Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(2-phenylethylammonium) tetraaquadichloridonickel(II) dichloride dihydrate

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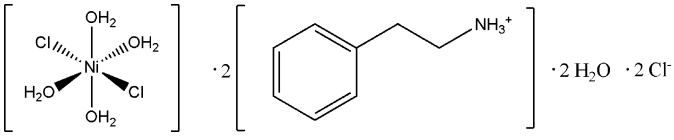
Received 13 August 2007; accepted 21 August 2007

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; R factor = 0.041; wR factor = 0.100; data-to-parameter ratio = 15.4.

The crystal structure of the title compound, $(C_8H_9NH_3)_2[NiCl_2(H_2O)_4]Cl_2\cdot 2H_2O$, was determined at 100 K. The nickel(II) ion is surrounded by an octahedron in which four water molecules and two Cl atoms are coordinated. These freestanding octahedra form a hydrogen-bonding network with the ammonium group of the 2-phenylethylammonium cations and the solvent water molecules and chloride ions. This results in sheets of freestanding octahedra alternating with layers of organic material.

Related literature

The crystal structure of the organic part of the title compound was published by Arkenbout *et al.* (2007). Other examples of organic–inorganic hybrids have been discussed by Papavassiliou (1997) and Mitzi (1999).



Experimental

Crystal data

$(C_8H_{12}N)_2[NiCl_2(H_2O)_4]Cl_2\cdot 2H_2O$
M_r = 552.98
Monoclinic, $P2_1/c$
a = 7.6977 (10) Å
b = 29.540 (4) Å
c = 10.7808 (15) Å
β = 90.760 (2)°
V = 2451.2 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.26 mm^{-1}
T = 100 (1) K
0.36 × 0.15 × 0.04 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
T_{min} = 0.625, T_{max} = 0.951
17481 measured reflections
4617 independent reflections
2752 reflections with $I > 2\sigma(I)$
R_{int} = 0.071

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.100
S = 0.96
4617 reflections
300 parameters
12 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6···Cl4	0.85 (2)	2.39 (2)	3.213 (2)	164 (3)
O6—H6′···Cl11	0.84 (2)	2.48 (3)	3.255 (2)	154 (2)
O11—H11···Cl5^{i}	0.84 (3)	2.36 (3)	3.190 (3)	172 (3)
O11—H11′···O7^{ii}	0.83 (3)	1.97 (3)	2.785 (4)	169 (3)
O12—H12···Cl4^{iii}	0.84 (2)	2.40 (3)	3.205 (3)	163 (3)
O12—H12′···Cl5^{iv}	0.83 (3)	2.30 (3)	3.125 (3)	168 (3)
O13—H13···Cl5	0.84 (3)	2.36 (3)	3.185 (3)	171 (3)
O13—H13′···Cl4^{v}	0.83 (3)	2.33 (3)	3.097 (3)	153 (3)
O14—H14···O6^{v}	0.84 (3)	1.97 (3)	2.800 (4)	176 (3)
O14—H14′···Cl4	0.84 (3)	2.37 (3)	3.198 (3)	169 (3)
N2—H20···Cl11^{vi}	0.91	2.46	3.285 (3)	151
N2—H20···O12^{vi}	0.91	2.56	3.244 (4)	133
N2—H20′···Cl4^{vi}	0.91	2.55	3.255 (3)	134
N2—H20′···Cl12^{v}	0.91	2.76	3.406 (3)	129
N2—H20″···O6	0.91	1.99	2.887 (3)	170
N3—H30···O7^{ii}	0.91	2.00	2.893 (3)	166
N3—H30′···Cl12^{i}	0.91	2.51	3.300 (3)	146
N3—H30′···O13^{i}	0.91	2.48	3.182 (4)	134
N3—H30″···Cl5	0.91	2.47	3.235 (3)	141

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

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 2006); data reduction: *SAINT-Plus*; program(s) used to solve structure: *DIRDIF99* (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006) and *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

The authors thank J. Baas for technical assistance, O. D. Jurchescu for discussions and the NanoNed for funding.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2059).

References

Arkenbout, A. H., Meetsma, A. & Palstra, T. T. M. (2007). *Acta Cryst.* E**63**, o2987.
Beurskens, P. T., Beurskens, G., de Gelder, R., García-Granda, S., Gould, R. O., Israël, R. & Smits, J. M. M. (1999). *The DIRDIF99 Program System.* Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.
Brandenburg, K. (2006). *DIAMOND*. Release 3.1. Crystal Impact GbR, Bonn, Germany.
Bruker (2006). *SMART* (Version 5.632), *SAINT-Plus* (Version 6.45) and *SADABS* (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Mitzi, D. B. (1999). *Prog. Inorg. Chem.* **48**, 1–121.
Papavassiliou, G. C. (1997). *Prog. Solid State Chem.* **25**, 125–270.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

 doi:10.1107/S160053680704130X

supplementary materials

Acta Cryst. (2007). E**63**, m2421 [**doi:10.1107/S160053680704130X**]

Bis(2-phenylethylammonium) tetraaquadichloridonickel(II) dichloride dihydrate

A. H. Arkenbout, A. Meetsma and T. T. M. Palstra

Comment

There is an interest to develop new multifunctional semiconductors that are easy to process and contain magnetic components. We present here the crystal structure of the title compound, which is a solution grown system build of organic conjugated molecules and paramagnetic Ni(II) ions. Every nickel(II) ion is surrounded by an octahedron in which 4 water molecules and 2 chlorine atoms are present. These octahedra are part of a complex network of hydrogen bonds between the ammonium groups, the water molecules and the free clorine ions, which stabilize this structure. The blue crystals of the title compound loose 20% of their weight when heated up to 350 K, leaving a pink amorphous powder, suggesting the evaporation of the crystal water. The material is paramagnetic down to 5 K. This lack of magnetic ordering can be explained by the large separation between the magnetic ions (d>6.631 Å) due to which the magnetic interactions are small and thus no magnetic ordering occurs.

