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Diaqua(cyclohexane-1,2-diamine-κ^2N,N')(*N*,*N*,*N*′,*N*′-tetramethylethylenediamine-κ^2N,N')nickel(II) dichloride dihydrate

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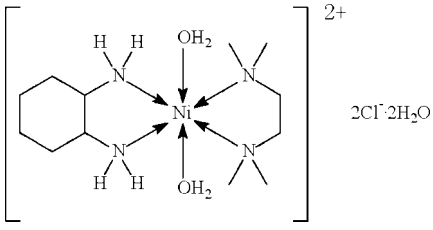
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.003 Å; R factor = 0.025; wR factor = 0.073; data-to-parameter ratio = 17.2.

In the title compound, $[Ni(C_6H_{14}N_2)(C_6H_{16}N_2)(H_2O)_2]Cl_2\cdot 2H_2O$, the cation lies about a twofold rotation axis. The octahedral complex cation is linked to the Cl^- anions by hydrogen bonds to form a layer structure.

Related literature

For other nickel dichloride complexes of substituted ethylenediamines, see Liang *et al.* (2007).



Experimental

Crystal data

$[Ni(C_6H_{14}N_2)(C_6H_{16}N_2)(H_2O)_2]Cl_2\cdot 2H_2O$
M_r = 432.07
Orthorhombic, *Aba*2
a = 14.159 (1) Å
b = 9.843 (1) Å
c = 15.221 (1) Å
V = 2121.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.19 mm^{-1}
T = 295 (2) K
0.30 × 0.30 × 0.20 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.702, T_{max} = 0.797
6267 measured reflections
2217 independent reflections
2103 reflections with $I > 2\sigma(I)$
R_{int} = 0.015

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.073
S = 1.12
2217 reflections
129 parameters
11 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Absolute structure: Flack (1983), with 944 Friedel pairs
Flack parameter: −0.01 (1)

Table 1
Selected bond lengths (Å).

Ni1—N1	2.091 (3)	Ni1—O1*W*	2.153 (2)
Ni1—N2	2.163 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*1···O2*W*	0.850 (10)	1.871 (10)	2.721 (3)	178 (3)
O1*W*—H1*W*2···Cl1	0.865 (10)	2.300 (11)	3.156 (2)	170 (3)
O2*W*—H2*W*1···Cl1^{i}	0.846 (10)	2.338 (17)	3.153 (2)	162 (5)
O2*W*—H2*W*2···Cl1^{ii}	0.841 (10)	2.401 (17)	3.215 (2)	163 (4)
N1—H1*N*1···Cl1	0.862 (9)	2.642 (10)	3.4910 (17)	168 (3)
N1—H1*N*2···Cl1^{iii}	0.847 (10)	2.712 (14)	3.529 (3)	163 (3)

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2296).

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supplementary materials

Acta Cryst. (2007). E**63**, m2228 [**doi:10.1107/S1600536807035386**]

Diaqua(cyclohexane-1,2-diamine-κ^2N,N')(*N*,*N*,*N*',*N*'-tetramethylethylenediamine-κ^2N,N')nickel(II) dichloride dihydrate

Y.-Z. Zhang, Z.-L. Chen and S. W. Ng

Comment

The crystal structures of few diamine complexes of nickel have been reported; a recent study described the mixed-ligand diaqua complex of substituted ethylenediamines (Liang *et al.*, 2007). The present mixed ligand complex also has coordinated water. The six-coordinate cation and free chloride anions are linked by hydrogen bonds into a layers structure.

Experimental

To an ethanol solution (5 ml) of nickel chloride hexahydrate (0.238 g, 1 mmol) was added an ethanol solution (10 ml) of 1,2-diaminocyclohexane (0.12 ml, 1 mmol) and *N*,*N*,*N*',*N*'-tetramethyethylenediamine (0.15 ml, 1 mmol). The solution was filtered. Ether was diffused into the filtrate; blue crystals were isolated in 50% yield after a week.

Refinement

Carbon-bound hydrogen atoms were placed at calculated positions in the riding model approximation [C—H 0.96 to 0.98 Å, U(H) = 1.2 or 1.5 U_{eq}(C)]. The water and amino H atoms were located in a difference Fourier map, and were refined with a distance restraint of O–H = N–H = 0.85±0.01 Å.

