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μ-Acetato-κ^2O:O'-[7,23-dibenzyl-15,31-dichloro-3,7,11,19,23,27-hexaazatricyclo[27.3.1.1^{13,17}]tetratriconta-1(32),2,11,13,15,17(34),18,27,29(33),30-decaene-33,34-diolato-$\kappa^{10}N^4,N^5,N^6,O^1,O^2$:$N^1,N^2,N^3,O^1,O^2$]dinickel(II) perchlorate acetonitrile disolvate

Juan Kong, Hong Zhou and Zhi-Quan Pan*

Hubei Key Laboratory of Novel Chemical Reactor and Green Chemical Technology, Wuhan Institute of Technology, Wuhan 430073, People's Republic of China
Correspondence e-mail: zhiqpan@163.com

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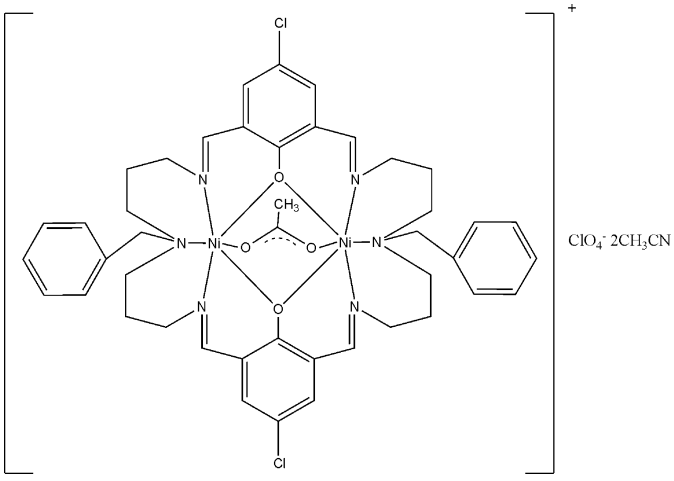
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.007 Å; R factor = 0.060; wR factor = 0.158; data-to-parameter ratio = 16.2.

The title complex, $[Ni_2(C_{42}H_{46}Cl_2N_6O_2)(C_2H_3O_2)]ClO_4\cdot 2CH_3CN$, was synthesized by condensation of 2,6-diformyl-4-chlorophenol with *N*,*N*-bis(aminopropyl)benzylamine in the presence of Ni^{II} ions. The ligand is a 28-membered macrocycle with two identical pendant arms. The coordination geometries of the Ni atoms are both octahedral. The two Ni atoms are bridged by two phenolate O atoms of the macrocyclic ligand and one acetate ligand, with an Ni···Ni distance of 3.147 (4) Å.

Related literature

For related literature, see: Gou & Fenton (1994); Luo *et al.* (2002); Turonek *et al.* (1995); Zeng *et al.* (1998).



Experimental

Crystal data

$[Ni_2(C_{42}H_{46}Cl_2N_6O_2)(C_2H_3O_2)]ClO_4\cdot 2C_2H_3N$
M_r = 1095.77
Monoclinic, $P2_1/n$
a = 16.7957 (14) Å
b = 17.2146 (15) Å
c = 18.0209 (15) Å
β = 99.305 (2)°
V = 5141.8 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.95 mm^{-1}
T = 273 (2) K
0.32 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.7, T_{max} = 0.8
29289 measured reflections
10096 independent reflections
7248 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.158
S = 1.08
10096 reflections
623 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.81 e Å^{-3}
$\Delta\rho_{min}$ = −0.98 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: HG2351).

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Zeng, Q., Sun, J., Gou, S., Zhou, K., Fang, J. & Chen, H. (1998). *Transition Met. Chem.* **23**, 371–373.

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supporting information

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μ-Acetato-κ^2O:O'-[7,23-dibenzyl-15,31-dichloro-3,7,11,19,23,27-hexaazatricyclo[27.3.1.1^{13,17}]tetratriconta-1(32),2,11,13,15,17(34),18,27,29(33),30-decaene-33,34-diolato-$\kappa^{10}N^4,N^5,N^6,O^1,O^2$:$N^1,N^2,N^3,O^1,O^2$]dinickel(II) perchlorate acetonitrile disolvate

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S1. Comment

Pendant-arm marocyclic complexes have attracted much interest in recent decades. Because there is a concept that, by having pendant arm attached at appropriate positions on the macrocyclic framework, an "opened cryptand" would result, leading to modified complexation property over the corresponding clathrochelates or simple macrocyclic precursors (Zeng *et al.*, 1998). Transition metal complexes with pendant-arm ligands, usually synthesized by cyclocondensation of 2,6-diformyl-4- chlorophenol and diamine the stepwise template reaction, have been studies extensively (Luo *et al.*, 2002, Zeng *et al.*, 1998). However, the dinuclear nickel complex of this ligand(I) has not been published. Here, we report the synthesis and crystal structure of the complex.

The crystal structure is composed of complex cations, perchlorate anions and solvent acetonitrile molecules. Neither the perchlorate ion nor the acetonitrile molecules participate in coordination of the Ni atoms. The complex cation is extremely twisted owing to the flexility of macrocyclic ligand as well as the requirement of Ni coordination with donor atoms. The structure of (I) is shown in Fig. 1. The coordination geometry of Ni1 is similar to that of Ni_2. The coordination polyhedron around Ni_1 is a distorted octahedron, whose equatorial plane is formed by one imine N_6, one tertiary N_5, two phenolate O_1 and O_2 with the mean deviation of 0.0541 (4) Å. The axial positions are occupied by N_4 and O_3, respectively. the band length of Ni_1—O is fall in the range 2.033 (3)–2.082 (3) Å, but the two Ni_1—*N*(imine) bonds[2.081 (4) and 2.092 (3) Å, respectively] are shorter significantly than the Ni_1—N(tertiary) [2.201 (3) Å]. The Ni···Ni separation is 3.147 (4) Å. The two benzyl groups attached to N_2 and N_5 respectively are *cis* to each other.