Experimental

The organic part was synthesized as described as in Arkenbout *et al.* (2007). The (2-phenylethyl)ammonium chloride was dissolved in water with $NiCl_2{\cdot}6H_2O$ in an approximate molar ratio of 2:1 with a slight excess of the Ni compound to account for the difference in solubility. After slow evaporation of the water blue crystals were obtained.

Refinement

The hydrogen atoms of the C—H and N—H bonds were generated by geometrical considerations, constrained to idealized geometries, and allowed to ride on the carrier atoms with an isotropic displacement parameter related to the equivalent displacement parameter of their carrier atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$ or $1.5U_{eq}$(methyl C and ammonium). The methyl-groups and ammonium were refined as rigid groups, which were allowed to rotate freely. Assigned values of bond distances: secondary C—H_2 = 0.99 Å, methyl C—H_3 = 0.98 Å and aromatic C—H = 0.95 Å, N—H = 0.91 Å. The hydrogen atoms of the O—H bonds were restrained to a target value of 0.84 (2) Å and their isotropic displacement parameters were set to 1.5 times the U_{eq} value of their parent atom.

Figures

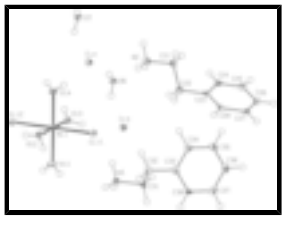


Fig. 1. Perspective view of all the moieties of the asymmetric unit illustrating the configuration and the adopted numbering scheme. Displacement ellipsoids for non H-atoms are represented at 50% probability level. The H-atoms are drawn with arbitrary radii.

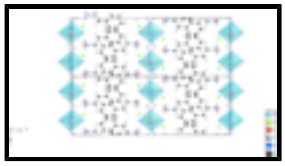


Fig. 2. Polyhedral drawing of the crystal structure viewed down the *a* axis.

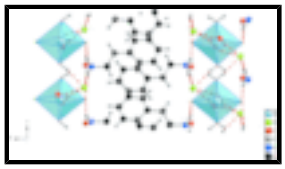


Fig. 3. Polyhedral drawing of a portion of the crystal packing. The hydrogen bonds (red dashed lines) connect the different parts in the crystal structure.

Bis(2-phenylethylammonium) tetraaquadichloridonickel(II) dichloride dihydrate

Crystal data

$(C_8H_{12}N_1)_2[NiCl_2(H_2O)_4]Cl_2{\cdot}2H_2O$	F_{000} = 1160
M_r = 552.98	The final unit cell was obtained from the xyz centroids of 3790 reflections after integration using the SAINTPLUS software package (Bruker, 2000). Reduced cell calculations did not indicate any higher metric lattice symmetry and examination of the finalatomic coordinates of the structure did not yield extra symmetry elements [Spek, A. L. (1988). J. Appl. Cryst. 21, 578–579; Le Page, Y. (1987). J. Appl. Cryst. 20, 264–269; Le Page, Y. (1988). J. Appl. Cryst. 21, 983–984]
Monoclinic, $P2_1/c$	D_x = 1.498 Mg m^{-3}
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.6977 (10) Å	Cell parameters from 3790 reflections
b = 29.540 (4) Å	θ = 2.8–28.3º
c = 10.7808 (15) Å	μ = 1.26 mm^{-1}
β = 90.760 (2)º	T = 100 (1) K
V = 2451.2 (6) Å^3	Platelet, green
Z = 4	0.36 × 0.15 × 0.04 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4617 independent reflections
Radiation source: fine focus sealed Siemens Mo tube	2752 reflections with $I > 2\sigma(I)$
Monochromator: parallel mounted graphite	R_{int} = 0.071
Detector resolution: 66.06 pixels mm^{-1}	θ_{max} = 25.7º
T = 100(1) K	θ_{min} = 2.7º
φ and ω scans	h = −9→9
Absorption correction: multi-scan (SADABS; Bruker, 2006)	k = −36→36
T_{min} = 0.625, T_{max} = 0.951	l = −13→12
17481 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: structure-invariant direct methods
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.100$	$w = 1/[\sigma^2(F_o^2)]$
$S = 0.96$	$(\Delta/\sigma)_{max} < 0.001$
4617 reflections	$\Delta\rho_{max} = 0.49$ e Å^{-3}
300 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}
12 restraints	Extinction correction: none
Primary atom site location: heavy-atom method	

