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Triaquachloridobis[4-(dimethylamino)-benzaldehyde-κO]nickel(II) chloride

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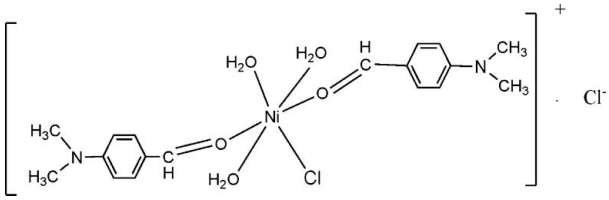
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.044; wR factor = 0.108; data-to-parameter ratio = 15.3.

In the title complex, $[NiCl(C_9H_{11}NO)_2(H_2O)_3]Cl$, the Ni^{II} ion is six-coordinated by one Cl atom [Ni—Cl = 2.3712 (11) Å], two O atoms from two 4-(dimethylamino)benzaldehyde (L) ligands and three water molecules in a distorted octahedral geometry [Ni—O = 2.076 (3)–2.094 (3) Å]. The mean planes of the two L ligands make a dihedral angle of 65.8 (1)°. In the crystal structure, intermolecular O—H···Cl hydrogen bonds link cations and anions into two-dimensional sheets parallel to the ab plane, with the L ligands protruding above and below.

Related literature

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



Experimental

Crystal data

$[NiCl(C_9H_{11}NO)_2(H_2O)_3]Cl$
M_r = 482.03
Monoclinic, $P2_1/n$
a = 11.632 (3) Å
b = 7.5534 (19) Å
c = 25.704 (6) Å
β = 102.87 (3)°
V = 2201.6 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.15 mm^{-1}
T = 298 (2) K
0.38 × 0.19 × 0.16 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.668, T_{max} = 0.837
9852 measured reflections
3861 independent reflections
2471 reflections with $I > 2\sigma(I)$
R_{int} = 0.097

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.109
S = 0.96
3861 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.44 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H19···Cl1^{i}	0.85	2.37	3.214 (3)	171
O3—H20···Cl2^{ii}	0.85	2.32	3.110 (3)	155
O4—H21···Cl1^{iii}	0.85	2.27	3.095 (3)	163
O5—H24···Cl2^{iv}	0.85	2.33	3.088 (3)	149

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: CV2311).

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supplementary materials

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Triaquachloridobis[4-(dimethylamino)benzaldehyde-*κO*]nickel(II) chloride

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Comment

Nickel is vital for organism as a trace element. It often acts as a component or secondary factor of enzyme. In the last two decades, biological inorganic chemistry of nickel has became one of the hottest fields and the coordination chemistry of nickel has made a great progress (Jiang *et al.*, 2003). We report here the synthesis and crystal structure of the title compound, a new nickel(II) complex.

The title complex (Fig.1) consists of cations $[Ni(C_9H_{11}NO)_2(H_2O)_3Cl]^+$ and non-coordinating chloride anions. The Ni^{II} ion is six-coordinated by one Cl atom, two O atoms of 4-dimethylaminobenzaldehyde molecules and three water molecules in a distorted octahedral geometry.

In the crystal, the intermolecular O—H···Cl hydrogen bonds (Table 1) link cations and anions into two-dimensional sheets parallel to *ab* plane with the up and down protruding ligands.

Experimental

Nickel chloride hexahydrate (5 mmol, 1189 mg) in absolute ethanol (15 ml) was added dropwise to an absolute ethanol solution (20 ml) of *P*-dimethylaminobenzaldehyde (10 mmol, 1492 mg). The mixture was heated under reflux with stirring for 4 h. The solution was kept at room temperature for three weeks, after which large green block-shaped crystals of the title complex suitable for X-ray diffraction analysis were obtained.

Refinement

All H-atoms were positioned geometrically (C—H 0.93–0.96 Å, O—H 0.85 Å) and refined using a riding model, with U_{iso} = 1.2–1.5U_{eq}(parent atom).

