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Diaquabis(malato-$\kappa^2 O,O'$)nickel(II)

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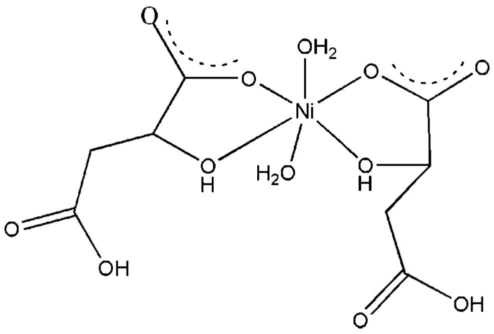
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.021; wR factor = 0.061; data-to-parameter ratio = 12.1.

In the title compound, $[Ni(C_6H_5O_5)_2(H_2O)_2]$, the Ni^{II} atom, located on an inversion centre, is coordinated by four O atoms from two malate ligands and two water molecules in an octahedral geometry showing a very large axial distortion. The packing is governed by intermolecular O—H$\cdots$O hydrogen bonds.

Related literature

For background, see: Kotsakis *et al.* (2003).



Experimental

Crystal data

$[Ni(C_6H_5O_5)_2(H_2O)_2]$
M_r = 360.90
Monoclinic, $P2_1/c$
a = 8.4762 (5) Å
b = 7.4377 (4) Å
c = 10.3117 (6) Å
β = 102.680 (1)°
V = 634.23 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.60 mm^{-1}
T = 298 (2) K
0.28 × 0.25 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.664, T_{max} = 0.762
3133 measured reflections
1171 independent reflections
1018 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.061
S = 1.02
1171 reflections
97 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni1—O3	1.9134 (12)	Ni1—O1*W*	2.5151 (13)
Ni1—O1	1.9509 (12)		

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
O1—H1$\cdots$O2^{i}	0.86	1.77	2.6245 (17)	172
O1*W*—H1*W*$\cdots$O2^{ii}	0.81	2.05	2.8374 (19)	163
O1*W*—H2*W*$\cdots$O4^{iii}	0.81	2.08	2.843 (2)	156
O5—H5$\cdots$O1*W*^{iv}	0.82	1.87	2.6835 (19)	171

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2529).

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supplementary materials

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Diaquabis(malato-κ^2*O*,*O*')nickel(II)

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Comment

Some hydroxypolycarboxylic acids are present in fruits and living cells and they also play an important role in biological processes (Kotsakis *et al.*, 2003). Hydroxypolycarboxylic acids can act not only as hydrogen-bond acceptors but also as hydrogen-bond donors, depending on the number of deprotonated carboxyl group.

In this paper, we report the synthesis and crystal structure of the title compound, (I). The Ni^{II} atom, located on an inversion center, is coordinated by four O atoms from two malate ligands and two water molecules in an axially distorted octahedral geometry (Fig. 1, Table 1).

Intermolecular O—H···O hydrogen bonds (Table 2) help to consolidate the crystal packing.

Experimental

Malic acid (0.15 g, 1.01 mmol) and $NiCl_2 \cdot 6H_2O$ (0.028 g, 0.12 mmol), were added to a mixed solvent system of methanol and acetonitrile. The mixture was heated for six hours under reflux at 389 K with stirring. The resultant solution was filtered and placed in a closed container, into which diethyl ether was allowed to infuse. After a week, green blocks of (I) were recovered.

Refinement

The water H atoms were located in a difference Fourier map and were refined as riding in their as-found relative positions with $U_{iso}(H) = 1.2U_{eq}(O)$. The other H atoms were placed in calculated positions (C—H = 0.93–0.97 Å, O—H = 0.82–0.86 Å) and refined using a riding model, with $U_{iso}(H) = 1.2U_{eq}$(carrier). The maximum difference peak is 1.12Å from C3.

Figures

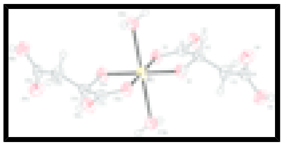


Fig. 1. The molecular structure of (I). Non-H atoms are shown as 50% probability displacement ellipsoids. Atoms marked with a ' are generated by the symmetry operation (−*x*, −*y*, −*z*).