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > 2$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	0.75797 (5)	0.00017 (2)	0.25209 (4)	0.0106 (1)
Cl11	0.73394 (10)	0.08087 (3)	0.25394 (8)	0.0145 (3)
Cl12	0.78520 (10)	−0.08070 (3)	0.25237 (8)	0.0137 (3)
O11	0.7986 (3)	0.00252 (9)	0.0639 (3)	0.0234 (10)
O12	1.0194 (3)	0.00810 (8)	0.2918 (3)	0.0164 (9)
O13	0.4969 (3)	−0.00747 (8)	0.2107 (3)	0.0176 (9)
O14	0.7171 (3)	−0.00256 (9)	0.4403 (2)	0.0200 (9)
H11	0.788 (5)	−0.0197 (10)	0.016 (3)	0.0352*
H11'	0.828 (5)	0.0248 (9)	0.022 (3)	0.0352*
H12	1.069 (4)	−0.0170 (7)	0.298 (4)	0.0247*
H12'	1.075 (4)	0.0239 (11)	0.242 (3)	0.0247*
H13	0.441 (4)	0.0152 (8)	0.186 (4)	0.0263*
H13'	0.446 (4)	−0.0198 (12)	0.269 (3)	0.0263*
H14	0.678 (5)	−0.0252 (9)	0.477 (3)	0.0301*
H14'	0.711 (5)	0.0200 (9)	0.487 (3)	0.0301*
N2	0.0460 (3)	0.09932 (10)	0.4585 (3)	0.0143 (10)
C21	0.0035 (4)	0.14876 (12)	0.4579 (3)	0.0154 (11)
C22	0.0760 (4)	0.17128 (12)	0.3429 (3)	0.0169 (11)

C23	0.0250 (4)	0.22067 (12)	0.3331 (3)	0.0129 (11)
C24	0.0765 (4)	0.25214 (12)	0.4228 (3)	0.0149 (11)
C25	0.0319 (4)	0.29747 (12)	0.4107 (4)	0.0175 (11)
C26	−0.0663 (4)	0.31217 (12)	0.3092 (3)	0.0169 (11)
C27	−0.1209 (4)	0.28086 (12)	0.2206 (3)	0.0148 (11)
C28	−0.0751 (4)	0.23574 (12)	0.2326 (3)	0.0144 (11)
H20	−0.01472	0.08523	0.39662	0.0214*
H20'	0.01669	0.08712	0.53282	0.0214*
H20"	0.16185	0.09556	0.44631	0.0214*
H21	0.05358	0.16323	0.53307	0.0183*
H21'	−0.12417	0.15281	0.45933	0.0183*
H22	0.20429	0.16891	0.34504	0.0202*
H22'	0.03305	0.15500	0.26834	0.0202*
H24	0.14288	0.24245	0.49283	0.0180*
H25	0.06852	0.31859	0.47217	0.0211*
H26	−0.09571	0.34325	0.30042	0.0202*
H27	−0.18983	0.29046	0.15178	0.0175*
H28	−0.11246	0.21468	0.17118	0.0171*
N3	0.5437 (3)	0.09956 (10)	−0.0599 (3)	0.0155 (10)
C31	0.5003 (4)	0.14887 (12)	−0.0633 (3)	0.0150 (11)
C32	0.5800 (4)	0.17265 (12)	0.0481 (3)	0.0184 (11)
C33	0.5255 (4)	0.22157 (12)	0.0587 (3)	0.0145 (11)
C34	0.4268 (4)	0.23574 (12)	0.1585 (3)	0.0149 (11)
C35	0.3802 (4)	0.28090 (12)	0.1721 (3)	0.0155 (11)
C36	0.4301 (4)	0.31266 (13)	0.0857 (3)	0.0181 (11)
C37	0.5282 (4)	0.29877 (12)	−0.0163 (3)	0.0158 (11)
C38	0.5753 (4)	0.25395 (12)	−0.0296 (3)	0.0132 (11)
H30	0.66040	0.09592	−0.06816	0.0231*
H30'	0.48728	0.08513	−0.12316	0.0231*
H30"	0.50999	0.08761	0.01377	0.0231*
H31	0.37259	0.15280	−0.06313	0.0183*
H31'	0.54515	0.16247	−0.14048	0.0183*
H32	0.54601	0.15640	0.12429	0.0223*
H32'	0.70816	0.17119	0.04240	0.0223*
H34	0.39058	0.21422	0.21838	0.0176*
H35	0.31361	0.28998	0.24133	0.0184*
H36	0.39830	0.34353	0.09510	0.0219*
H37	0.56252	0.32035	−0.07660	0.0188*
