Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis{1-[bis(2-aminoethyl)amino]propan-2-ol}nickel(II) bis(perchlorate)

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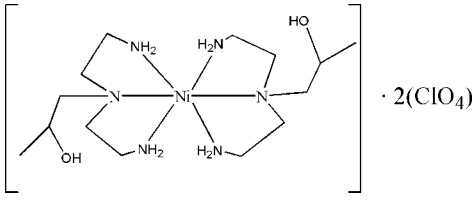
Received 11 October 2007; accepted 14 October 2007

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; disorder in main residue; R factor = 0.056; wR factor = 0.167; data-to-parameter ratio = 16.2.

In the title compound, $[Ni(C_7H_{19}N_3O)_2](ClO_4)_2$, the Ni atom, which is located on an inversion center, is hexacoordinated by six N atoms from two ligands, forming a distorted octahedron. The complex molecules are linked by N—H···O hydrogen bonding, forming a one-dimensional chain structure along the a axis, which is further hydrogen bonded with the perchlorate anion to form a three-dimensional framework. One $—CH_2—CH_2—$ group is disordered over two positions; the site occupancy factors are *ca.* 0.6 and 0.4.

Related literature

For related literature, see: Huang *et al.* (2004); Li *et al.* (2002); Xia *et al.* (2001).



· 2(ClO_4)

Experimental

Crystal data

$[Ni(C_7H_{19}N_3O)_2](ClO_4)_2$
M_r = 580.11
Monoclinic, $P2_1/n$
a = 7.915 (3) Å
b = 17.243 (6) Å
c = 9.127 (3) Å
β = 106.442 (4)°
V = 1194.6 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.10 mm^{-1}
T = 298 (2) K
0.62 × 0.54 × 0.18 mm

Data collection

Bruker SMART 1000 diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.550, T_{max} = 0.827
7286 measured reflections
2796 independent reflections
2060 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.167
S = 1.04
2796 reflections
173 parameters
14 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.94 e Å^{-3}
$\Delta\rho_{min}$ = −0.62 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O4	0.90	2.42	3.293 (6)	164
N2—H2A···O3	0.90	2.69	3.416 (6)	139
N2—H2B···O1^{i}	0.90	2.31	3.067 (4)	141
O1—H1···O2^{ii}	0.82	2.36	3.043 (6)	142

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z+2$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); 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) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

The authors thank the National Natural Science Foundation of China (grant Nos. 505720319), and the Natural Science Foundation of Jiangsu Province (grant Nos. BK2005130).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2243).

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  doi:10.1107/S1600536807050386

supplementary materials

Acta Cryst. (2007). E**63**, m2754 [**doi:10.1107/S1600536807050386**]

Bis{1-[bis(2-aminoethyl)amino]propan-2-ol}nickel(II) bis(perchlorate)

B.-H. Qian, Z.-M. Zhang, L.-D. Lu, X.-J. Yang and X. Wang

Comment

Transition-metal polyamine complexes with hydroxypropyl pendant group play important roles in hydrolytic metalloenzymes (Xia *et al.*,2001;Huang *et al.*,2004). The syntheses of such complexes acting as model compounds for hydrolytic metalloenzymes have attracted much attention (Li *et al.*,2002). In present work, we report the syntheses and the crystal structure of the title compound, $[Ni(C_7H_{19}N_3O)_2](ClO_4)_2$,(I).

The Ni(II) located on an inversion center, is coordinated octahedrally by six N-atoms from the two ligands, forming a centrosymmetric distorted octahedral geometry. The bond lengths of Ni—N1, Ni—N2 and Ni—N3 are 2.170 (4), 2.128 (4) and 2.146 (3) Å., respectively.

One of the H atom attached to N2 is engaged in intermolecular hydrogen bonds with O1 (Table 1). These intermolecular hydrogen bonds result in the formation of one-dimensional chain structure along the *a* axis (Fig.2). The chains are further interconnected by the perchlorate anions through O—H···O and N—H···O hydrogen bonds to form a three-dimensional network.

Experimental

To a stirred solution of nickel perchlorate hexahydrate $(Ni(ClO)_4{\cdot}6H_2O$,0.366 g, 1.0 mmol) in methanol (20 ml), 1-[bis(aminoethyl)amino]-2-propanol ($C_7H_{19}N_3O$, 0.16 g, 1.0 mmol) in methanol (10 ml) was dropwise added in 10 min, then the solution was refluxed for 30 min. The green crystals of (I) suitable for X-ray diffraction were obtained after 3 days.