S2. Experimental

2,6-diformyl-4-chlorophenol was prepared by a modification of the literature method (Gou & Fenton, 1994). *N*,*N*-bis-(aminopropyl)- benzylamine prepared by literature method of (Turonek *et al.*, 1995). The title complex was synthesized by the following procedure: 0.5 mmol *N*,*N*-bis(aminopropyl)-benzylamine in 15 ml of absolute methanol was added dropwise to a methanol solution (30 ml) containing 0.5 mmol 2,6-diformyl-4-methylphenol and 0.5 mmol $Ni(OAc)_2.H_2O$. After stirring the mixture for 10 h at room temperature, a green solution formed. A methanol solution (10 ml) containing $Ni(ClO_4)_2.4H_2O$(0.5 mmol) was added dropwise. A yellow-green solution was produced after stirring at room temperature for 4 h. Green needle-shaped crystals suitable for X-ray diffraction were obtained by slow evaporation from acetonitrile over three days.

S3. Refinement

All H atoms for C—H distances were placed in calculated positions in the range 0.93–0.97 Å, and included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2–1.5 U_{eq}(C).

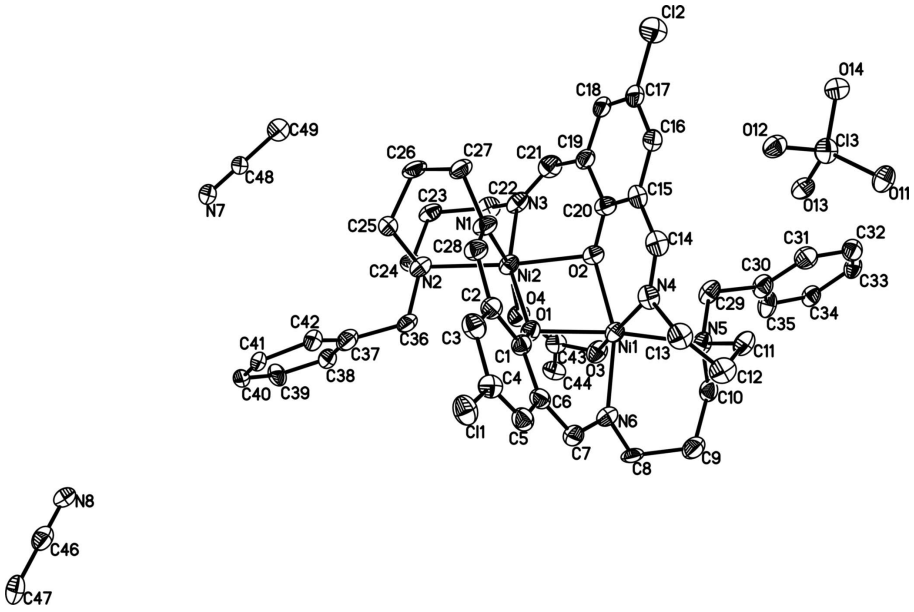

Figure 1

A view of the title complex, showing the labeling of the non-H atoms and 30% probability ellipsoids. H atoms have been omitted.

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Crystal data

$[Ni_2(C_{42}H_{46}Cl_2N_6O_2)(C_2H_3O_2)]ClO_4\cdot 2C_2H_3N$
M_r = 1095.77
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 16.7957 (14) Å
b = 17.2146 (15) Å
c = 18.0209 (15) Å
β = 99.305 (2)°
V = 5141.8 (8) Å^3
Z = 4
$F(000)$ = 2280
D_x = 1.415 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5427 reflections
θ = 2.2–26.0°
μ = 0.95 mm^{-1}
T = 273 K
Needle, green
0.32 × 0.26 × 0.24 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.7, T_{max} = 0.8
29289 measured reflections
10096 independent reflections
7248 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 26.0°, θ_{min} = 2.2°
h = −16→20
k = −19→21
l = −22→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.158$
$S = 1.08$
10096 reflections
623 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.080P)^2 + 1.990P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.81$ e Å^{-3}
$\Delta\rho_{min} = -0.98$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5447 (2)	0.4856 (2)	0.3737 (2)	0.0384 (8)
C2	0.6266 (3)	0.5068 (2)	0.3850 (2)	0.0433 (9)
C3	0.6790 (3)	0.4856 (3)	0.4510 (2)	0.0504 (11)
H3	0.7328	0.5010	0.4588	0.060*
C4	0.6471 (3)	0.4392 (2)	0.5066 (2)	0.0437 (9)
C5	0.5708 (3)	0.4157 (2)	0.4929 (2)	0.0466 (10)
H5	0.5513	0.3851	0.5285	0.056*
C6	0.5182 (3)	0.4354 (2)	0.4263 (2)	0.0414 (9)
C7	0.4348 (3)	0.4073 (2)	0.4149 (2)	0.0441 (10)
H7	0.4104	0.3986	0.4570	0.053*
C8	0.3076 (2)	0.3736 (3)	0.3447 (2)	0.0441 (10)
H8A	0.2761	0.4198	0.3293	0.053*
H8B	0.2976	0.3597	0.3945	0.053*
C9	0.2760 (3)	0.3071 (3)	0.2904 (3)	0.0587 (12)
H9A	0.2184	0.3020	0.2897	0.070*
H9B	0.3011	0.2590	0.3100	0.070*
C10	0.2912 (3)	0.3175 (3)	0.2086 (2)	0.0449 (10)
H10A	0.2648	0.2748	0.1792	0.054*
H10B	0.2643	0.3648	0.1891	0.054*
C11	0.4153 (3)	0.2448 (2)	0.2070 (3)	0.0480 (10)
H11A	0.4513	0.2384	0.1704	0.058*
H11B	0.3749	0.2041	0.1982	0.058*
C12	0.4646 (3)	0.2343 (3)	0.2870 (3)	0.0528 (11)
H12A	0.4307	0.2494	0.3233	0.063*
H12B	0.4769	0.1796	0.2946	0.063*