H38	0.64177	0.24491	−0.09895	0.0158*
Cl4	0.72994 (10)	0.07568 (3)	0.64530 (8)	0.0146 (3)
Cl5	0.24825 (10)	0.07499 (3)	0.13985 (8)	0.0151 (3)
O6	0.4138 (3)	0.08084 (9)	0.4482 (2)	0.0155 (8)
H6	0.489 (3)	0.0848 (13)	0.505 (2)	0.0233*
H6'	0.468 (4)	0.0815 (12)	0.3807 (19)	0.0233*
O7	0.9133 (3)	0.08230 (9)	0.9544 (3)	0.0180 (9)
H7	0.977 (4)	0.0833 (13)	0.892 (2)	0.0271*
H7'	0.986 (4)	0.0897 (13)	1.009 (3)	0.0271*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N2	0.0120 (14)	0.0184 (19)	0.0124 (19)	−0.0021 (12)	0.0006 (12)	0.0001 (14)
C21	0.0134 (16)	0.014 (2)	0.019 (2)	0.0008 (14)	0.0032 (16)	−0.0017 (17)
C22	0.0158 (17)	0.019 (2)	0.016 (2)	−0.0001 (15)	0.0022 (16)	−0.0003 (17)
C23	0.0117 (16)	0.016 (2)	0.011 (2)	−0.0003 (14)	0.0047 (15)	−0.0018 (17)
C24	0.0138 (17)	0.020 (2)	0.011 (2)	0.0001 (15)	0.0003 (15)	0.0007 (18)
C25	0.0146 (17)	0.019 (2)	0.019 (2)	−0.0056 (15)	0.0019 (16)	−0.0027 (18)
C26	0.0169 (18)	0.018 (2)	0.016 (2)	0.0015 (15)	0.0031 (16)	0.0034 (18)
C27	0.0133 (17)	0.023 (2)	0.008 (2)	0.0002 (15)	−0.0003 (15)	0.0033 (17)
C28	0.0122 (16)	0.019 (2)	0.012 (2)	−0.0019 (15)	0.0043 (16)	−0.0037 (17)
N3	0.0160 (15)	0.0155 (18)	0.015 (2)	−0.0020 (12)	0.0018 (13)	−0.0008 (13)
C31	0.0148 (16)	0.015 (2)	0.015 (2)	0.0044 (15)	−0.0024 (15)	−0.0025 (17)
C32	0.0190 (18)	0.017 (2)	0.019 (2)	0.0013 (15)	−0.0014 (16)	−0.0002 (17)
C33	0.0114 (16)	0.020 (2)	0.012 (2)	−0.0020 (14)	−0.0047 (15)	−0.0036 (17)
C34	0.0125 (17)	0.019 (2)	0.013 (2)	−0.0011 (14)	−0.0034 (15)	0.0018 (17)
C35	0.0106 (16)	0.021 (2)	0.015 (2)	−0.0018 (14)	0.0008 (15)	−0.0034 (18)
C36	0.0151 (17)	0.018 (2)	0.021 (2)	0.0028 (15)	−0.0044 (16)	−0.0026 (18)
C37	0.0114 (17)	0.021 (2)	0.015 (2)	−0.0038 (14)	−0.0049 (15)	0.0037 (18)
C38	0.0117 (16)	0.020 (2)	0.008 (2)	0.0020 (14)	−0.0008 (15)	−0.0024 (17)
Ni1	0.0095 (2)	0.0126 (2)	0.0098 (3)	−0.0001 (2)	0.0005 (2)	0.0010 (2)
Cl11	0.0146 (4)	0.0134 (5)	0.0155 (6)	0.0001 (3)	−0.0002 (4)	0.0009 (4)
Cl12	0.0142 (4)	0.0132 (5)	0.0136 (5)	−0.0006 (3)	−0.0001 (4)	0.0009 (4)
O11	0.0407 (15)	0.0185 (17)	0.0113 (18)	−0.0108 (14)	0.0076 (13)	−0.0010 (14)
O12	0.0131 (12)	0.0171 (16)	0.0191 (18)	−0.0021 (10)	−0.0003 (11)	0.0022 (13)
O13	0.0137 (12)	0.0177 (16)	0.0214 (18)	0.0014 (11)	0.0005 (11)	0.0081 (13)
O14	0.0345 (14)	0.0130 (16)	0.0128 (18)	−0.0047 (13)	0.0066 (12)	0.0001 (14)
O6	0.0166 (13)	0.0197 (15)	0.0102 (16)	0.0001 (11)	−0.0008 (11)	−0.0002 (13)
O7	0.0130 (12)	0.0231 (16)	0.0181 (18)	−0.0001 (11)	0.0032 (11)	0.0012 (14)
Cl4	0.0131 (4)	0.0162 (5)	0.0144 (5)	−0.0013 (3)	0.0011 (4)	0.0001 (4)
Cl5	0.0143 (4)	0.0160 (5)	0.0150 (6)	−0.0005 (3)	0.0008 (4)	0.0004 (4)