Figures

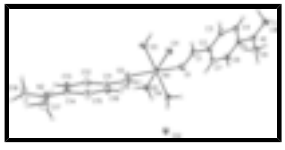


Fig. 1. The structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Triaquachloridobis[4-(dimethylamino)benzaldehyde-κ*O*]nickel(II) chloride

Crystal data

$[NiCl(C_9H_{11}NO)_2(H_2O)_3]Cl$ F_{000} = 1008

M_r = 482.03	D_x = 1.454 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3371 reflections
a = 11.632 (3) Å	θ = 2.1–27.5º
b = 7.5534 (19) Å	μ = 1.15 mm^{-1}
c = 25.704 (6) Å	T = 298 (2) K
β = 102.87 (3)º	Block, green
V = 2201.6 (9) Å^3	0.38 × 0.19 × 0.16 mm
Z = 4	

Data collection

Siemens SMART CCD area-detector diffractometer	3861 independent reflections
Radiation source: fine-focus sealed tube	2471 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.097
T = 298(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −10→13
T_{min} = 0.668, T_{max} = 0.837	k = −8→8
9852 measured reflections	l = −30→30

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H-atom parameters constrained
$wR(F^2)$ = 0.109	$w = 1/[\sigma^2(F_o^2) + (0.095P)^2 + 4.2002P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.96	$(\Delta/\sigma)_{max}$ = 0.001
3861 reflections	$\Delta\rho_{max}$ = 0.53 e Å^{-3}
253 parameters	$\Delta\rho_{min}$ = −0.44 e Å^{-3}
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 $F^2 > \sigma(F^2)$ 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	0.31610 (4)	0.73333 (6)	0.514421 (19)	0.02885 (16)
Cl1	0.52350 (7)	0.76902 (13)	0.54060 (4)	0.0355 (3)
Cl2	0.09692 (8)	0.27614 (14)	0.47263 (4)	0.0420 (3)
N1	0.3676 (3)	0.8312 (5)	0.19083 (13)	0.0357 (9)
N2	0.3835 (3)	0.6733 (5)	0.84909 (13)	0.0414 (9)
O1	0.3131 (2)	0.7215 (4)	0.43276 (10)	0.0409 (7)
O2	0.3021 (2)	0.7448 (3)	0.59345 (10)	0.0356 (7)
O3	0.2972 (2)	1.0016 (3)	0.51083 (10)	0.0415 (8)
H19	0.3510	1.0577	0.5002	0.062*
H20	0.2326	1.0483	0.4946	0.062*
O4	0.3347 (2)	0.4651 (3)	0.52158 (10)	0.0350 (7)
H21	0.3830	0.4208	0.5048	0.053*
H22	0.2718	0.4068	0.5097	0.053*
O5	0.1378 (2)	0.6939 (4)	0.49070 (10)	0.0405 (7)
H23	0.1152	0.5896	0.4811	0.061*
H24	0.0857	0.7427	0.5044	0.061*
C1	0.3864 (3)	0.7913 (5)	0.41131 (16)	0.0369 (10)
H1	0.4523	0.8416	0.4336	0.044*
C2	0.3790 (3)	0.8017 (5)	0.35487 (15)	0.0318 (10)
C3	0.4687 (3)	0.8831 (5)	0.33590 (16)	0.0364 (10)
H3A	0.5328	0.9302	0.3601	0.044*
C4	0.4649 (3)	0.8958 (5)	0.28259 (16)	0.0347 (10)
H4A	0.5259	0.9536	0.2715	0.042*
C5	0.3717 (3)	0.8239 (5)	0.24395 (16)	0.0307 (9)
C6	0.2794 (3)	0.7441 (5)	0.26374 (16)	0.0375 (11)
H6	0.2139	0.6993	0.2398	0.045*
C7	0.2852 (3)	0.7321 (5)	0.31661 (16)	0.0368 (10)
H7	0.2244	0.6752	0.3281	0.044*
C8	0.4634 (3)	0.9121 (6)	0.17189 (16)	0.0485 (12)
H8A	0.4644	1.0371	0.1787	0.073*
H8B	0.4524	0.8921	0.1342	0.073*
H8C	0.5368	0.8608	0.1901	0.073*
C9	0.2689 (4)	0.7645 (6)	0.15098 (15)	0.0533 (13)
H9A	0.2579	0.6410	0.1573	0.080*
H9B	0.2847	0.7795	0.1161	0.080*
H9C	0.1989	0.8287	0.1531	0.080*
C10	0.3730 (3)	0.6683 (5)	0.62962 (15)	0.0350 (10)
H10	0.4313	0.6008	0.6195	0.042*
C11	0.3740 (3)	0.6735 (5)	0.68512 (15)	0.0294 (9)
C12	0.2921 (3)	0.7697 (5)	0.70616 (15)	0.0331 (10)
H12	0.2340	0.8344	0.6832	0.040*
C13	0.2954 (3)	0.7707 (5)	0.75929 (15)	0.0367 (10)

