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[μ-4,7-Dioxa-1,10-diazaoctane-1,1,10,-10-tetraacetat0-1$\kappa^4O^1,O^{1'},N^1,O^4$:-2$\kappa^4O^7,N^{10},O^{10},O^{10'}$]bis[aquanickel(II)] dihydrate

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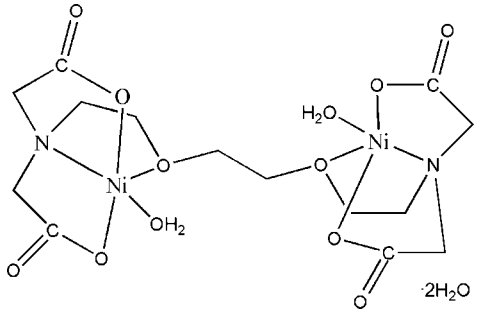
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.024; wR factor = 0.060; data-to-parameter ratio = 12.8.

The Ni atom in the title complex, $[Ni_2(C_{14}H_{20}N_2O_{10})(H_2O)_2]\cdot 2H_2O$ or $\{[Ni_2(egta)\cdot 2H_2O]\cdot 2H_2O\}$ [egtaH4 is 3,12-bis-(carboxymethyl)-6,9-dioxa-3,12-diazatetradecanedioic acid], has a square-pyramidal coordination geometry. Both Ni^{II} centres are pentacoordinated by one N atom and three O atoms of egta, forming the basal plane, and one O donor water molecule in the apical position. The dinuclear complex is arranged around an inversion centre. The solvent water molecules and coordinated water molecules are involved in hydrogen bonds with uncoordinated O atoms of carboxylate groups, which link the complex molecules to form a three-dimensional supramolecular network structure.

Related literature

For related literature, see: Bomas-Almenar *et al.* (1993).



Experimental

Crystal data

$[Ni_2(C_{14}H_{20}N_2O_{10})(H_2O)_2]\cdot 2H_2O$
M_r = 565.80
Monoclinic, $C2/c$
a = 21.031 (2) Å
b = 7.5299 (9) Å
c = 13.5789 (16) Å
β = 90.464 (1)°
V = 2150.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.83 mm^{-1}
T = 298 (2) K
0.34 × 0.12 × 0.04 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan *SADABS* (Sheldrick, 1996)
T_{min} = 0.580, T_{max} = 0.931
7928 measured reflections
2007 independent reflections
1756 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.060
S = 1.08
2007 reflections
157 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O6—H1W···O7	0.823 (16)	1.859 (17)	2.671 (3)	169 (3)
O6—H2W···O4^{i}	0.821 (16)	1.832 (18)	2.625 (2)	162 (3)
O7—H3W···O2^{ii}	0.847 (16)	1.924 (17)	2.758 (2)	168 (3)
O7—H4W···O2^{iii}	0.826 (16)	1.991 (17)	2.785 (2)	161 (3)

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

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: *ORTEPIII* (Burnett & Johnson, 1996); *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: DN2227).

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supplementary materials

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[μ-4,7-Dioxa-1,10-diazaoctane-1,1,10,10-tetraacetat0-1$\kappa^4O^1,O^{1'},N^1,O^4$:2$\kappa^4O^7,N^{10},O^{10},O^{10'}$]bis[aquanickel(II)] dihydrate

H.-Q. Liu

Comment

The coordination of multipyridine and multi-carboxylate ligands to metal centers has proved to be an excellent tool in the assembly process and has been highly influenced by the structural characterizations of tectonic spacers. Polydentate amino-polycarboxylate acid [H4egta=3,12-bis (carboxymethyl)-6, 9-dioxa-3, 12-diazatetradecanedioic acid], being extensively used as a calcium buffer in biological research, possesses symmetrical four potential coordinating sites and renders it an appropriate candidate to improve the extension of the ferrimagnetic lattice from one dimensional (one-dimensional) systems to two-dimensional and three-dimensional networks (Bomas-Almenar *et al.*, 1993). In this paper, we report the synthesis and crystal structure of the title complex,(I).