Geometric parameters (Å, °)

Ni1—O13	2.065 (2)	C24—C25	1.388 (5)
Ni1—O14	2.059 (2)	C25—C26	1.391 (5)
Ni1—Cl12	2.3981 (11)	C26—C27	1.391 (5)
Ni1—O11	2.058 (3)	C27—C28	1.384 (5)
Ni1—Cl11	2.3912 (11)	C21—H21	0.9900
Ni1—O12	2.065 (2)	C21—H21'	0.9900
O11—H11	0.84 (3)	C22—H22'	0.9900
O11—H11'	0.83 (3)	C22—H22	0.9900
O12—H12	0.84 (2)	C24—H24	0.9500
O12—H12'	0.83 (3)	C25—H25	0.9500
O13—H13	0.84 (3)	C26—H26	0.9500
O13—H13'	0.83 (3)	C27—H27	0.9500
O14—H14'	0.84 (3)	C28—H28	0.9500

O14—H14	0.84 (3)	C31—C32	1.514 (5)
O6—H6	0.85 (2)	C32—C33	1.510 (5)
O6—H6'	0.84 (2)	C33—C38	1.406 (5)
O7—H7'	0.84 (3)	C33—C34	1.390 (5)
O7—H7	0.84 (3)	C34—C35	1.390 (5)
N2—C21	1.497 (5)	C35—C36	1.380 (5)
N2—H20"	0.9100	C36—C37	1.404 (5)
N2—H20	0.9100	C37—C38	1.381 (5)
N2—H20'	0.9100	C31—H31	0.9900
N3—C31	1.495 (5)	C31—H31'	0.9900
N3—H30	0.9100	C32—H32	0.9900
N3—H30'	0.9100	C32—H32'	0.9900
N3—H30"	0.9100	C34—H34	0.9500
C21—C22	1.520 (5)	C35—H35	0.9500
C22—C23	1.514 (5)	C36—H36	0.9500
C23—C28	1.394 (5)	C37—H37	0.9500
C23—C24	1.395 (5)	C38—H38	0.9500
Ni1···H30'^{i}	3.4300	C31···H38	3.0600
Ni1···H7^{ii}	3.57 (3)	C32···H28^{iii}	2.9700
Ni1···H20^{iii}	3.4300	C33···H28^{iii}	3.0300
Ni1···H6'	3.57 (3)	C34···H24^{ix}	2.8800
Cl4···N2^{iii}	3.255 (3)	C35···H24^{ix}	2.7300
Cl4···O14	3.198 (3)	C35···H31'^{viii}	2.9000
Cl4···O13^{iv}	3.097 (3)	C36···H21^{ix}	3.0300
Cl4···O6	3.213 (2)	C36···H27^{iii}	3.0700
Cl4···C21^{iii}	3.645 (3)	C36···H24^{ix}	2.9100
Cl5···N3	3.235 (3)	C37···H27^{iii}	2.8200
Cl5···O13	3.185 (3)	C37···H21'^{xi}	3.0500
Cl5···O11^{i}	3.190 (3)	C37···H22^{ix}	3.0400
Cl5···O12^{v}	3.125 (3)	C37···H34^{ix}	3.0600
Cl11···C21^{iii}	3.611 (3)	C38···H31'	2.9600
Cl11···O14	3.183 (3)	C38···H27^{iii}	2.8600
Cl11···O6	3.255 (2)	H6···Cl4	2.39 (2)
Cl11···N2^{iii}	3.285 (3)	H6···H36^{viii}	2.4400
Cl11···O13	3.215 (3)	H6···H14^{iv}	2.19 (5)
Cl11···O11	3.135 (3)	H6···H14'	2.58 (5)
Cl11···O12	3.097 (3)	H6'···H20"	2.5000
Cl12···O13	3.127 (3)	H6'···H14^{iv}	2.54 (4)
Cl12···O14	3.120 (3)	H6'···Cl11	2.48 (3)
Cl12···C31^{i}	3.595 (3)	H6'···Ni1	3.57 (3)
Cl12···N2^{iv}	3.406 (3)	H6'···Cl5	3.09 (2)
Cl12···O12	3.208 (3)	H11···Cl5^{i}	2.36 (3)
Cl12···N3^{i}	3.300 (3)	H11'···H7^{vii}	2.51 (5)
Cl12···O11	3.192 (3)	H11'···O7^{vii}	1.97 (3)

Cl12···O7^{ii}	3.240 (3)	H11'···H7''^{vii}	2.28 (5)
Cl4···H20''^{iii}	2.5500	H12···Cl4^{ii}	2.40 (3)
Cl4···H14'	2.37 (3)	H12'···Cl5^{iii}	2.30 (3)
Cl4···H6	2.39 (2)	H12'···H20^{iii}	2.5600
Cl4···H13''^{iv}	2.33 (3)	H13···Cl5	2.36 (3)
Cl5···H13	2.36 (3)	H13'···Cl4^{iv}	2.33 (3)
Cl5···H12''^{v}	2.30 (3)	H13'···H30''^{i}	2.5500
Cl5···H6'	3.09 (2)	H14···H6^{iv}	2.19 (5)
Cl5···H30''	2.4700	H14···O6^{iv}	1.97 (3)
Cl5···H11^{i}	2.36 (3)	H14···H6''^{iv}	2.54 (4)
Cl11···H20^{iii}	2.4600	H14···H20''^{iv}	2.5500
Cl11···H14'	3.10 (3)	H14'···Cl4	2.37 (3)
Cl11···H13	3.06 (3)	H14'···H6	2.58 (5)
Cl11···H6'	2.48 (3)	H20···Cl11^{v}	2.4600
Cl11···H30''	3.1000	H20···Ni1^{v}	3.4300
Cl11···H32	3.0000	H20···H22'	2.5100
Cl11···H12'	3.12 (3)	H20···H12''^{v}	2.5600
Cl11···H11'	3.09 (3)	H20···O12^{v}	2.5600
Cl12···H12	2.92 (3)	H20'···Cl4^{v}	2.5500
Cl12···H14	3.05 (3)	H20'···Cl12^{iv}	2.7600
Cl12···H20''^{iv}	2.7600	H20''···O6	1.9900
Cl12···H11	3.12 (3)	H20''···H6'	2.5000
Cl12···H7^{ii}	2.42 (3)	H20''···H22	2.4500
Cl12···H36^{vi}	3.1300	H20''···H14^{iv}	2.5500
Cl12···H30''^{i}	2.5100	H21···H24	2.4800
O6···O14^{iv}	2.800 (4)	H21···C27^{viii}	2.9500
O6···Cl4	3.213 (2)	H21···C36^{viii}	3.0300
O6···Cl11	3.255 (2)	H21···C24	2.8900
O6···N2	2.887 (3)	H21'···C37^{x}	3.0500
O11···O13	2.843 (4)	H21'···H37^{x}	2.5600
O11···O7^{vii}	2.785 (4)	H22···H20''	2.4500
O11···O12	2.974 (4)	H22···H34	2.4000
O11···Cl12	3.192 (3)	H22···C37^{viii}	3.0400
O11···Cl5^{i}	3.190 (3)	H22'···H20	2.5100
O11···Cl11	3.135 (3)	H22'···H28	2.3300
O12···N2^{iii}	3.244 (4)	H24···C34^{viii}	2.8800
O12···O14	2.859 (3)	H24···C35^{viii}	2.7300
O12···Cl4^{ii}	3.205 (3)	H24···C36^{viii}	2.9100
O12···Cl5^{iii}	3.125 (3)	H24···H21	2.4800
O12···O11	2.974 (4)	H24···C21	2.9900
O12···Cl11	3.097 (3)	H25···H31^{viii}	2.5200
O12···Cl12	3.208 (3)	H26···H7^{xii}	2.4500