C13	0.5404 (3)	0.2781 (3)	0.3032 (3)	0.0536 (11)
H13A	0.5496	0.2934	0.3556	0.064*
H13B	0.5847	0.2449	0.2947	0.064*
C14	0.5897 (3)	0.3469 (3)	0.2101 (3)	0.0495 (10)
H14	0.6267	0.3062	0.2151	0.059*
C15	0.5962 (3)	0.4036 (2)	0.1500 (2)	0.0457 (10)
C16	0.6554 (3)	0.3915 (3)	0.1043 (2)	0.0472 (10)
H16	0.6873	0.3472	0.1118	0.057*
C17	0.6677 (3)	0.4424 (3)	0.0493 (2)	0.0490 (11)
C18	0.6236 (3)	0.5072 (3)	0.0399 (2)	0.0471 (10)
H18	0.6330	0.5419	0.0028	0.056*
C19	0.5643 (3)	0.5255 (2)	0.0825 (2)	0.0440 (10)
C20	0.5473 (2)	0.4692 (3)	0.1378 (2)	0.0427 (9)
C21	0.5214 (3)	0.5981 (3)	0.0724 (3)	0.0547 (12)
H21	0.5127	0.6200	0.0246	0.066*
C22	0.4451 (3)	0.7055 (3)	0.1046 (3)	0.0513 (11)
H22A	0.3897	0.6932	0.1094	0.062*
H22B	0.4457	0.7186	0.0524	0.062*
C23	0.4703 (3)	0.7769 (3)	0.1520 (2)	0.0480 (10)
H23A	0.4356	0.8200	0.1334	0.058*
H23B	0.5251	0.7907	0.1467	0.058*
C24	0.4660 (3)	0.7642 (2)	0.2374 (2)	0.0438 (9)
H24A	0.4738	0.8141	0.2623	0.053*
H24B	0.4119	0.7468	0.2413	0.053*
C25	0.6092 (3)	0.7352 (3)	0.2870 (2)	0.0491 (11)
H25A	0.6374	0.7196	0.3360	0.059*
H25B	0.6077	0.7915	0.2866	0.059*
C26	0.6611 (3)	0.7089 (3)	0.2275 (2)	0.0527 (12)
H26A	0.6321	0.7218	0.1780	0.063*
H26B	0.7107	0.7388	0.2350	0.063*
C27	0.6823 (3)	0.6259 (3)	0.2279 (3)	0.0508 (11)
H27A	0.7379	0.6194	0.2519	0.061*
H27B	0.6779	0.6077	0.1765	0.061*
C28	0.6636 (3)	0.5506 (3)	0.3313 (2)	0.0467 (10)
H28	0.7187	0.5596	0.3441	0.056*
C29	0.3746 (3)	0.3459 (3)	0.1126 (2)	0.0484 (10)
H29A	0.3524	0.3979	0.1061	0.058*
H29B	0.4303	0.3487	0.1046	0.058*
C30	0.3289 (3)	0.2947 (3)	0.0519 (2)	0.0500 (11)
C31	0.3671 (3)	0.2357 (3)	0.0197 (3)	0.0542 (11)
H31	0.4209	0.2244	0.0374	0.065*
C32	0.3247 (3)	0.1931 (3)	−0.0397 (3)	0.0491 (10)
H32	0.3497	0.1515	−0.0594	0.059*
C33	0.2477 (3)	0.2114 (2)	−0.0690 (3)	0.0462 (10)
H33	0.2216	0.1849	−0.1110	0.055*
C34	0.2073 (3)	0.2696 (2)	−0.0366 (2)	0.0432 (9)
H34	0.1534	0.2805	−0.0546	0.052*
C35	0.2489 (3)	0.3106 (3)	0.0226 (3)	0.0552 (12)