Refinement

All H atoms attached to C, N and O atoms were fixed geometrically and treated as riding with C—H = 0.97 Å (methylene), 0.96Å (methyl), 0.90Å (NH_2) and 0.82Å (OH) with $U_{iso}(H) = 1.2U_{eq}(CH_2, NH_2$ or OH) and $U_{iso}(H) = 1.5U_{eq}(CH_3)$.

One of the CH_2—CH_2 group is disordered over two positions in an approximately 0.6/0.4 ratio·This disorder was treated using the tools available in *SHELXL97* (Sheldrick, 1997).

Figures

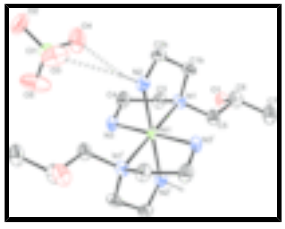

Fig. 1. Molecular structure showing the atom labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Only the major component of the CH_2—CH_2 group is shown. Hydrogen atoms have been ommited for clarity except the one showing some hydrogen bonds with the ClO_4 anion. [Symmetry code: (i) $1-x, 1-y, 1-z$]

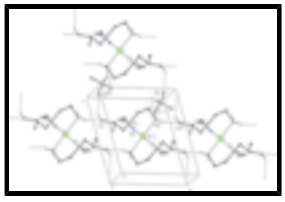

Fig. 2. Partial packing view of (I) showing the hydrogen bonding interactions. Hydrogen bonds are shown as dashed lines. Hydrogen atoms not involved in hydrogen bonding have been ommited for clarity·[Symmetry codes: (iii) −*x* + 1, −*y* + 1, −*z* + 2; (ii) *x* + 1, *y*, *z*]

Bis{1-[bis(2-aminoethyl)amino]propan-2-ol}nickel(II) bis(perchlorate)

Crystal data

$[Ni(C_7H_{19}N_3O)_2](ClO_4)_2$	F_{000} = 612
M_r = 580.11	D_x = 1.613 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2626 reflections
a = 7.915 (3) Å	θ = 2.4–26.9º
b = 17.243 (6) Å	μ = 1.10 mm^{-1}
c = 9.127 (3) Å	*T* = 298 (2) K
β = 106.442 (4)º	Prism, green
V = 1194.6 (7) Å^3	0.62 × 0.54 × 0.18 mm
Z = 2	

Data collection

Bruker SMART 1000 diffractometer	2796 independent reflections
Radiation source: fine-focus sealed tube	2060 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.044
T = 298(2) K	θ_{max} = 28.4º
φ and ω scans	θ_{min} = 2.4º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −10→6
T_{min} = 0.550, T_{max} = 0.827	*k* = −22→22
7286 measured reflections	*l* = −11→11

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.057	H-atom parameters constrained
$wR(F^2)$ = 0.167	$w = 1/[\sigma^2(F_o^2) + (0.0867P)^2 + 1.1111P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.04	$(\Delta/\sigma)_{max}$ = 0.001
2796 reflections	$\Delta\rho_{max}$ = 0.94 e Å^{-3}
173 parameters	$\Delta\rho_{min}$ = −0.62 e Å^{-3}

14 restraints
Primary atom site location: structure-invariant direct methods

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 $F^2 > 2\text{sigma}(F^2)$ 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}	Occ. (<1)
Ni1	0.5000	0.5000	0.5000	0.0300 (2)	
N1	0.3581 (4)	0.55710 (18)	0.6413 (4)	0.0408 (7)	
C1A	0.4904 (17)	0.5660 (10)	0.7986 (16)	0.081 (7)	0.38
H1A1	0.4534	0.6075	0.8541	0.097*	0.38
H1A2	0.4951	0.5184	0.8563	0.097*	0.38
C2A	0.6715 (14)	0.5839 (7)	0.7814 (15)	0.042 (3)	0.38
H2A1	0.6690	0.6319	0.7255	0.051*	0.38
H2A2	0.7568	0.5890	0.8809	0.051*	0.38
N2	0.7189 (4)	0.51711 (19)	0.6953 (4)	0.0428 (8)	
H2A	0.7392	0.4742	0.7538	0.051*	
H2B	0.8163	0.5281	0.6670	0.051*	
C1B	0.4976 (10)	0.6000 (5)	0.7595 (10)	0.050 (2)	0.62
H1B1	0.5278	0.6471	0.7144	0.060*	0.62
H1B2	0.4508	0.6149	0.8428	0.060*	0.62
C2B	0.6606 (13)	0.5528 (6)	0.8216 (9)	0.054 (2)	0.62
H2B1	0.7537	0.5856	0.8826	0.065*	0.62
H2B2	0.6376	0.5123	0.8872	0.065*	0.62
N3	0.4179 (4)	0.39852 (18)	0.5974 (4)	0.0421 (7)	
H3A	0.3418	0.3712	0.5234	0.051*	
H3B	0.5121	0.3683	0.6386	0.051*	
O1	−0.0021 (4)	0.63113 (18)	0.6513 (4)	0.0593 (8)	
H1	−0.0445	0.6570	0.7075	0.089*	
C3	0.2468 (6)	0.4976 (2)	0.6853 (6)	0.0512 (10)	
H3C	0.1373	0.4928	0.6044	0.061*	
H3D	0.2182	0.5146	0.7767	0.061*	
C4	0.3337 (7)	0.4194 (3)	0.7147 (6)	0.0579 (12)	
H4A	0.4214	0.4199	0.8135	0.069*	
H4B	0.2462	0.3806	0.7178	0.069*	
C5	0.2437 (6)	0.6200 (2)	0.5482 (5)	0.0510 (10)	
H5A	0.3185	0.6523	0.5056	0.061*	