The C–C bond in the ethylenediamine part of the molecule was restrained to 1.500±0.002 Å; a somewhat tight restraint was necessary.

Figures

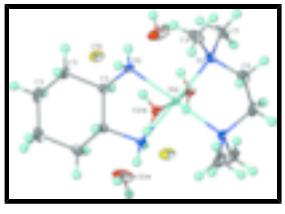

Fig. 1. Thermal ellipsoid plot. The molecule lies about a twofold rotation axis, and the un-labeled atoms are related to the labeled ones by $1-x$, $1-y$, z.

Diaqua(cyclohexane-1,2-diamine-κ$^2N,N'$)(N,*N*,*N*',*N*'- tetramethylethylenediamine-κ$^2N,N'$)nickel(II) dichloride dihydrate

Crystal data

$[Ni(C_6H_{14}N_2)(C_6H_{16}N_2)(H_2O)_2]Cl_2\cdot 2H_2O$	F_{000} = 928
M_r = 432.07	D_x = 1.353 Mg m^{-3}

Orthorhombic, *Aba*2	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: A 2 -2ac	Cell parameters from 4527 reflections
a = 14.159 (1) Å	θ = 2.7–27.4º
b = 9.843 (1) Å	μ = 1.19 mm^{-1}
c = 15.221 (1) Å	*T* = 295 (2) K
V = 2121.4 (3) Å^3	Prism, blue
Z = 4	0.30 × 0.30 × 0.20 mm

Data collection

Bruker APEX area-detector diffractometer	2217 independent reflections
Radiation source: fine-focus sealed tube	2103 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.015
T = 295(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 2.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −18→16
T_{min} = 0.702, T_{max} = 0.797	*k* = −12→8
6267 measured reflections	*l* = −19→19

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.025	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.4568P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.073	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.12	$\Delta\rho_{max}$ = 0.50 e Å^{-3}
2217 reflections	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
129 parameters	Extinction correction: none
11 restraints	Absolute structure: Flack (1983), with 944 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.01 (1)
Secondary atom site location: difference Fourier map	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.5000	0.5000	0.49999 (2)	0.02217 (10)
Cl1	0.31836 (4)	0.83038 (6)	0.61089 (9)	0.05226 (16)
O1W	0.49376 (9)	0.71842 (15)	0.5054 (2)	0.0329 (3)
O2W	0.63652 (17)	0.8514 (2)	0.5887 (2)	0.0839 (10)
N1	0.4022 (2)	0.49706 (13)	0.60291 (18)	0.0270 (5)