H13	0.2395	0.8364	0.7718	0.044*
C14	0.3813 (3)	0.6748 (5)	0.79631 (16)	0.0311 (10)
C15	0.4646 (3)	0.5804 (5)	0.77481 (16)	0.0361 (11)
H15	0.5240	0.5172	0.7975	0.043*
C16	0.4592 (3)	0.5804 (5)	0.72124 (16)	0.0347 (10)
H16	0.5149	0.5154	0.7084	0.042*
C17	0.4703 (4)	0.5718 (7)	0.88665 (16)	0.0621 (15)
H17A	0.4611	0.4482	0.8779	0.093*
H17B	0.4593	0.5905	0.9221	0.093*
H17C	0.5481	0.6094	0.8847	0.093*
C18	0.3019 (4)	0.7759 (7)	0.87143 (18)	0.0772 (18)
H18A	0.3198	0.8995	0.8695	0.116*
H18B	0.3087	0.7427	0.9080	0.116*
H18C	0.2230	0.7539	0.8517	0.116*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0264 (3)	0.0323 (3)	0.0302 (3)	0.0008 (2)	0.01120 (19)	0.0021 (3)
Cl1	0.0274 (5)	0.0439 (6)	0.0374 (6)	−0.0022 (4)	0.0115 (4)	0.0000 (5)
Cl2	0.0311 (5)	0.0508 (7)	0.0459 (6)	−0.0013 (5)	0.0125 (4)	−0.0062 (6)
N1	0.0366 (18)	0.042 (2)	0.027 (2)	−0.0032 (16)	0.0047 (15)	0.0023 (18)
N2	0.053 (2)	0.047 (2)	0.024 (2)	0.0080 (18)	0.0071 (16)	−0.0006 (19)
O1	0.0411 (15)	0.0500 (19)	0.0353 (17)	−0.0068 (14)	0.0161 (13)	0.0011 (16)
O2	0.0388 (15)	0.0413 (18)	0.0298 (15)	0.0024 (13)	0.0142 (12)	0.0023 (15)
O3	0.0348 (15)	0.0335 (16)	0.059 (2)	0.0025 (12)	0.0169 (14)	0.0110 (15)
O4	0.0301 (13)	0.0331 (16)	0.0445 (18)	−0.0011 (12)	0.0138 (12)	−0.0081 (14)
O5	0.0260 (13)	0.0481 (18)	0.0500 (18)	0.0022 (13)	0.0143 (12)	0.0012 (16)
C1	0.037 (2)	0.036 (3)	0.038 (3)	0.0002 (19)	0.0095 (18)	−0.005 (2)
C2	0.033 (2)	0.035 (2)	0.029 (2)	−0.0019 (18)	0.0107 (17)	0.001 (2)
C3	0.034 (2)	0.044 (3)	0.030 (3)	−0.0075 (19)	0.0048 (18)	−0.006 (2)
C4	0.030 (2)	0.041 (3)	0.035 (3)	−0.0075 (18)	0.0122 (18)	−0.001 (2)
C5	0.029 (2)	0.030 (2)	0.034 (3)	0.0000 (17)	0.0077 (17)	0.002 (2)
C6	0.032 (2)	0.044 (3)	0.035 (3)	−0.0097 (19)	0.0033 (17)	0.000 (2)
C7	0.032 (2)	0.039 (3)	0.041 (3)	−0.0062 (18)	0.0112 (17)	0.004 (2)
C8	0.052 (3)	0.063 (3)	0.034 (3)	−0.010 (2)	0.018 (2)	−0.001 (3)
C9	0.051 (3)	0.074 (4)	0.032 (3)	−0.010 (2)	0.003 (2)	−0.005 (3)
C10	0.035 (2)	0.037 (3)	0.036 (3)	−0.0008 (19)	0.0148 (19)	0.002 (2)
C11	0.029 (2)	0.032 (2)	0.028 (2)	−0.0009 (17)	0.0066 (17)	0.003 (2)
C12	0.0303 (19)	0.039 (3)	0.029 (2)	0.0022 (18)	0.0040 (16)	0.005 (2)
C13	0.032 (2)	0.042 (3)	0.037 (3)	0.0103 (19)	0.0115 (17)	0.005 (2)
C14	0.034 (2)	0.027 (2)	0.031 (3)	−0.0046 (18)	0.0052 (18)	0.000 (2)
C15	0.033 (2)	0.038 (3)	0.034 (3)	0.0075 (19)	0.0024 (18)	−0.001 (2)
C16	0.031 (2)	0.035 (2)	0.040 (3)	0.0023 (18)	0.0136 (18)	0.000 (2)
C17	0.077 (3)	0.072 (4)	0.032 (3)	0.018 (3)	0.000 (2)	0.006 (3)
C18	0.089 (4)	0.111 (5)	0.036 (3)	0.032 (4)	0.023 (3)	0.000 (3)