Diaquabis(malato-κ^2*O*,*O*')nickel(II)

Crystal data

$[Ni(C_6H_5O_5)_2(H_2O)_2]$	$F_{000} = 372$
$M_r = 360.90$	$D_x = 1.890$ Mg m^{-3}

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 8.4762$ (5) Å

$b = 7.4377$ (4) Å

$c = 10.3117$ (6) Å

$\beta = 102.680$ (1)°

$V = 634.23$ (6) Å^3

$Z = 2$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 1171 reflections

$\theta = 2.5$–25.5°

$\mu = 1.60$ mm^{-1}

$T = 298$ (2) K

Block, green

0.28 × 0.25 × 0.18 mm

Data collection

Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2)$ K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.664$, $T_{\max} = 0.762$

3133 measured reflections

1171 independent reflections

1018 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.016$

$\theta_{\max} = 25.5$°

$\theta_{\min} = 2.5$°

$h = -10 \rightarrow 5$

$k = -8 \rightarrow 9$

$l = -11 \rightarrow 12$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.021$

$wR(F^2) = 0.061$

$S = 1.02$

1171 reflections

97 parameters

3 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: difmap and geom

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.0527P]$
where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.24$ e Å^{-3}

$\Delta\rho_{\min} = -0.20$ e Å^{-3}

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}
Ni1	0.0000	0.0000	0.0000	0.02590 (14)
O1	0.16156 (16)	0.12408 (17)	0.13402 (12)	0.0315 (3)
H1	0.1591	0.1089	0.2162	0.047*
O1W	−0.22841 (16)	0.18371 (18)	0.05033 (13)	0.0382 (3)
H1W	−0.2138	0.2826	0.0843	0.057*
H2W	−0.2735	0.1242	0.0974	0.057*
O2	0.1701 (2)	0.44803 (18)	−0.11539 (13)	0.0406 (4)
O3	0.03754 (16)	0.19406 (17)	−0.11125 (12)	0.0329 (3)
O4	0.39452 (18)	0.4001 (2)	0.35137 (14)	0.0438 (4)
O5	0.61196 (18)	0.2846 (2)	0.29611 (14)	0.0490 (4)
H5	0.6515	0.2998	0.3753	0.074*
C1	0.1317 (2)	0.3162 (2)	−0.05532 (17)	0.0281 (4)
C2	0.2029 (2)	0.3002 (2)	0.09437 (17)	0.0292 (4)
H2	0.1550	0.3919	0.1422	0.035*
C3	0.3842 (2)	0.3234 (3)	0.12309 (19)	0.0351 (5)
H3A	0.4314	0.2211	0.0872	0.042*
H3B	0.4096	0.4301	0.0776	0.042*
C4	0.4609 (2)	0.3402 (2)	0.26915 (18)	0.0311 (4)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0297 (2)	0.0300 (2)	0.01576 (19)	−0.00928 (13)	0.00016 (13)	0.00256 (12)
O1	0.0419 (7)	0.0330 (7)	0.0178 (6)	−0.0093 (6)	0.0025 (5)	0.0026 (5)
O1W	0.0401 (8)	0.0379 (8)	0.0383 (8)	−0.0043 (6)	0.0123 (7)	−0.0047 (6)
O2	0.0631 (10)	0.0318 (7)	0.0247 (7)	−0.0091 (7)	0.0052 (7)	0.0041 (6)
O3	0.0375 (7)	0.0379 (7)	0.0206 (7)	−0.0083 (6)	0.0001 (6)	0.0029 (5)
O4	0.0424 (8)	0.0591 (10)	0.0299 (8)	0.0064 (7)	0.0083 (6)	−0.0017 (7)
O5	0.0381 (8)	0.0670 (11)	0.0379 (9)	0.0049 (8)	−0.0003 (7)	−0.0145 (7)
C1	0.0321 (10)	0.0296 (10)	0.0229 (9)	0.0010 (8)	0.0064 (8)	0.0012 (7)
C2	0.0352 (10)	0.0286 (10)	0.0233 (9)	−0.0024 (8)	0.0055 (8)	−0.0001 (7)
C3	0.0363 (11)	0.0426 (11)	0.0263 (10)	−0.0050 (9)	0.0066 (8)	−0.0012 (8)
C4	0.0353 (10)	0.0294 (10)	0.0283 (10)	−0.0061 (8)	0.0066 (8)	−0.0004 (7)

Geometric parameters (Å, °)

Ni1—O3	1.9134 (12)	O3—C1	1.262 (2)
Ni1—O3^{i}	1.9134 (12)	O4—C4	1.202 (2)
Ni1—O1^{i}	1.9509 (12)	O5—C4	1.316 (2)
Ni1—O1	1.9509 (12)	O5—H5	0.8200
Ni1—O1W	2.5151 (13)	C1—C2	1.534 (2)
Ni1—O1W^{i}	2.5151 (13)	C2—C3	1.510 (3)
O1—C2	1.438 (2)	C2—H2	0.9800
O1—H1	0.8600	C3—C4	1.509 (3)