H35	0.2227	0.3504	0.0439	0.066*
C36	0.5011 (3)	0.6952 (3)	0.3556 (2)	0.0460 (10)
H36A	0.5317	0.6517	0.3795	0.055*
H36B	0.4446	0.6803	0.3479	0.055*
C37	0.5120 (3)	0.7639 (3)	0.4106 (3)	0.0527 (11)
C38	0.4489 (3)	0.8212 (3)	0.4079 (3)	0.0478 (10)
H38	0.4029	0.8176	0.3716	0.057*
C39	0.4566 (3)	0.8796 (3)	0.4576 (2)	0.0455 (10)
H39	0.4172	0.9180	0.4536	0.055*
C40	0.5231 (2)	0.8839 (2)	0.5158 (2)	0.0428 (10)
H40	0.5266	0.9232	0.5516	0.051*
C41	0.5814 (3)	0.8304 (2)	0.5188 (2)	0.0431 (10)
H41	0.6255	0.8334	0.5573	0.052*
C42	0.5786 (3)	0.7691 (3)	0.4654 (2)	0.0463 (10)
H42	0.6207	0.7337	0.4677	0.056*
C43	0.3289 (2)	0.5568 (2)	0.2186 (2)	0.0410 (9)
C44	0.2420 (3)	0.5854 (3)	0.2063 (2)	0.0459 (10)
H44A	0.2380	0.6304	0.2370	0.069*
H44B	0.2075	0.5452	0.2200	0.069*
H44C	0.2258	0.5986	0.1544	0.069*
C46	0.5706 (3)	1.0022 (3)	0.8446 (3)	0.0529 (11)
C48	0.6614 (3)	0.9861 (3)	0.2799 (2)	0.0444 (10)
C111	0.5752 (3)	0.9916 (3)	0.9238 (3)	0.0587 (12)
H7A	0.5754	0.9404	0.9343	0.088*
H7B	0.6210	1.0134	0.9477	0.088*
H7C	0.5325	1.0142	0.9393	0.088*
C112	0.6952 (3)	0.9316 (3)	0.2343 (2)	0.0481 (10)
H8C	0.6900	0.8833	0.2521	0.072*
H8D	0.6693	0.9345	0.1865	0.072*
H8E	0.7478	0.9424	0.2356	0.072*
Cl1	0.70869 (8)	0.41975 (7)	0.58362 (7)	0.0568 (3)
Cl2	0.73998 (8)	0.42420 (7)	−0.00057 (7)	0.0620 (3)
Cl3	0.64598 (7)	0.15587 (6)	0.06422 (6)	0.0473 (3)
N1	0.6295 (2)	0.5785 (2)	0.2680 (2)	0.0492 (9)
N2	0.5251 (2)	0.7073 (2)	0.2805 (2)	0.0444 (8)
N3	0.4957 (2)	0.6330 (2)	0.1232 (2)	0.0467 (9)
N4	0.5390 (2)	0.3470 (2)	0.25636 (19)	0.0436 (8)
N5	0.3744 (2)	0.3212 (2)	0.1943 (2)	0.0457 (8)
N6	0.3943 (2)	0.3941 (2)	0.3504 (2)	0.0432 (8)
N7	0.6331 (2)	1.0322 (2)	0.3179 (2)	0.0484 (9)
N8	0.5666 (2)	1.0108 (2)	0.7794 (2)	0.0506 (9)
Ni1	0.43937 (3)	0.42058 (3)	0.25238 (3)	0.03695 (14)
Ni2	0.50518 (3)	0.58905 (3)	0.23164 (3)	0.03986 (15)
O1	0.49362 (17)	0.51229 (16)	0.31702 (15)	0.0408 (6)
O2	0.48682 (16)	0.48320 (16)	0.17442 (15)	0.0418 (6)
O3	0.33703 (18)	0.48490 (17)	0.22122 (18)	0.0493 (7)
O4	0.38232 (19)	0.60574 (17)	0.22422 (17)	0.0496 (7)
O11	0.64001 (18)	0.06817 (17)	0.08605 (17)	0.0492 (7)