As illustrated in Fig. 1, the neutral dinuclear molecule, lies on a centre of symmetry. Both nickel(II) centers are pentacoordinated to one N atom and three O atoms of egta, forming the basal plane, and one O donors of water molecule being in axial position, then leading to a distorted square-pyramid environment. The structural components are connected through O—H···O hydrogen bonding involving the lattice water molecules as donors and the uncoordinated O atoms as acceptors thus forming a three dimensionnal supramolecular network structure (Table 1).

Experimental

The title complex was prepared by the addition of a stoichiometric amount of nickel chloride (1 mmol) to a hot methanol solution (10 ml) of egtaH4 (1 mmol). The resulting solution was filtered, and pale green blocky crystals were obtained on slow evaporation of the solvent over several days at room temperature.

Refinement

Carbon-bound H atoms were placed at calculated positions and were treated as riding on the parent C atoms with C—H= 97Å and with U_{iso}(H) = 1.2U_{eq}(C). H atoms attached to water molecules were found in a difference map and their positions were refined using restraints (O—H= 0.84 (2)Å and H···H= 1.38 Å) with U_{iso}(H) = 1.5U_{eq}(O).

Figures

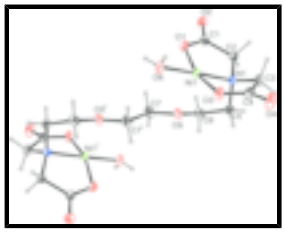

Fig. 1. The structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 30% probability level. Free water molecule has been omitted for clarity. [Symmetry code: (i) 1 − *x*, 1 − *y*, 1 − *z*].

[μ-4,7-dioxa-1,10-diazaoctane-1,1,10,10-tetraacetato- 1κ$^4O^1$,$O^{1'}$,N^1,O^4:2κ$^4O^7$,N^{10},O^{10},$O^{10'}$] bis[aquanickel(II)] dihydrate

Crystal data

$[Ni_2(C_{14}H_{20}N_2O_{10})(H_2O)_2]\cdot 2H_2O$	F_{000} = 1176
M_r = 565.80	D_x = 1.748 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2007 reflections
a = 21.031 (2) Å	θ = 2.9–25.5º
b = 7.5299 (9) Å	μ = 1.83 mm^{-1}
c = 13.5789 (16) Å	T = 298 (2) K
β = 90.4640 (10)º	Block, green
V = 2150.3 (4) Å^3	0.34 × 0.12 × 0.04 mm
Z = 4	

Data collection

Bruker APEXII area-detector diffractometer	2007 independent reflections
Radiation source: fine-focus sealed tube	1756 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.027
T = 298(2) K	θ_{max} = 25.5º
φ and ω scans	θ_{min} = 2.9º
Absorption correction: multi-scan SADABS (Sheldrick, 1996)	h = −25→25
T_{min} = 0.580, T_{max} = 0.931	k = −9→9
7928 measured reflections	l = −16→15