O13···N3^{i}	3.182 (4)	H26···O7^{xii}	2.7600
O13···O14	2.985 (4)	H27···C38^{v}	2.8600
O13···Cl5	3.185 (3)	H27···C37^{v}	2.8200
O13···Cl12	3.127 (3)	H27···C36^{v}	3.0700
O13···Cl4^{iv}	3.097 (3)	H28···C32^{v}	2.9700
O13···Cl11	3.215 (3)	H28···H32'^{v}	2.3300
O13···O11	2.843 (4)	H28···H22'	2.3300
O14···Cl4	3.198 (3)	H28···C25^{ix}	3.0500
O14···O6^{iv}	2.800 (4)	H28···C33^{v}	3.0300
O14···Cl12	3.120 (3)	H30···O7^{vii}	2.0000
O14···O13	2.985 (4)	H30···H7^{vii}	2.5100
O14···Cl11	3.183 (3)	H30···H32'	2.5500
O14···O12	2.859 (3)	H30'···Ni1^{i}	3.4300
O6···H14^{iv}	1.97 (3)	H30'···H13"^{i}	2.5500
O6···H20"	1.9900	H30'···Cl12^{i}	2.5100
O6···H36^{viii}	2.7400	H30'···O13^{i}	2.4800
O11···H12'	2.92 (3)	H30'···Cl4^{vii}	3.1500
O12···H20^{iii}	2.5600	H30"···H32	2.3700
O13···H30"^{i}	2.4800	H30"···Cl11	3.1000
O14···H13'	2.82 (3)	H30"···Cl5	2.4700
N2···Cl12^{iv}	3.406 (3)	H31···H25^{ix}	2.5200
N2···O6	2.887 (3)	H31···C25^{ix}	3.0200
N2···O12^{v}	3.244 (4)	H31'···C35^{ix}	2.9000
N2···Cl11^{v}	3.285 (3)	H31'···C38	2.9600
N2···Cl4^{v}	3.255 (3)	H31'···H38	2.5800
N3···Cl12^{i}	3.300 (3)	H31'···H35^{ix}	2.5900
N3···O13^{i}	3.182 (4)	H32···H30"	2.3700
N3···Cl5	3.235 (3)	H32···H34	2.3300
N3···O7^{vii}	2.893 (3)	H32···Cl11	3.0000
C21···Cl11^{v}	3.611 (3)	H32'···C25^{xi}	3.0300
C21···Cl4^{v}	3.645 (3)	H32'···H28^{iii}	2.3300
C24···C28^{viii}	3.571 (5)	H32'···H30	2.5500
C28···C24^{ix}	3.571 (5)	H34···C37^{viii}	3.0600
C31···Cl12^{i}	3.595 (3)	H34···H32	2.3300
C34···C38^{viii}	3.550 (5)	H34···H22	2.4000
C38···C34^{ix}	3.550 (5)	H34···C22	3.0600
C21···H24	2.9900	H34···C23	3.0900
C22···H34	3.0600	H35···C26	3.0900
C23···H34	3.0900	H35···H31'^{viii}	2.5900
C24···H35	2.9200	H35···C24	2.9200
C24···H21	2.8900	H35···C25	2.8600
C25···H32'^{x}	3.0300	H36···O6^{ix}	2.7400

C25···H31^{viii}	3.0200	H36···Cl12^{xiii}	3.1300
C25···H28^{viii}	3.0500	H36···H6^{ix}	2.4400
C25···H35	2.8600	H37···H21'^{xi}	2.5600
C26···H38^{x}	2.9900	H38···C28^{xi}	2.9100
C26···H35	3.0900	H38···C27^{xi}	2.7900
C27···H21^{ix}	2.9500	H38···C31	3.0600
C27···H38^{x}	2.7900	H38···H31'	2.5800
C28···H38^{x}	2.9100	H38···C26^{xi}	2.9900
Cl12—Ni1—O12	91.59 (7)	H21—C21—H21'	108.00
Cl12—Ni1—O13	88.64 (7)	C22—C21—H21'	110.00
Cl12—Ni1—O14	88.52 (8)	N2—C21—H21	110.00
O11—Ni1—O12	92.30 (11)	C22—C21—H21	110.00
O11—Ni1—O13	87.18 (11)	N2—C21—H21'	110.00
O11—Ni1—O14	179.68 (11)	C23—C22—H22	109.00
O12—Ni1—O13	179.44 (12)	C21—C22—H22'	109.00
O12—Ni1—O14	87.79 (11)	C23—C22—H22'	109.00
O13—Ni1—O14	92.73 (11)	C21—C22—H22	109.00
Cl11—Ni1—Cl12	179.21 (3)	H22—C22—H22'	108.00
Cl11—Ni1—O11	89.28 (8)	C25—C24—H24	120.00
Cl11—Ni1—O12	87.73 (7)	C23—C24—H24	120.00
Cl11—Ni1—O13	92.04 (7)	C26—C25—H25	120.00
Cl11—Ni1—O14	91.03 (8)	C24—C25—H25	120.00
Cl12—Ni1—O11	91.17 (8)	C25—C26—H26	120.00
Ni1—O11—H11'	128 (2)	C27—C26—H26	120.00
H11—O11—H11'	108 (3)	C28—C27—H27	120.00
Ni1—O11—H11	125 (2)	C26—C27—H27	120.00
H12—O12—H12'	108 (3)	C27—C28—H28	119.00
Ni1—O12—H12	111 (2)	C23—C28—H28	119.00
Ni1—O12—H12'	116 (2)	N3—C31—C32	110.2 (3)
H13—O13—H13'	110 (3)	C31—C32—C33	113.2 (3)
Ni1—O13—H13'	111 (2)	C32—C33—C34	120.2 (3)
Ni1—O13—H13	118 (2)	C34—C33—C38	118.4 (3)
Ni1—O14—H14	124 (2)	C32—C33—C38	121.4 (3)
Ni1—O14—H14'	125 (2)	C33—C34—C35	121.0 (3)
H14—O14—H14'	109 (3)	C34—C35—C36	120.5 (3)
H6—O6—H6'	106 (2)	C35—C36—C37	119.2 (3)
H7—O7—H7'	99 (3)	C36—C37—C38	120.4 (3)
C21—N2—H20'	109.00	C33—C38—C37	120.5 (3)
C21—N2—H20"	109.00	C32—C31—H31	110.00
H20—N2—H20"	109.00	C32—C31—H31'	110.00
H20'—N2—H20"	110.00	N3—C31—H31	110.00
C21—N2—H20	109.00	N3—C31—H31'	110.00
H20—N2—H20'	109.00	H31—C31—H31'	108.00
C31—N3—H30"	109.00	H32—C32—H32'	108.00
H30—N3—H30'	109.00	C31—C32—H32'	109.00
H30'—N3—H30"	109.00	C31—C32—H32	109.00
C31—N3—H30	109.00	C33—C32—H32	109.00