H5B	0.1593	0.5951	0.4629	0.061*
C6	0.1434 (6)	0.6724 (3)	0.6229 (6)	0.0540 (11)
H6	0.2218	0.6902	0.7206	0.065*
C7	0.0782 (7)	0.7419 (3)	0.5227 (7)	0.0688 (14)
H7A	0.0052	0.7249	0.4251	0.103*
H7B	0.1769	0.7703	0.5091	0.103*
H7C	0.0109	0.7747	0.5702	0.103*
Cl1	0.87920 (13)	0.32829 (6)	0.91703 (10)	0.0455 (3)
O2	0.9702 (7)	0.2862 (3)	1.0475 (5)	0.0985 (15)
O3	0.9946 (5)	0.3676 (3)	0.8507 (5)	0.0966 (15)
O4	0.7661 (7)	0.3822 (3)	0.9591 (6)	0.1084 (16)
O5	0.7691 (8)	0.2780 (4)	0.8070 (6)	0.131 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0237 (3)	0.0276 (3)	0.0384 (4)	−0.0022 (2)	0.0081 (2)	0.0034 (2)
N1	0.0303 (15)	0.0401 (17)	0.0543 (19)	−0.0027 (13)	0.0160 (14)	−0.0064 (15)
C1A	0.064 (10)	0.089 (14)	0.112 (16)	−0.042 (10)	0.061 (11)	−0.066 (12)
C2A	0.031 (5)	0.052 (8)	0.041 (7)	−0.009 (6)	0.006 (5)	−0.013 (6)
N2	0.0310 (16)	0.0457 (18)	0.0488 (18)	0.0005 (13)	0.0066 (14)	0.0035 (14)
C1B	0.042 (4)	0.057 (5)	0.046 (4)	−0.008 (3)	0.003 (3)	−0.022 (4)
C2B	0.061 (5)	0.057 (5)	0.035 (4)	0.006 (4)	−0.002 (3)	−0.005 (4)
N3	0.0395 (17)	0.0314 (15)	0.0574 (19)	−0.0027 (13)	0.0167 (15)	0.0060 (14)
O1	0.0466 (17)	0.0539 (18)	0.092 (2)	−0.0103 (14)	0.0423 (17)	−0.0096 (17)
C3	0.045 (2)	0.054 (2)	0.063 (3)	0.0052 (19)	0.030 (2)	0.013 (2)
C4	0.063 (3)	0.051 (2)	0.070 (3)	0.003 (2)	0.034 (2)	0.015 (2)
C5	0.056 (3)	0.043 (2)	0.061 (3)	0.0048 (19)	0.029 (2)	0.0029 (19)
C6	0.041 (2)	0.055 (3)	0.073 (3)	−0.004 (2)	0.026 (2)	−0.001 (2)
C7	0.067 (3)	0.051 (3)	0.100 (4)	0.013 (2)	0.043 (3)	0.009 (3)
Cl1	0.0399 (5)	0.0540 (6)	0.0414 (5)	0.0024 (4)	0.0095 (4)	0.0041 (4)
O2	0.118 (4)	0.099 (3)	0.074 (2)	0.045 (3)	0.021 (3)	0.036 (2)
O3	0.069 (2)	0.147 (4)	0.079 (2)	−0.032 (3)	0.030 (2)	0.018 (3)
O4	0.112 (4)	0.093 (3)	0.137 (4)	0.042 (3)	0.062 (3)	0.013 (3)
O5	0.115 (4)	0.186 (5)	0.097 (3)	−0.078 (4)	0.039 (3)	−0.062 (4)