O12	0.70489 (18)	0.19800 (17)	0.11967 (17)	0.0481 (7)
O13	0.56391 (17)	0.19927 (16)	0.06237 (16)	0.0445 (7)
O14	0.68326 (18)	0.16370 (17)	−0.00305 (16)	0.0476 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.043 (2)	0.0327 (19)	0.039 (2)	0.0000 (16)	0.0061 (17)	0.0031 (16)
C2	0.050 (2)	0.037 (2)	0.043 (2)	0.0014 (18)	0.0087 (19)	0.0033 (17)
C3	0.055 (3)	0.053 (3)	0.040 (2)	0.005 (2)	0.000 (2)	−0.0005 (19)
C4	0.043 (2)	0.040 (2)	0.049 (2)	0.0114 (17)	0.0092 (18)	0.0056 (18)
C5	0.061 (3)	0.041 (2)	0.038 (2)	0.0020 (19)	0.0070 (19)	−0.0003 (17)
C6	0.054 (3)	0.037 (2)	0.034 (2)	0.0032 (18)	0.0107 (18)	0.0008 (16)
C7	0.051 (3)	0.035 (2)	0.048 (2)	−0.0010 (18)	0.013 (2)	0.0031 (17)
C8	0.039 (2)	0.058 (3)	0.040 (2)	−0.0178 (19)	0.0186 (18)	0.0039 (18)
C9	0.059 (3)	0.057 (3)	0.061 (3)	−0.020 (2)	0.012 (2)	0.002 (2)
C10	0.052 (3)	0.042 (2)	0.038 (2)	−0.0114 (19)	−0.0018 (18)	−0.0038 (17)
C11	0.039 (2)	0.041 (2)	0.060 (3)	−0.0062 (18)	−0.002 (2)	0.007 (2)
C12	0.048 (3)	0.055 (3)	0.052 (3)	0.011 (2)	−0.004 (2)	0.016 (2)
C13	0.055 (3)	0.050 (3)	0.053 (3)	0.010 (2)	0.002 (2)	0.007 (2)
C14	0.045 (2)	0.050 (2)	0.051 (2)	0.0066 (19)	0.000 (2)	−0.003 (2)
C15	0.051 (3)	0.042 (2)	0.043 (2)	−0.0012 (18)	0.0052 (19)	−0.0095 (17)
C16	0.047 (2)	0.042 (2)	0.055 (3)	−0.0082 (18)	0.014 (2)	−0.0110 (19)
C17	0.055 (3)	0.047 (2)	0.044 (2)	−0.018 (2)	0.008 (2)	−0.0114 (19)
C18	0.053 (3)	0.044 (2)	0.044 (2)	−0.021 (2)	0.010 (2)	−0.0078 (18)
C19	0.050 (3)	0.046 (2)	0.035 (2)	−0.0175 (19)	0.0041 (18)	−0.0001 (17)
C20	0.038 (2)	0.051 (2)	0.039 (2)	−0.0065 (18)	0.0061 (17)	0.0036 (18)
C21	0.060 (3)	0.059 (3)	0.043 (2)	−0.005 (2)	0.002 (2)	0.011 (2)
C22	0.043 (2)	0.058 (3)	0.050 (2)	0.004 (2)	−0.001 (2)	0.012 (2)
C23	0.046 (2)	0.051 (2)	0.047 (2)	−0.0090 (19)	0.0079 (19)	0.0175 (19)
C24	0.048 (2)	0.043 (2)	0.042 (2)	0.0093 (18)	0.0145 (19)	0.0119 (18)
C25	0.060 (3)	0.047 (2)	0.043 (2)	−0.014 (2)	0.017 (2)	−0.0071 (19)
C26	0.058 (3)	0.059 (3)	0.044 (2)	−0.021 (2)	0.018 (2)	0.015 (2)
C27	0.054 (3)	0.052 (3)	0.049 (2)	−0.020 (2)	0.015 (2)	0.006 (2)
C28	0.039 (2)	0.055 (3)	0.045 (2)	−0.0066 (19)	0.0055 (18)	0.004 (2)
C29	0.045 (2)	0.049 (2)	0.044 (2)	−0.0147 (19)	−0.0133 (19)	0.0090 (19)
C30	0.053 (3)	0.051 (3)	0.042 (2)	−0.014 (2)	−0.007 (2)	−0.0004 (19)
C31	0.044 (3)	0.058 (3)	0.059 (3)	−0.004 (2)	0.007 (2)	0.000 (2)
C32	0.041 (2)	0.043 (2)	0.064 (3)	0.0026 (18)	0.011 (2)	−0.003 (2)
C33	0.040 (2)	0.044 (2)	0.052 (2)	−0.0163 (18)	−0.0002 (19)	−0.0088 (19)
C34	0.038 (2)	0.046 (2)	0.042 (2)	−0.0115 (17)	−0.0052 (17)	−0.0014 (18)
C35	0.049 (3)	0.047 (3)	0.063 (3)	−0.011 (2)	−0.013 (2)	−0.007 (2)
C36	0.040 (2)	0.050 (2)	0.049 (2)	−0.0181 (19)	0.0121 (19)	0.0007 (19)
C37	0.044 (3)	0.048 (2)	0.067 (3)	−0.011 (2)	0.014 (2)	0.001 (2)
C38	0.042 (2)	0.051 (2)	0.053 (2)	−0.0150 (19)	0.014 (2)	−0.013 (2)
C39	0.047 (2)	0.042 (2)	0.049 (2)	0.0074 (18)	0.0093 (19)	−0.0139 (18)
C40	0.042 (2)	0.049 (2)	0.041 (2)	−0.0155 (19)	0.0162 (18)	−0.0165 (18)
C41	0.044 (2)	0.044 (2)	0.042 (2)	−0.0215 (18)	0.0106 (18)	−0.0119 (17)

C42	0.050 (3)	0.043 (2)	0.046 (2)	−0.0134 (19)	0.0082 (19)	−0.0047 (18)
C43	0.040 (2)	0.039 (2)	0.044 (2)	0.0084 (17)	0.0056 (17)	0.0072 (17)
C44	0.043 (2)	0.056 (3)	0.038 (2)	0.0119 (19)	0.0066 (18)	0.0082 (18)
C46	0.040 (2)	0.047 (3)	0.072 (3)	−0.0084 (19)	0.011 (2)	−0.005 (2)
C48	0.047 (2)	0.047 (2)	0.043 (2)	−0.0166 (18)	0.0157 (19)	−0.0142 (18)
C111	0.053 (3)	0.048 (3)	0.069 (3)	−0.014 (2)	−0.008 (2)	−0.009 (2)
C112	0.049 (2)	0.045 (2)	0.049 (2)	0.0059 (19)	0.004 (2)	0.0067 (19)
Cl1	0.0675 (8)	0.0461 (6)	0.0541 (6)	0.0129 (5)	0.0015 (5)	−0.0038 (5)
Cl2	0.0666 (8)	0.0560 (7)	0.0645 (7)	0.0165 (6)	0.0137 (6)	0.0127 (6)
Cl3	0.0482 (6)	0.0417 (5)	0.0497 (6)	0.0084 (4)	0.0013 (4)	0.0062 (4)
N1	0.0360 (19)	0.061 (2)	0.051 (2)	−0.0126 (16)	0.0080 (16)	0.0057 (17)
N2	0.0396 (19)	0.0402 (18)	0.054 (2)	−0.0043 (15)	0.0079 (16)	0.0092 (15)
N3	0.0375 (19)	0.049 (2)	0.051 (2)	−0.0089 (15)	−0.0018 (16)	0.0133 (17)
N4	0.048 (2)	0.0424 (19)	0.0374 (18)	0.0069 (15)	−0.0029 (16)	0.0008 (14)
N5	0.045 (2)	0.0403 (19)	0.048 (2)	−0.0078 (15)	−0.0030 (16)	0.0002 (15)
N6	0.045 (2)	0.0385 (18)	0.047 (2)	−0.0050 (15)	0.0108 (16)	0.0077 (15)
N7	0.047 (2)	0.050 (2)	0.049 (2)	−0.0150 (16)	0.0102 (17)	−0.0119 (17)
N8	0.045 (2)	0.053 (2)	0.057 (2)	−0.0151 (17)	0.0171 (18)	0.0003 (18)
Ni1	0.0350 (3)	0.0339 (3)	0.0409 (3)	−0.00144 (19)	0.0027 (2)	0.0042 (2)
Ni2	0.0403 (3)	0.0377 (3)	0.0403 (3)	−0.0052 (2)	0.0027 (2)	0.0072 (2)
O1	0.0424 (15)	0.0386 (14)	0.0391 (15)	−0.0069 (12)	−0.0002 (12)	0.0029 (11)
O2	0.0382 (15)	0.0452 (16)	0.0422 (15)	−0.0033 (12)	0.0071 (12)	0.0042 (12)
O3	0.0396 (16)	0.0440 (17)	0.0633 (19)	−0.0017 (13)	0.0055 (14)	0.0025 (14)
O4	0.0487 (18)	0.0436 (17)	0.0556 (18)	0.0033 (14)	0.0056 (14)	0.0090 (14)
O11	0.0451 (17)	0.0441 (16)	0.0544 (18)	−0.0075 (13)	−0.0042 (14)	0.0017 (13)
O12	0.0498 (18)	0.0424 (16)	0.0535 (18)	−0.0031 (13)	0.0120 (14)	0.0082 (13)
O13	0.0435 (16)	0.0440 (16)	0.0451 (15)	−0.0043 (12)	0.0047 (13)	0.0107 (13)
O14	0.0449 (17)	0.0519 (17)	0.0470 (16)	−0.0004 (13)	0.0102 (13)	0.0050 (13)