Geometric parameters (Å, °)

Ni1—O3	2.038 (3)	C5—C6	1.420 (5)
Ni1—O4	2.042 (3)	C6—C7	1.349 (5)
Ni1—O5	2.049 (2)	C6—H6	0.9300
Ni1—O2	2.075 (3)	C7—H7	0.9300
Ni1—O1	2.094 (3)	C8—H8A	0.9600
Ni1—Cl1	2.3712 (11)	C8—H8B	0.9600
N1—C5	1.357 (5)	C8—H8C	0.9600
N1—C8	1.447 (5)	C9—H9A	0.9600
N1—C9	1.448 (4)	C9—H9B	0.9600
N2—C14	1.351 (5)	C9—H9C	0.9600
N2—C18	1.439 (5)	C10—C11	1.425 (5)
N2—C17	1.451 (5)	C10—H10	0.9300
O1—C1	1.232 (4)	C11—C16	1.389 (5)
O2—C10	1.240 (4)	C11—C12	1.399 (5)
O3—H19	0.8500	C12—C13	1.357 (5)
O3—H20	0.8500	C12—H12	0.9300
O4—H21	0.8500	C13—C14	1.416 (5)
O4—H22	0.8501	C13—H13	0.9300
O5—H23	0.8500	C14—C15	1.411 (5)
O5—H24	0.8499	C15—C16	1.364 (5)
C1—C2	1.436 (5)	C15—H15	0.9300
C1—H1	0.9300	C16—H16	0.9300
C2—C3	1.390 (5)	C17—H17A	0.9600
C2—C7	1.398 (5)	C17—H17B	0.9600
C3—C4	1.365 (5)	C17—H17C	0.9600
C3—H3A	0.9300	C18—H18A	0.9600
C4—C5	1.406 (5)	C18—H18B	0.9600
C4—H4A	0.9300	C18—H18C	0.9600
O3—Ni1—O4	177.40 (10)	C5—C6—H6	119.4
O3—Ni1—O5	92.24 (10)	C6—C7—C2	122.7 (4)
O4—Ni1—O5	87.84 (10)	C6—C7—H7	118.7
O3—Ni1—O2	88.37 (11)	C2—C7—H7	118.7
O4—Ni1—O2	89.02 (10)	N1—C8—H8A	109.5
O5—Ni1—O2	90.15 (10)	N1—C8—H8B	109.5
O3—Ni1—O1	91.12 (11)	H8A—C8—H8B	109.5
O4—Ni1—O1	91.48 (10)	N1—C8—H8C	109.5
O5—Ni1—O1	84.59 (10)	H8A—C8—H8C	109.5
O2—Ni1—O1	174.70 (10)	H8B—C8—H8C	109.5
O3—Ni1—Cl1	89.57 (7)	N1—C9—H9A	109.5
O4—Ni1—Cl1	90.39 (7)	N1—C9—H9B	109.5
O5—Ni1—Cl1	177.98 (8)	H9A—C9—H9B	109.5
O2—Ni1—Cl1	90.79 (8)	N1—C9—H9C	109.5
O1—Ni1—Cl1	94.48 (8)	H9A—C9—H9C	109.5
C5—N1—C8	119.9 (3)	H9B—C9—H9C	109.5
C5—N1—C9	122.8 (3)	O2—C10—C11	126.4 (4)
C8—N1—C9	117.2 (3)	O2—C10—H10	116.8