H30—N3—H30"	109.00	C33—C32—H32'	109.00
C31—N3—H30'	110.00	C33—C34—H34	120.00
N2—C21—C22	110.4 (3)	C35—C34—H34	119.00
C21—C22—C23	112.4 (3)	C34—C35—H35	120.00
C24—C23—C28	118.4 (3)	C36—C35—H35	120.00
C22—C23—C28	120.1 (3)	C37—C36—H36	120.00
C22—C23—C24	121.5 (3)	C35—C36—H36	120.00
C23—C24—C25	120.7 (3)	C36—C37—H37	120.00
C24—C25—C26	120.4 (3)	C38—C37—H37	120.00
C25—C26—C27	119.3 (3)	C37—C38—H38	120.00
C26—C27—C28	120.1 (3)	C33—C38—H38	120.00
C23—C28—C27	121.2 (3)		
N2—C21—C22—C23	175.8 (2)	N3—C31—C32—C33	−173.6 (2)
C21—C22—C23—C24	62.1 (4)	C31—C32—C33—C34	115.0 (3)
C21—C22—C23—C28	−118.2 (3)	C31—C32—C33—C38	−66.3 (4)
C22—C23—C24—C25	178.5 (3)	C32—C33—C34—C35	177.9 (3)
C28—C23—C24—C25	−1.3 (5)	C38—C33—C34—C35	−0.9 (5)
C22—C23—C28—C27	−178.9 (3)	C32—C33—C38—C37	−178.2 (3)
C24—C23—C28—C27	0.8 (5)	C34—C33—C38—C37	0.6 (5)
C23—C24—C25—C26	0.5 (5)	C33—C34—C35—C36	0.6 (5)
C24—C25—C26—C27	0.7 (5)	C34—C35—C36—C37	0.2 (5)
C25—C26—C27—C28	−1.1 (5)	C35—C36—C37—C38	−0.5 (5)
C26—C27—C28—C23	0.4 (5)	C36—C37—C38—C33	0.1 (5)

Symmetry codes: (i) −*x*+1, −*y*, −*z*; (ii) −*x*+2, −*y*, −*z*+1; (iii) *x*+1, *y*, *z*; (iv) −*x*+1, −*y*, −*z*+1; (v) *x*−1, *y*, *z*; (vi) −*x*+1, *y*−1/2, −*z*+1/2; (vii) *x*, *y*, *z*−1; (viii) *x*, −*y*+1/2, *z*+1/2; (ix) *x*, −*y*+1/2, *z*−1/2; (x) *x*−1, −*y*+1/2, *z*+1/2; (xi) *x*+1, −*y*+1/2, *z*−1/2; (xii) *x*−1, −*y*+1/2, *z*−1/2; (xiii) −*x*+1, *y*+1/2, −*z*+1/2.