Geometric parameters (Å, º)

C1—O1	1.306 (5)	C27—N1	1.478 (5)
C1—C2	1.405 (6)	C27—H27A	0.9700
C1—C6	1.407 (5)	C27—H27B	0.9700
C2—C3	1.408 (6)	C28—N1	1.285 (6)
C2—C28	1.445 (6)	C28—H28	0.9300
C3—C4	1.450 (6)	C29—C30	1.514 (6)
C3—H3	0.9300	C29—N5	1.533 (5)
C4—C5	1.328 (6)	C29—H29A	0.9700
C4—Cl1	1.626 (4)	C29—H29B	0.9700
C5—C6	1.413 (6)	C30—C31	1.378 (7)
C5—H5	0.9300	C30—C35	1.389 (6)
C6—C7	1.465 (6)	C31—C32	1.394 (7)
C7—N6	1.270 (6)	C31—H31	0.9300
C7—H7	0.9300	C32—C33	1.353 (6)
C8—N6	1.484 (5)	C32—H32	0.9300
C8—C9	1.543 (6)	C33—C34	1.390 (6)
C8—H8A	0.9700	C33—H33	0.9300

C8—H8B	0.9700	C34—C35	1.372 (6)
C9—C10	1.546 (6)	C34—H34	0.9300
C9—H9A	0.9700	C35—H35	0.9300
C9—H9B	0.9700	C36—N2	1.488 (5)
C10—N5	1.462 (6)	C36—C37	1.534 (6)
C10—H10A	0.9700	C36—H36A	0.9700
C10—H10B	0.9700	C36—H36B	0.9700
C11—N5	1.485 (5)	C37—C42	1.369 (6)
C11—C12	1.553 (6)	C37—C38	1.444 (7)
C11—H11A	0.9700	C38—C39	1.338 (6)
C11—H11B	0.9700	C38—H38	0.9300
C12—C13	1.468 (7)	C39—C40	1.406 (6)
C12—H12A	0.9700	C39—H39	0.9300
C12—H12B	0.9700	C40—C41	1.338 (6)
C13—N4	1.452 (6)	C40—H40	0.9300
C13—H13A	0.9700	C41—C42	1.423 (6)
C13—H13B	0.9700	C41—H41	0.9300
C14—N4	1.283 (6)	C42—H42	0.9300
C14—C15	1.476 (6)	C43—O4	1.224 (5)
C14—H14	0.9300	C43—O3	1.245 (5)
C15—C20	1.394 (6)	C43—C44	1.521 (6)
C15—C16	1.405 (6)	C44—H44A	0.9600
C16—C17	1.363 (6)	C44—H44B	0.9600
C16—H16	0.9300	C44—H44C	0.9600
C17—C18	1.335 (7)	C46—N8	1.175 (6)
C17—Cl2	1.652 (5)	C46—C111	1.430 (7)
C18—C19	1.388 (6)	C48—N7	1.195 (5)
C18—H18	0.9300	C48—C112	1.424 (6)
C19—C21	1.440 (7)	C111—H7A	0.8999
C19—C20	1.450 (6)	C111—H7B	0.8999
C20—O2	1.320 (5)	C111—H7C	0.9001
C21—N3	1.230 (6)	C112—H8C	0.9000
C21—H21	0.9300	C112—H8D	0.9000
C22—N3	1.517 (6)	C112—H8E	0.9000
C22—C23	1.518 (7)	Cl3—O14	1.458 (3)
C22—H22A	0.9700	Cl3—O12	1.478 (3)
C22—H22B	0.9700	Cl3—O13	1.564 (3)
C23—C24	1.566 (6)	Cl3—O11	1.567 (3)
C23—H23A	0.9700	N1—Ni2	2.092 (4)
C23—H23B	0.9700	N2—Ni2	2.222 (4)
C24—N2	1.515 (5)	N3—Ni2	2.078 (4)
C24—H24A	0.9700	N4—Ni1	2.091 (4)
C24—H24B	0.9700	N5—Ni1	2.200 (3)
C25—N2	1.479 (6)	N6—Ni1	2.082 (3)
C25—C26	1.556 (6)	Ni1—O2	2.033 (3)
C25—H25A	0.9700	Ni1—O3	2.046 (3)
C25—H25B	0.9700	Ni1—O1	2.082 (3)
C26—C27	1.471 (7)	Ni2—O1	2.061 (3)