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 atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.060	$w = 1/[\sigma^2(F_o^2) + (0.0294P)^2 + 0.9421P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08	$(\Delta/\sigma)_{max}$ = 0.002
2007 reflections	$\Delta\rho_{max}$ = 0.34 e Å^{-3}
157 parameters	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
6 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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	0.628725 (12)	0.21993 (3)	0.564464 (18)	0.02584 (10)
O1	0.67589 (7)	0.37797 (18)	0.47843 (11)	0.0328 (3)
O2	0.74168 (7)	0.3724 (2)	0.35187 (12)	0.0412 (4)
O3	0.60468 (7)	0.02307 (18)	0.64666 (10)	0.0352 (4)
O4	0.62324 (9)	−0.2649 (2)	0.66266 (12)	0.0497 (5)
O5	0.53305 (7)	0.28393 (19)	0.47083 (11)	0.0336 (4)
O6	0.61807 (8)	0.3871 (2)	0.67071 (12)	0.0383 (4)
H1W	0.6396 (12)	0.354 (3)	0.7181 (16)	0.057*
H2W	0.6247 (12)	0.492 (2)	0.6580 (18)	0.057*
O7	0.70120 (9)	0.2874 (2)	0.80982 (13)	0.0453 (4)
H3W	0.7178 (12)	0.385 (3)	0.828 (2)	0.068*
H4W	0.7255 (12)	0.239 (3)	0.7706 (19)	0.068*
N1	0.63903 (8)	0.0375 (2)	0.45889 (11)	0.0250 (4)
C1	0.70315 (10)	0.2990 (3)	0.40776 (16)	0.0290 (5)
C2	0.68935 (9)	0.1023 (3)	0.39122 (15)	0.0282 (5)
H2A	0.6757	0.0840	0.3236	0.034*
H2B	0.7280	0.0344	0.4020	0.034*
C3	0.65593 (10)	−0.1272 (3)	0.51335 (15)	0.0306 (5)
H3A	0.7018	−0.1353	0.5206	0.037*
H3B	0.6414	−0.2300	0.4765	0.037*
C4	0.62514 (10)	−0.1259 (3)	0.61503 (15)	0.0318 (5)
C5	0.57655 (10)	0.0158 (3)	0.40651 (15)	0.0315 (5)
H5A	0.5477	−0.0502	0.4482	0.038*
H5B	0.5829	−0.0522	0.3467	0.038*
C6	0.54713 (10)	0.1921 (3)	0.38073 (16)	0.0346 (5)
H6A	0.5764	0.2617	0.3416	0.042*
H6B	0.5084	0.1742	0.3426	0.042*
C7	0.51520 (11)	0.4654 (3)	0.45390 (16)	0.0399 (6)
H7A	0.4855	0.4729	0.3991	0.048*
H7B	0.5524	0.5357	0.4382	0.048*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.03413 (17)	0.01904 (16)	0.02442 (16)	−0.00075 (11)	0.00398 (11)	−0.00223 (11)
O1	0.0396 (8)	0.0230 (8)	0.0360 (8)	−0.0019 (6)	0.0064 (7)	−0.0023 (6)
O2	0.0463 (10)	0.0303 (9)	0.0472 (10)	−0.0061 (7)	0.0184 (8)	0.0033 (7)
O3	0.0509 (9)	0.0244 (8)	0.0305 (8)	−0.0004 (7)	0.0112 (7)	−0.0017 (6)
O4	0.0880 (14)	0.0262 (9)	0.0349 (9)	0.0034 (9)	0.0107 (9)	0.0057 (7)
O5	0.0361 (9)	0.0321 (8)	0.0326 (8)	0.0080 (7)	0.0034 (7)	0.0013 (6)
O6	0.0560 (11)	0.0231 (8)	0.0358 (9)	0.0043 (8)	0.0001 (8)	−0.0041 (7)
O7	0.0633 (12)	0.0324 (9)	0.0404 (10)	−0.0015 (8)	0.0109 (9)	−0.0031 (8)
N1	0.0269 (9)	0.0236 (9)	0.0247 (9)	−0.0006 (7)	0.0034 (7)	0.0001 (7)
C1	0.0274 (11)	0.0259 (11)	0.0336 (12)	0.0016 (9)	−0.0005 (9)	0.0032 (9)
C2	0.0288 (11)	0.0267 (11)	0.0293 (11)	−0.0013 (9)	0.0066 (9)	−0.0014 (9)
C3	0.0390 (12)	0.0225 (11)	0.0306 (11)	0.0030 (9)	0.0043 (9)	−0.0015 (9)
C4	0.0390 (13)	0.0285 (12)	0.0279 (11)	−0.0033 (9)	0.0005 (9)	0.0008 (10)
C5	0.0318 (12)	0.0320 (12)	0.0308 (11)	−0.0044 (9)	0.0027 (9)	−0.0060 (9)
C6	0.0325 (12)	0.0442 (14)	0.0273 (12)	0.0036 (10)	0.0003 (9)	−0.0013 (10)
C7	0.0415 (13)	0.0351 (13)	0.0433 (14)	0.0119 (10)	0.0036 (10)	0.0064 (11)