N2	0.3974 (2)	0.49513 (15)	0.3947 (2)	0.0339 (6)
C1	0.45361 (14)	0.4615 (2)	0.68462 (15)	0.0283 (4)
H1	0.4667	0.3638	0.6847	0.034*
C2	0.3966 (2)	0.49752 (19)	0.76682 (18)	0.0408 (6)
H2A	0.3804	0.5932	0.7654	0.049*
H2B	0.3383	0.4457	0.7671	0.049*
C3	0.4519 (2)	0.4673 (3)	0.85041 (18)	0.0520 (6)
H3A	0.4164	0.4998	0.9007	0.062*
H3B	0.4592	0.3697	0.8565	0.062*
C4	0.32382 (19)	0.5997 (3)	0.4023 (2)	0.0580 (8)
H4A	0.2809	0.5920	0.3537	0.087*
H4B	0.3525	0.6881	0.4018	0.087*
H4C	0.2899	0.5874	0.4563	0.087*
C5	0.3493 (2)	0.3631 (3)	0.3864 (2)	0.0549 (7)
H5A	0.3051	0.3668	0.3387	0.082*
H5B	0.3164	0.3430	0.4400	0.082*
H5C	0.3952	0.2934	0.3751	0.082*
C6	0.45062 (13)	0.5269 (4)	0.3154 (2)	0.0640 (9)
H6A	0.4529	0.6248	0.3084	0.077*
H6B	0.4173	0.4899	0.2652	0.077*
H1W1	0.5390 (12)	0.760 (3)	0.5303 (18)	0.062 (10)*
H1W2	0.4418 (10)	0.748 (3)	0.5284 (19)	0.070 (11)*
H2W1	0.6916 (15)	0.818 (4)	0.589 (5)	0.17 (3)*
H2W2	0.638 (3)	0.9368 (10)	0.587 (3)	0.103 (14)*
H1N1	0.3817 (15)	0.5779 (11)	0.6136 (18)	0.035 (6)*
H1N2	0.3567 (14)	0.4420 (18)	0.609 (3)	0.068 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.01982 (15)	0.02320 (15)	0.02349 (15)	0.00020 (10)	0.000	0.000
Cl1	0.0385 (3)	0.0394 (3)	0.0788 (4)	0.0064 (2)	0.0048 (3)	−0.0112 (3)
O1W	0.0306 (6)	0.0288 (6)	0.0393 (9)	0.0005 (5)	0.0005 (7)	0.0019 (10)
O2W	0.0492 (11)	0.0482 (11)	0.154 (3)	−0.0034 (10)	−0.0260 (14)	−0.0254 (14)
N1	0.0215 (12)	0.0280 (11)	0.0313 (13)	−0.0018 (5)	0.0014 (11)	−0.0013 (6)
N2	0.0307 (14)	0.0373 (13)	0.0338 (15)	−0.0002 (6)	−0.0075 (13)	−0.0009 (6)
C1	0.0297 (11)	0.0261 (8)	0.0290 (10)	−0.0011 (9)	0.0014 (9)	0.0009 (9)
C2	0.0420 (14)	0.0450 (13)	0.0354 (13)	0.0001 (7)	0.0129 (11)	−0.0014 (9)
C3	0.0686 (19)	0.0550 (13)	0.0325 (12)	0.0040 (14)	0.0104 (12)	0.0025 (11)
C4	0.0478 (14)	0.0505 (14)	0.0756 (19)	0.0168 (11)	−0.0311 (14)	−0.0161 (13)
C5	0.0621 (15)	0.0419 (12)	0.0608 (15)	−0.0105 (11)	−0.0321 (14)	−0.0009 (11)
C6	0.060 (2)	0.097 (2)	0.0350 (16)	0.0045 (17)	−0.0107 (15)	0.0078 (15)

Geometric parameters (Å, °)

Ni1—N1^{i}	2.091 (3)	C1—C2	1.530 (3)
Ni1—N1	2.091 (3)	C1—H1	0.9800
Ni1—O1W^{i}	2.153 (2)	C2—C3	1.523 (4)