Geometric parameters (Å, °)

Ni1—N2^{i}	2.125 (3)	C2B—H2B2	0.9700
Ni1—N2	2.125 (3)	N3—C4	1.457 (6)
Ni1—N3	2.145 (3)	N3—H3A	0.9000
Ni1—N3^{i}	2.145 (3)	N3—H3B	0.9000
Ni1—N1^{i}	2.171 (3)	O1—C6	1.438 (5)
Ni1—N1	2.171 (3)	O1—H1	0.8200
N1—C3	1.480 (5)	C3—C4	1.502 (6)
N1—C1B	1.502 (7)	C3—H3C	0.9700
N1—C5	1.511 (5)	C3—H3D	0.9700
N1—C1A	1.526 (14)	C4—H4A	0.9700

C1A—C2A	1.517 (13)	C4—H4B	0.9700
C1A—H1A1	0.9700	C5—C6	1.490 (6)
C1A—H1A2	0.9700	C5—H5A	0.9700
C2A—N2	1.501 (11)	C5—H5B	0.9700
C2A—H2A1	0.9700	C6—C7	1.507 (7)
C2A—H2A2	0.9700	C6—H6	0.9800
N2—C2B	1.490 (9)	C7—H7A	0.9600
N2—H2A	0.9000	C7—H7B	0.9600
N2—H2B	0.9000	C7—H7C	0.9600
C1B—C2B	1.494 (10)	Cl1—O3	1.405 (4)
C1B—H1B1	0.9700	Cl1—O2	1.406 (4)
C1B—H1B2	0.9700	Cl1—O4	1.417 (4)
C2B—H2B1	0.9700	Cl1—O5	1.423 (5)
$N2^{i}$—Ni1—N2	180.000 (1)	N1—C1B—H1B2	109.1
$N2^{i}$—Ni1—N3	87.91 (13)	H1B1—C1B—H1B2	107.8
N2—Ni1—N3	92.09 (13)	N2—C2B—C1B	110.7 (6)
$N2^{i}$—Ni1—$N3^{i}$	92.09 (13)	N2—C2B—H2B1	109.5
N2—Ni1—$N3^{i}$	87.91 (13)	C1B—C2B—H2B1	109.5
N3—Ni1—$N3^{i}$	180.00 (9)	N2—C2B—H2B2	109.5
$N2^{i}$—Ni1—$N1^{i}$	82.81 (13)	C1B—C2B—H2B2	109.5
N2—Ni1—$N1^{i}$	97.19 (13)	H2B1—C2B—H2B2	108.1
N3—Ni1—$N1^{i}$	98.31 (12)	C4—N3—Ni1	111.0 (2)
$N3^{i}$—Ni1—$N1^{i}$	81.69 (12)	C4—N3—H3A	109.4
$N2^{i}$—Ni1—N1	97.19 (13)	Ni1—N3—H3A	109.4
N2—Ni1—N1	82.81 (13)	C4—N3—H3B	109.4
N3—Ni1—N1	81.69 (12)	Ni1—N3—H3B	109.4
$N3^{i}$—Ni1—N1	98.31 (12)	H3A—N3—H3B	108.0
$N1^{i}$—Ni1—N1	180.000 (1)	C6—O1—H1	109.5
C3—N1—C1B	121.3 (5)	N1—C3—C4	113.1 (4)
C3—N1—C5	110.0 (3)	N1—C3—H3C	109.0
C1B—N1—C5	104.6 (4)	C4—C3—H3C	109.0
C3—N1—C1A	96.7 (6)	N1—C3—H3D	109.0
C1B—N1—C1A	26.6 (5)	C4—C3—H3D	109.0
C5—N1—C1A	126.8 (7)	H3C—C3—H3D	107.8
C3—N1—Ni1	107.1 (2)	N3—C4—C3	111.8 (4)
C1B—N1—Ni1	104.5 (4)	N3—C4—H4A	109.2
C5—N1—Ni1	108.7 (2)	C3—C4—H4A	109.2
C1A—N1—Ni1	105.7 (6)	N3—C4—H4B	109.2
C2A—C1A—N1	109.9 (10)	C3—C4—H4B	109.2
C2A—C1A—H1A1	109.7	H4A—C4—H4B	107.9
N1—C1A—H1A1	109.7	C6—C5—N1	119.3 (4)
C2A—C1A—H1A2	109.7	C6—C5—H5A	107.5
N1—C1A—H1A2	109.7	N1—C5—H5A	107.5
H1A1—C1A—H1A2	108.2	C6—C5—H5B	107.5
N2—C2A—C1A	106.3 (10)	N1—C5—H5B	107.5
N2—C2A—H2A1	110.5	H5A—C5—H5B	107.0