Geometric parameters (Å, °)

Ni1—O3	1.9255 (14)	N1—C5	1.498 (3)
Ni1—O6	1.9293 (15)	C1—C2	1.525 (3)
Ni1—O1	1.9451 (14)	C2—H2A	0.9700
Ni1—N1	1.9986 (16)	C2—H2B	0.9700
O1—C1	1.270 (2)	C3—C4	1.530 (3)
O2—C1	1.244 (2)	C3—H3A	0.9700
O3—C4	1.277 (2)	C3—H3B	0.9700
O4—C4	1.231 (3)	C5—C6	1.505 (3)
O5—C7	1.436 (3)	C5—H5A	0.9700
O5—C6	1.438 (2)	C5—H5B	0.9700
O6—H1W	0.823 (16)	C6—H6A	0.9700
O6—H2W	0.821 (16)	C6—H6B	0.9700
O7—H3W	0.847 (16)	C7—C7^{i}	1.503 (4)
O7—H4W	0.826 (16)	C7—H7A	0.9700
N1—C3	1.486 (2)	C7—H7B	0.9700
N1—C2	1.489 (2)		
O3—Ni1—O6	92.08 (7)	N1—C3—C4	110.04 (16)
O3—Ni1—O1	163.08 (6)	N1—C3—H3A	109.7
O6—Ni1—O1	96.47 (7)	C4—C3—H3A	109.7
O3—Ni1—N1	85.25 (6)	N1—C3—H3B	109.7
O6—Ni1—N1	177.32 (7)	C4—C3—H3B	109.7
O1—Ni1—N1	86.04 (6)	H3A—C3—H3B	108.2
C1—O1—Ni1	113.76 (13)	O4—C4—O3	123.9 (2)
C4—O3—Ni1	112.98 (13)	O4—C4—C3	118.98 (19)
C7—O5—C6	112.13 (16)	O3—C4—C3	117.09 (18)

Ni1—O6—H1W	108.7 (19)	N1—C5—C6	111.81 (17)
Ni1—O6—H2W	116.7 (19)	N1—C5—H5A	109.3
H1W—O6—H2W	111 (2)	C6—C5—H5A	109.3
H3W—O7—H4W	108 (2)	N1—C5—H5B	109.3
C3—N1—C2	114.36 (16)	C6—C5—H5B	109.3
C3—N1—C5	110.52 (16)	H5A—C5—H5B	107.9
C2—N1—C5	111.58 (15)	O5—C6—C5	108.25 (17)
C3—N1—Ni1	104.10 (11)	O5—C6—H6A	110.0
C2—N1—Ni1	107.40 (12)	C5—C6—H6A	110.0
C5—N1—Ni1	108.41 (12)	O5—C6—H6B	110.0
O2—C1—O1	123.58 (19)	C5—C6—H6B	110.0
O2—C1—C2	117.76 (18)	H6A—C6—H6B	108.4
O1—C1—C2	118.65 (18)	O5—C7—C7^{i}	108.0 (2)
N1—C2—C1	111.27 (16)	O5—C7—H7A	110.1
N1—C2—H2A	109.4	C7^{i}—C7—H7A	110.1
C1—C2—H2A	109.4	O5—C7—H7B	110.1
N1—C2—H2B	109.4	C7^{i}—C7—H7B	110.1
C1—C2—H2B	109.4	H7A—C7—H7B	108.4
H2A—C2—H2B	108.0		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H1W···O7	0.823 (16)	1.859 (17)	2.671 (3)	169 (3)
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O7—H4W···O2^{iv}	0.826 (16)	1.991 (17)	2.785 (2)	161 (3)

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

Fig. 1