Ni—O and Ni—N bond lengths (Table 1) are almost equal to those of the aforementioned *cis*- Ni^{II} complex (Wang *et al.*, 2004).

The crystal structure of (I) appears to be tightly consolidated by extensive hydrogen bonds, as detailed in Table 2. Neighbouring complexes are connected to each other *via* $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds. Atom H5 is involved in a bifurcated $\text{O}-\text{H}\cdots(\text{O},\text{O})$ hydrogen bond; the sum of the angles about this atom is 359° .

Experimental

Nickel chloride hexahydrate (0.0475 g, 0.2 mmol) and malonic acid (0.0208 g, 0.2 mmol) were dissolved in water (20 ml). The resulting solution was added to an absolute ethanol solution (10 ml) containing 1,10-phenanthroline (0.0396 g, 0.2 mmol), and stirred for 5 h until a small amount of precipitate appeared. This solution was filtered and the filtrate was set aside at room temperature. After three weeks, pale-blue single crystals were obtained.

Crystal data



$M_r = 431.04$

Monoclinic, $P2_1/n$

$a = 8.9389$ (3) Å

$b = 12.1466$ (4) Å

$c = 9.4334$ (3) Å

$\beta = 116.189$ (1)°

$V = 919.11$ (5) Å³

$Z = 2$

$D_x = 1.558$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 4010

reflections

$\theta = 2.5\text{--}27.4^\circ$

$\mu = 1.11$ mm⁻¹

$T = 295$ (1) K

Block, pale blue

$0.40 \times 0.37 \times 0.30$ mm

Data collection

Rigaku R-Axis RAPID
diffractometer

ω scans

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.632$, $T_{\max} = 0.717$

4010 measured reflections

2097 independent reflections
1893 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

$\theta_{\text{max}} = 27.4^\circ$

$h = -11 \rightarrow 11$

$k = -15 \rightarrow 15$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.025$

$wR(F^2) = 0.076$

$S = 1.09$

2097 reflections

129 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.2459P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.34$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

Extinction correction: *SHELXL97*

Extinction coefficient: 0.054 (3)

Table 1

Selected geometric parameters (Å, °).

Ni—O1	2.0042 (12)	Ni—N1	2.0714 (13)
Ni—O3	2.1111 (12)		
O1—Ni—O3	90.58 (5)	O3—Ni—O3 ⁱ	177.11 (6)
O1—Ni—N1	93.65 (5)	O3—Ni—N1 ⁱ	90.69 (5)
O1—Ni—O1 ⁱ	92.35 (7)	N1—Ni—O3	87.10 (5)
O1—Ni—N1 ⁱ	173.83 (5)	N1—Ni—N1 ⁱ	80.38 (7)
O1—Ni—O3 ⁱ	91.42 (5)		

Symmetry code: (i) $-x + \frac{3}{2}, y, -z + \frac{3}{2}$.

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O3—H31 \cdots O2 ⁱⁱ	0.81	1.98	2.7580 (18)	162
O3—H32 \cdots O4	0.91	1.85	2.748 (2)	171
O4—H41 \cdots O2 ⁱⁱⁱ	0.93	1.97	2.897 (2)	177
O4—H42 \cdots O5	0.95	1.88	2.824 (2)	175
O5—H5 \cdots O2 ^{iv}	0.88	1.97	2.846 (2)	173
O5—H5 \cdots O1 ^{iv}	0.88	2.54	3.1637 (15)	129

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

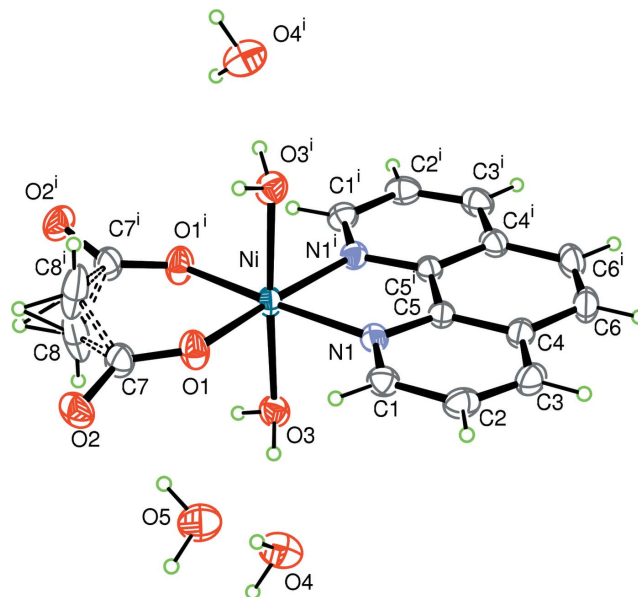


Figure 1

The molecular structure of (I), shown with 40% probability displacement ellipsoids. Both sites of the disordered C8 atom are shown. [Symmetry code: (i) $1.5 - x, y, 1.5 - z$.]

Aromatic H atoms were placed in calculated positions, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The H atoms of the water molecules were located in difference Fourier maps and made to ride on the O atoms in their as-found relative positions, $U_{\text{iso}}(\text{H}) = 0.05$ Å². Atom C8 of the malonate ligand was found to be disordered over two positions about the twofold axis. This atom was refined anisotropically, with 0.50 site-occupancy factors, and the H atoms were idealized and made to ride with C—H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C8})$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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