C26—H26A	0.9700	Ni2—O4	2.066 (3)
C26—H26B	0.9700	Ni2—O2	2.092 (3)
O1—C1—C2	121.8 (3)	C30—C31—H31	120.1
O1—C1—C6	120.2 (4)	C32—C31—H31	120.1
C2—C1—C6	118.0 (4)	C33—C32—C31	121.0 (4)
C1—C2—C3	121.3 (4)	C33—C32—H32	119.5
C1—C2—C28	123.7 (4)	C31—C32—H32	119.5
C3—C2—C28	115.0 (4)	C32—C33—C34	120.2 (4)
C2—C3—C4	118.4 (4)	C32—C33—H33	119.9
C2—C3—H3	120.8	C34—C33—H33	119.9
C4—C3—H3	120.8	C35—C34—C33	118.4 (4)
C5—C4—C3	119.5 (4)	C35—C34—H34	120.8
C5—C4—Cl1	123.9 (4)	C33—C34—H34	120.8
C3—C4—Cl1	116.6 (3)	C34—C35—C30	122.3 (5)
C4—C5—C6	122.5 (4)	C34—C35—H35	118.9
C4—C5—H5	118.7	C30—C35—H35	118.9
C6—C5—H5	118.7	N2—C36—C37	117.3 (3)
C1—C6—C5	119.8 (4)	N2—C36—H36A	108.0
C1—C6—C7	120.6 (4)	C37—C36—H36A	108.0
C5—C6—C7	119.5 (4)	N2—C36—H36B	108.0
N6—C7—C6	123.1 (4)	C37—C36—H36B	108.0
N6—C7—H7	118.5	H36A—C36—H36B	107.2
C6—C7—H7	118.5	C42—C37—C38	119.2 (4)
N6—C8—C9	117.0 (4)	C42—C37—C36	121.0 (4)
N6—C8—H8A	108.1	C38—C37—C36	119.6 (4)
C9—C8—H8A	108.1	C39—C38—C37	120.0 (4)
N6—C8—H8B	108.1	C39—C38—H38	120.0
C9—C8—H8B	108.1	C37—C38—H38	120.0
H8A—C8—H8B	107.3	C38—C39—C40	121.2 (4)
C8—C9—C10	115.2 (4)	C38—C39—H39	119.4
C8—C9—H9A	108.5	C40—C39—H39	119.4
C10—C9—H9A	108.5	C41—C40—C39	118.8 (4)
C8—C9—H9B	108.5	C41—C40—H40	120.6
C10—C9—H9B	108.5	C39—C40—H40	120.6
H9A—C9—H9B	107.5	C40—C41—C42	122.6 (4)
N5—C10—C9	118.8 (4)	C40—C41—H41	118.7
N5—C10—H10A	107.6	C42—C41—H41	118.7
C9—C10—H10A	107.6	C37—C42—C41	118.1 (4)
N5—C10—H10B	107.6	C37—C42—H42	121.0
C9—C10—H10B	107.6	C41—C42—H42	121.0
H10A—C10—H10B	107.0	O4—C43—O3	127.4 (4)
N5—C11—C12	114.1 (4)	O4—C43—C44	117.5 (4)
N5—C11—H11A	108.7	O3—C43—C44	115.1 (4)
C12—C11—H11A	108.7	C43—C44—H44A	109.5
N5—C11—H11B	108.7	C43—C44—H44B	109.5
C12—C11—H11B	108.7	H44A—C44—H44B	109.5
H11A—C11—H11B	107.6	C43—C44—H44C	109.5