C1A—C2A—H2A1	110.5	O1—C6—C5	109.9 (4)
N2—C2A—H2A2	110.5	O1—C6—C7	110.4 (4)
C1A—C2A—H2A2	110.5	C5—C6—C7	109.8 (4)
H2A1—C2A—H2A2	108.7	O1—C6—H6	108.9
C2B—N2—C2A	25.8 (4)	C5—C6—H6	108.9
C2B—N2—Ni1	110.4 (4)	C7—C6—H6	108.9
C2A—N2—Ni1	106.3 (5)	C6—C7—H7A	109.5
C2B—N2—H2A	85.6	C6—C7—H7B	109.5
C2A—N2—H2A	110.5	H7A—C7—H7B	109.5
Ni1—N2—H2A	110.5	C6—C7—H7C	109.5
C2B—N2—H2B	127.8	H7A—C7—H7C	109.5
C2A—N2—H2B	110.5	H7B—C7—H7C	109.5
Ni1—N2—H2B	110.5	O3—Cl1—O2	112.0 (3)
H2A—N2—H2B	108.7	O3—Cl1—O4	109.8 (3)
C2B—C1B—N1	112.6 (7)	O2—Cl1—O4	108.7 (3)
C2B—C1B—H1B1	109.1	O3—Cl1—O5	109.5 (3)
N1—C1B—H1B1	109.1	O2—Cl1—O5	110.3 (4)
C2B—C1B—H1B2	109.1	O4—Cl1—O5	106.5 (4)

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*
N2—H2A···O4	0.90	2.42	3.293 (6)	164
N2—H2A···O3	0.90	2.69	3.416 (6)	139
N2—H2B···O1^{ii}	0.90	2.31	3.067 (4)	141
O1—H1···O2^{iii}	0.82	2.36	3.043 (6)	142

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

Fig. 1

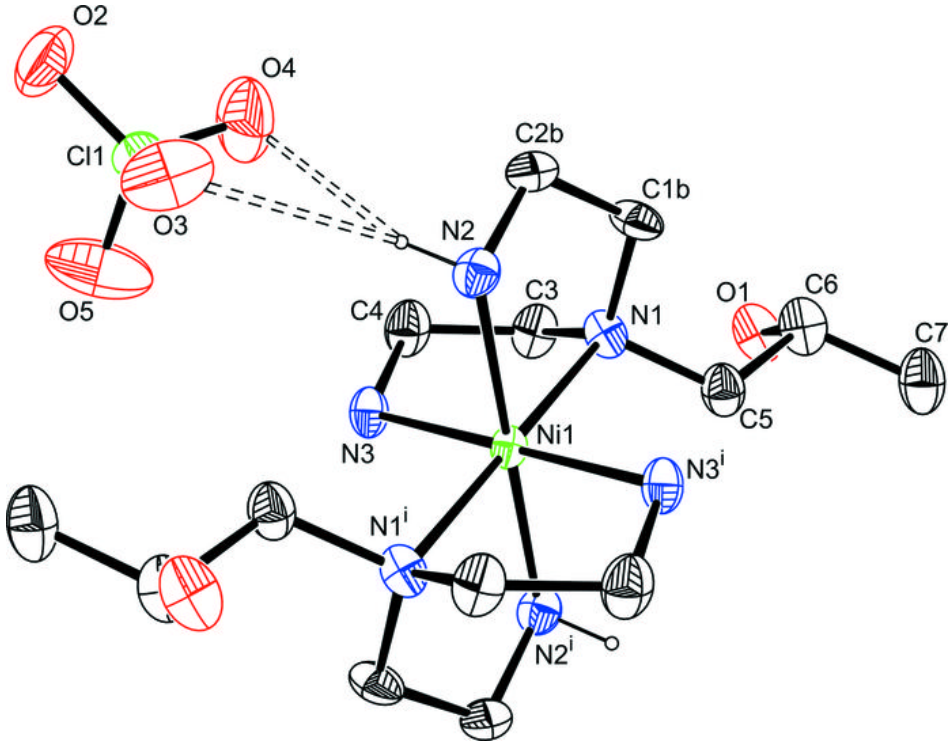

Fig. 2