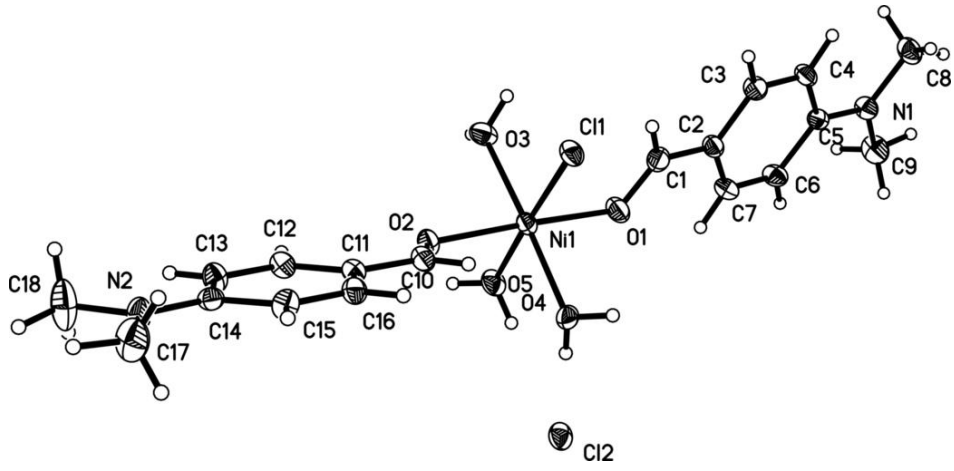
C14—N2—C18	122.0 (3)	C11—C10—H10	116.8
C14—N2—C17	121.8 (4)	C16—C11—C12	116.8 (3)
C18—N2—C17	116.2 (4)	C16—C11—C10	120.0 (4)
C1—O1—Ni1	124.9 (3)	C12—C11—C10	123.2 (3)
C10—O2—Ni1	122.1 (2)	C13—C12—C11	121.5 (3)
Ni1—O3—H19	115.5	C13—C12—H12	119.3
Ni1—O3—H20	120.7	C11—C12—H12	119.3
H19—O3—H20	105.6	C12—C13—C14	122.1 (4)
Ni1—O4—H21	114.4	C12—C13—H13	119.0
Ni1—O4—H22	114.6	C14—C13—H13	119.0
H21—O4—H22	103.1	N2—C14—C15	121.9 (4)
Ni1—O5—H23	116.6	N2—C14—C13	122.1 (4)
Ni1—O5—H24	125.6	C15—C14—C13	116.0 (4)
H23—O5—H24	108.6	C16—C15—C14	120.9 (4)
O1—C1—C2	125.7 (4)	C16—C15—H15	119.5
O1—C1—H1	117.2	C14—C15—H15	119.5
C2—C1—H1	117.2	C15—C16—C11	122.7 (4)
C3—C2—C7	116.6 (4)	C15—C16—H16	118.7
C3—C2—C1	119.8 (4)	C11—C16—H16	118.7
C7—C2—C1	123.5 (3)	N2—C17—H17A	109.5
C4—C3—C2	121.7 (4)	N2—C17—H17B	109.5
C4—C3—H3A	119.2	H17A—C17—H17B	109.5
C2—C3—H3A	119.2	N2—C17—H17C	109.5
C3—C4—C5	122.0 (4)	H17A—C17—H17C	109.5
C3—C4—H4A	119.0	H17B—C17—H17C	109.5
C5—C4—H4A	119.0	N2—C18—H18A	109.5
N1—C5—C4	122.9 (3)	N2—C18—H18B	109.5
N1—C5—C6	121.2 (3)	H18A—C18—H18B	109.5
C4—C5—C6	115.9 (4)	N2—C18—H18C	109.5
C7—C6—C5	121.1 (3)	H18A—C18—H18C	109.5
C7—C6—H6	119.4	H18B—C18—H18C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H19···Cl1^{i}	0.85	2.37	3.214 (3)	171
O3—H20···Cl2^{ii}	0.85	2.32	3.110 (3)	155
O4—H21···Cl1^{iii}	0.85	2.27	3.095 (3)	163
O5—H24···Cl2^{iv}	0.85	2.33	3.088 (3)	149

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

Fig. 1


C3
C4
C8
N1
C5
C9
C2
C1
C6
C7
O3
Cl1
O2
Ni1
O1
C13
C12
C11
C10
O5
O4
C18
N2
C14
C15
C16
C17
Cl2