C13—C12—C11	116.1 (4)	H44A—C44—H44C	109.5
C13—C12—H12A	108.3	H44B—C44—H44C	109.5
C11—C12—H12A	108.3	N8—C46—C111	179.8 (6)
C13—C12—H12B	108.3	N7—C48—C112	179.6 (5)
C11—C12—H12B	108.3	C46—C111—H7A	109.5
H12A—C12—H12B	107.4	C46—C111—H7B	109.2
N4—C13—C12	111.8 (4)	H7A—C111—H7B	109.5
N4—C13—H13A	109.3	C46—C111—H7C	109.7
C12—C13—H13A	109.3	H7A—C111—H7C	109.5
N4—C13—H13B	109.3	H7B—C111—H7C	109.5
C12—C13—H13B	109.3	C48—C112—H8C	109.4
H13A—C13—H13B	107.9	C48—C112—H8D	109.8
N4—C14—C15	127.7 (4)	H8C—C112—H8D	109.5
N4—C14—H14	116.2	C48—C112—H8E	109.2
C15—C14—H14	116.2	H8C—C112—H8E	109.5
C20—C15—C16	118.8 (4)	H8D—C112—H8E	109.5
C20—C15—C14	122.7 (4)	O14—Cl3—O12	100.74 (18)
C16—C15—C14	118.5 (4)	O14—Cl3—O13	115.71 (17)
C17—C16—C15	122.5 (4)	O12—Cl3—O13	105.76 (17)
C17—C16—H16	118.8	O14—Cl3—O11	110.56 (18)
C15—C16—H16	118.8	O12—Cl3—O11	111.48 (17)
C18—C17—C16	118.8 (4)	O13—Cl3—O11	111.93 (16)
C18—C17—Cl2	122.1 (4)	C28—N1—C27	115.1 (4)
C16—C17—Cl2	119.0 (4)	C28—N1—Ni2	126.2 (3)
C17—C18—C19	123.6 (4)	C27—N1—Ni2	116.5 (3)
C17—C18—H18	118.2	C25—N2—C36	111.6 (3)
C19—C18—H18	118.2	C25—N2—C24	111.6 (3)
C18—C19—C21	121.1 (4)	C36—N2—C24	107.8 (3)
C18—C19—C20	117.8 (4)	C25—N2—Ni2	114.2 (3)
C21—C19—C20	121.1 (4)	C36—N2—Ni2	100.8 (2)
O2—C20—C15	123.4 (4)	C24—N2—Ni2	110.1 (3)
O2—C20—C19	118.3 (4)	C21—N3—C22	119.3 (4)
C15—C20—C19	118.3 (4)	C21—N3—Ni2	122.6 (3)
N3—C21—C19	124.0 (4)	C22—N3—Ni2	117.5 (3)
N3—C21—H21	118.0	C14—N4—C13	115.2 (4)
C19—C21—H21	118.0	C14—N4—Ni1	126.1 (3)
N3—C22—C23	116.5 (4)	C13—N4—Ni1	116.9 (3)
N3—C22—H22A	108.2	C10—N5—C11	111.5 (3)
C23—C22—H22A	108.2	C10—N5—C29	109.4 (3)
N3—C22—H22B	108.2	C11—N5—C29	108.6 (4)
C23—C22—H22B	108.2	C10—N5—Ni1	111.4 (3)
H22A—C22—H22B	107.3	C11—N5—Ni1	115.7 (3)
C22—C23—C24	112.7 (3)	C29—N5—Ni1	99.4 (2)
C22—C23—H23A	109.1	C7—N6—C8	118.7 (3)
C24—C23—H23A	109.1	C7—N6—Ni1	121.6 (3)
C22—C23—H23B	109.1	C8—N6—Ni1	118.7 (3)
C24—C23—H23B	109.1	O2—Ni1—O3	85.94 (12)
H23A—C23—H23B	107.8	O2—Ni1—N6	159.97 (13)

N2—C24—C23	117.7 (3)	O3—Ni1—N6	86.93 (13)
N2—C24—H24A	107.9	O2—Ni1—O1	78.44 (11)
C23—C24—H24A	107.9	O3—Ni1—O1	90.88 (12)
N2—C24—H24B	107.9	N6—Ni1—O1	82.98 (12)
C23—C24—H24B	107.9	O2—Ni1—N4	86.79 (13)
H24A—C24—H24B	107.2	O3—Ni1—N4	165.88 (13)
N2—C25—C26	119.0 (4)	N6—Ni1—N4	103.73 (14)
N2—C25—H25A	107.6	O1—Ni1—N4	99.49 (13)
C26—C25—H25A	107.6	O2—Ni1—N5	107.66 (12)
N2—C25—H25B	107.6	O3—Ni1—N5	87.70 (13)
C26—C25—H25B	107.6	N6—Ni1—N5	90.71 (14)
H25A—C25—H25B	107.0	O1—Ni1—N5	173.61 (12)
C27—C26—C25	116.2 (3)	N4—Ni1—N5	83.00 (14)
C27—C26—H26A	108.2	O1—Ni2—O4	85.63 (11)
C25—C26—H26A	108.2	O1—Ni2—N3	159.00 (13)
C27—C26—H26B	108.2	O4—Ni2—N3	87.96 (13)
C25—C26—H26B	108.2	O1—Ni2—O2	77.61 (11)
H26A—C26—H26B	107.4	O4—Ni2—O2	91.33 (12)
C26—C27—N1	112.0 (4)	N3—Ni2—O2	82.58 (13)
C26—C27—H27A	109.2	O1—Ni2—N1	85.41 (13)
N1—C27—H27A	109.2	O4—Ni2—N1	165.40 (14)
C26—C27—H27B	109.2	N3—Ni2—N1	104.32 (14)
N1—C27—H27B	109.2	O2—Ni2—N1	97.98 (14)
H27A—C27—H27B	107.9	O1—Ni2—N2	108.53 (12)
N1—C28—C2	127.7 (4)	O4—Ni2—N2	89.01 (13)
N1—C28—H28	116.1	N3—Ni2—N2	91.31 (14)
C2—C28—H28	116.1	O2—Ni2—N2	173.86 (12)
C30—C29—N5	117.0 (3)	N1—Ni2—N2	82.94 (14)
C30—C29—H29A	108.1	C1—O1—Ni2	132.8 (2)
N5—C29—H29A	108.1	C1—O1—Ni1	110.1 (2)
C30—C29—H29B	108.1	Ni2—O1—Ni1	98.86 (11)
N5—C29—H29B	108.1	C20—O2—Ni1	132.0 (3)
H29A—C29—H29B	107.3	C20—O2—Ni2	109.9 (2)
C31—C30—C35	118.2 (4)	Ni1—O2—Ni2	99.45 (12)
C31—C30—C29	121.1 (4)	C43—O3—Ni1	129.1 (3)
C35—C30—C29	120.4 (4)	C43—O4—Ni2	128.4 (3)
C30—C31—C32	119.8 (4)		