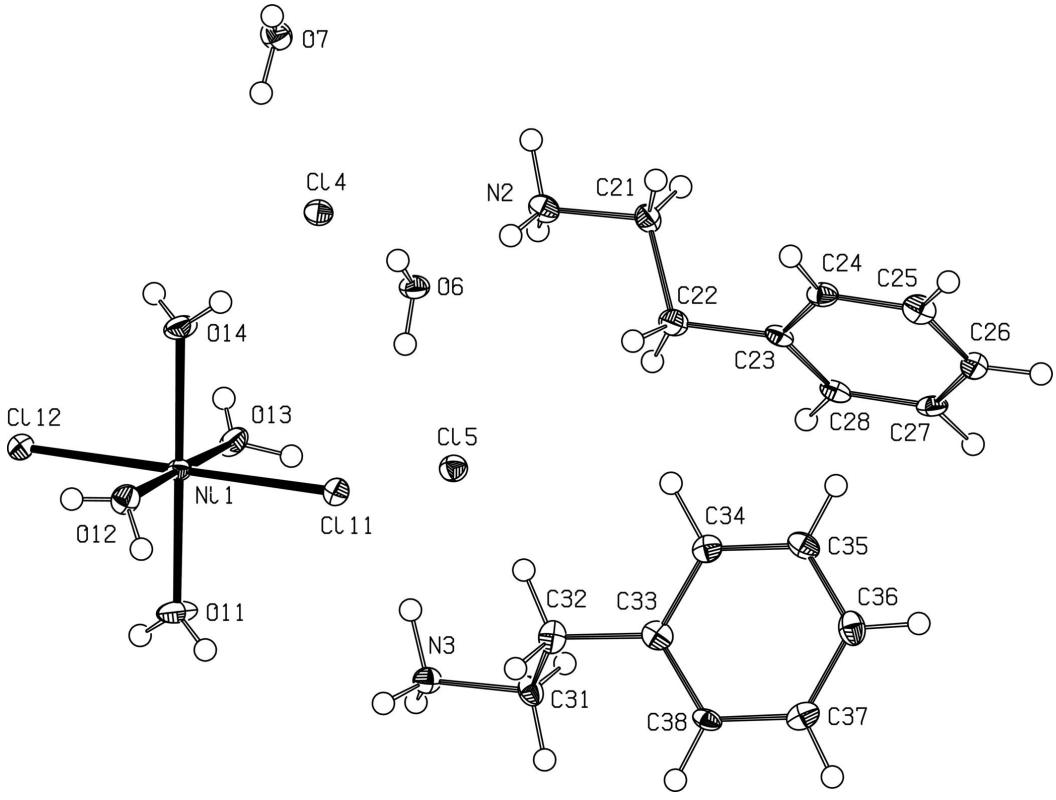
Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6···Cl4	0.85 (2)	2.39 (2)	3.213 (2)	164 (3)
O6—H6'···Cl11	0.84 (2)	2.48 (3)	3.255 (2)	154 (2)
O11—H11···Cl5^{i}	0.84 (3)	2.36 (3)	3.190 (3)	172 (3)
O11—H11'···O7^{vii}	0.83 (3)	1.97 (3)	2.785 (4)	169 (3)
O12—H12···Cl4^{ii}	0.84 (2)	2.40 (3)	3.205 (3)	163 (3)
O12—H12'···Cl5^{iii}	0.83 (3)	2.30 (3)	3.125 (3)	168 (3)
O13—H13···Cl5	0.84 (3)	2.36 (3)	3.185 (3)	171 (3)
O13—H13'···Cl4^{iv}	0.83 (3)	2.33 (3)	3.097 (3)	153 (3)
O14—H14···O6^{iv}	0.84 (3)	1.97 (3)	2.800 (4)	176 (3)
O14—H14'···Cl4	0.84 (3)	2.37 (3)	3.198 (3)	169 (3)
N2—H20···Cl11^{v}	0.9100	2.4600	3.285 (3)	151.00
N2—H20···O12^{v}	0.9100	2.5600	3.244 (4)	133.00
N2—H20'···Cl4^{v}	0.9100	2.5500	3.255 (3)	134.00
N2—H20'···Cl12^{iv}	0.9100	2.7600	3.406 (3)	129.00
N2—H20"···O6	0.9100	1.9900	2.887 (3)	170.00
N3—H30···O7^{vii}	0.9100	2.0000	2.893 (3)	166.00
N3—H30'···Cl12^{i}	0.9100	2.5100	3.300 (3)	146.00

N3—H30'···O13^{i}	0.9100	2.4800	3.182 (4)	134.00
N3—H30"···Cl5	0.9100	2.4700	3.235 (3)	141.00

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

Fig. 1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N2
C21
O6
C24
C25
C22
O14
C26
C23
Cl12
O13
C28
C27
Cl5
Nl1
C34
Cl11
O12
C35
C32
C33
C36
O11
N3
C31
C38
C37

Fig. 2

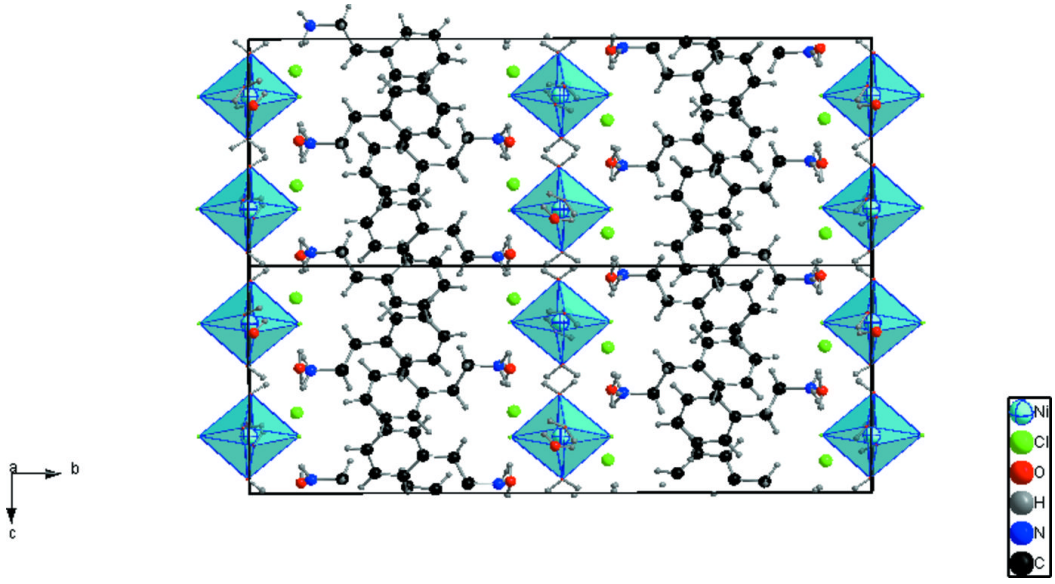

Fig. 3