O1W—H1W	0.8126	C3—H3A	0.9700
O1W—H2W	0.8117	C3—H3B	0.9700
O2—C1	1.241 (2)		

O3—Ni1—O3$^{\text{i}}$	180.0	C1—O3—Ni1	116.15 (11)
O3—Ni1—O1$^{\text{i}}$	96.63 (5)	C4—O5—H5	109.4
O3$^{\text{i}}$—Ni1—O1$^{\text{i}}$	83.37 (5)	O2—C1—O3	123.34 (17)
O3—Ni1—O1	83.37 (5)	O2—C1—C2	118.40 (16)
O3$^{\text{i}}$—Ni1—O1	96.63 (5)	O3—C1—C2	118.26 (15)
O1$^{\text{i}}$—Ni1—O1	180.0	O1—C2—C3	110.43 (16)
O3—Ni1—O1W	87.18 (5)	O1—C2—C1	106.89 (14)
O3$^{\text{i}}$—Ni1—O1W	92.82 (5)	C3—C2—C1	110.30 (15)
O1$^{\text{i}}$—Ni1—O1W	87.17 (5)	O1—C2—H2	109.7
O1—Ni1—O1W	92.83 (5)	C3—C2—H2	109.7
O1—Ni1—O1W$^{\text{i}}$	87.18 (5)	C1—C2—H2	109.7
O1$^{\text{i}}$—Ni1—O1W$^{\text{i}}$	92.82 (5)	C4—C3—C2	113.71 (16)
O3$^{\text{i}}$—Ni1—O1W$^{\text{i}}$	87.17 (5)	C4—C3—H3A	108.8
O3—Ni1—O1W$^{\text{i}}$	92.83 (5)	C2—C3—H3A	108.8
O1W—Ni1—O1W$^{\text{i}}$	180.0	C4—C3—H3B	108.8
C2—O1—Ni1	113.97 (10)	C2—C3—H3B	108.8
C2—O1—H1	117.4	H3A—C3—H3B	107.7
Ni1—O1—H1	118.1	O4—C4—O5	123.53 (19)
Ni1—O1W—H1W	122.0	O4—C4—C3	124.69 (18)
Ni1—O1W—H2W	108.1	O5—C4—C3	111.76 (16)
H1W—O1W—H2W	106.4		

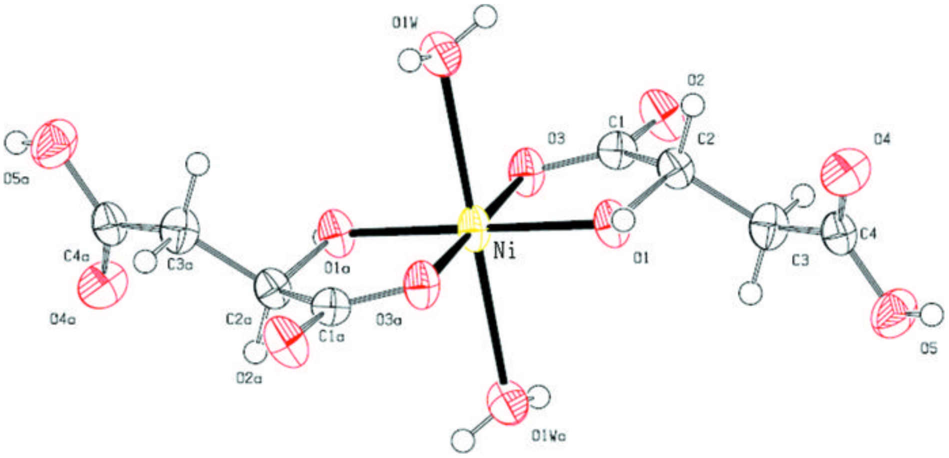
Symmetry codes: (i) −*x*, −*y*, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O2$^{\text{ii}}$	0.86	1.77	2.6245 (17)	172
O1W—H1W···O2$^{\text{iii}}$	0.81	2.05	2.8374 (19)	163
O1W—H2W···O4$^{\text{iv}}$	0.81	2.08	2.843 (2)	156
O5—H5···O1W$^{\text{v}}$	0.82	1.87	2.6835 (19)	171

Symmetry codes: (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*, −*y*+1, −*z*; (iv) −*x*, *y*−1/2, −*z*+1/2; (v) *x*+1, −*y*+1/2, *z*+1/2.

Fig. 1


O1W
O2
C1
O3
C2
O4
O5a
C4a
C3a
O1a
Ni
O1
C3
C4
O4a
C2a
C1a
O3a
O5
O2a
O1Wa