Ni1—N2^{i}	2.163 (3)	C2—H2A	0.9700
Ni1—N2	2.163 (3)	C2—H2B	0.9700
Ni1—O1W	2.153 (2)	C3—C3^{i}	1.506 (7)
O1W—H1W1	0.850 (10)	C3—H3A	0.9700
O1W—H1W2	0.865 (10)	C3—H3B	0.9700
O2W—H2W1	0.846 (10)	C4—H4A	0.9600
O2W—H2W2	0.841 (10)	C4—H4B	0.9600
N1—C1	1.483 (4)	C4—H4C	0.9600
N1—H1N1	0.862 (9)	C5—H5A	0.9600
N1—H1N2	0.847 (10)	C5—H5B	0.9600
N2—C6	1.457 (4)	C5—H5C	0.9600
N2—C4	1.470 (3)	C6—C6^{i}	1.496 (2)
N2—C5	1.473 (3)	C6—H6A	0.9700
C1—C1^{i}	1.516 (4)	C6—H6B	0.9700
N1^{i}—Ni1—N1	82.98 (15)	N1—C1—H1	109.0
N1^{i}—Ni1—O1W^{i}	87.59 (7)	C1^{i}—C1—H1	109.0
N1—Ni1—O1W^{i}	89.12 (7)	C2—C1—H1	109.0
N1^{i}—Ni1—O1W	89.12 (7)	C3—C2—C1	111.5 (2)
N1—Ni1—O1W	87.59 (7)	C3—C2—H2A	109.3
O1W^{i}—Ni1—O1W	175.61 (16)	C1—C2—H2A	109.3
N1^{i}—Ni1—N2^{i}	96.31 (6)	C3—C2—H2B	109.3
N1—Ni1—N2^{i}	179.15 (13)	C1—C2—H2B	109.3
O1W^{i}—Ni1—N2^{i}	91.32 (8)	H2A—C2—H2B	108.0
O1W—Ni1—N2^{i}	91.94 (7)	C3^{i}—C3—C2	112.41 (18)
N1^{i}—Ni1—N2	179.15 (12)	C3^{i}—C3—H3A	109.1
N1—Ni1—N2	96.31 (6)	C2—C3—H3A	109.1
O1W^{i}—Ni1—N2	91.94 (7)	C3^{i}—C3—H3B	109.1
O1W—Ni1—N2	91.32 (8)	C2—C3—H3B	109.1
N2^{i}—Ni1—N2	84.41 (16)	H3A—C3—H3B	107.9
Ni1—O1W—H1W1	118 (2)	N2—C4—H4A	109.5
Ni1—O1W—H1W2	113 (2)	N2—C4—H4B	109.5
H1W1—O1W—H1W2	107.3 (16)	H4A—C4—H4B	109.5
H2W1—O2W—H2W2	111.4 (18)	N2—C4—H4C	109.5
C1—N1—Ni1	107.83 (18)	H4A—C4—H4C	109.5
C1—N1—H1N1	103.0 (19)	H4B—C4—H4C	109.5
Ni1—N1—H1N1	110.5 (16)	N2—C5—H5A	109.5
C1—N1—H1N2	97 (3)	N2—C5—H5B	109.5
Ni1—N1—H1N2	127 (2)	H5A—C5—H5B	109.5
H1N1—N1—H1N2	108.3 (15)	N2—C5—H5C	109.5
C6—N2—C4	106.3 (2)	H5A—C5—H5C	109.5
C6—N2—C5	110.9 (3)	H5B—C5—H5C	109.5
C4—N2—C5	107.3 (3)	N2—C6—C6^{i}	114.0 (2)
C6—N2—Ni1	105.19 (19)	N2—C6—H6A	108.7
C4—N2—Ni1	113.75 (19)	C6^{i}—C6—H6A	108.7
C5—N2—Ni1	113.20 (17)	N2—C6—H6B	108.7

N1—C1—C1^{i}	107.93 (16)	C6^{i}—C6—H6B	108.7
N1—C1—C2	111.8 (2)	H6A—C6—H6B	107.6
C1^{i}—C1—C2	109.96 (16)		
N1^{i}—Ni1—N1—C1	−14.97 (10)	N1—Ni1—N2—C5	−71.0 (2)
O1W^{i}—Ni1—N1—C1	72.71 (13)	O1W^{i}—Ni1—N2—C5	18.3 (2)
O1W—Ni1—N1—C1	−104.38 (13)	O1W—Ni1—N2—C5	−158.7 (2)
N2—Ni1—N1—C1	164.56 (12)	N2^{i}—Ni1—N2—C5	109.5 (2)
N1—Ni1—N2—C6	167.72 (18)	Ni1—N1—C1—C1^{i}	41.8 (2)
O1W^{i}—Ni1—N2—C6	−102.95 (18)	Ni1—N1—C1—C2	162.90 (13)
O1W—Ni1—N2—C6	80.00 (19)	N1—C1—C2—C3	−177.1 (2)
N2^{i}—Ni1—N2—C6	−11.82 (16)	C1^{i}—C1—C2—C3	−57.3 (3)
N1—Ni1—N2—C4	51.8 (2)	C1—C2—C3—C3^{i}	53.0 (3)
O1W^{i}—Ni1—N2—C4	141.1 (2)	C4—N2—C6—C6^{i}	155.4 (4)
O1W—Ni1—N2—C4	−35.9 (2)	C5—N2—C6—C6^{i}	−88.3 (5)
N2^{i}—Ni1—N2—C4	−127.8 (2)	Ni1—N2—C6—C6^{i}	34.5 (5)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W1···O2W	0.850 (10)	1.871 (10)	2.721 (3)	178 (3)
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O2W—H2W2···Cl1^{iii}	0.841 (10)	2.401 (17)	3.215 (2)	163 (4)
N1—H1N1···Cl1	0.862 (9)	2.642 (10)	3.4910 (17)	168 (3)
N1—H1N2···Cl1^{iv}	0.847 (10)	2.712 (14)	3.529 (3)	163 (3)

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

Fig. 1