# research communications

# Crystal structure of [3,10-bis(4-fluorophenethyl)-1,3,5,8,10,12-hexaazacyclotetradecane]nickel(II) diperchlorate

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[a]Department of Chemistry Education, Sunchon National University, 255 Jungang-ro, Sunchon, 57922, South Korea, and [b]Polymerization Manufacturing Technology Research Team, Lotte Chemicals, 334-27 Yeosu Sandan-ro, Yeosu, 59616, South Korea. *Correspondence e-mail: chkwak@sunchon.ac.kr

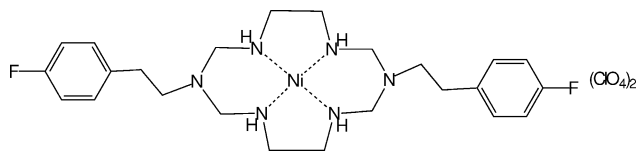The square-planar nickel(II) title complex, $[Ni(C_{24}H_{36}F_2N_6)](ClO_4)_2$ or $[NiL](ClO_4)_2$ ($L$ = 3,10-bis(4-fluorophenethyl)-1,3,5,8,10,12-hexaazacyclotetradecane) was synthesized by a one-pot reaction of template condensation and its X-ray crystal structure was determined. The nickel(II) ion lies close by a twofold axis and the complex displays whole-molecule disorder. Ligand $L$, a hexaazacyclotetradecane ring having 4-fluorophenethyl side chains attached to uncoordinated nitrogen atoms, adopts a trans III ($R,R,S,S$) configuration. The average Ni—N bond distance is 1.934 (9) Å, which is quite similar to those of other nickel(II) complexes with similar ligands. The nickel(II) ion is located 0.051 (7) Å above the least-squares plane through the four coordinated N atoms. The average C—N bond distance and C—N—C angle involving uncoordinated nitrogen atoms are 1.425 (12) Å and 118.0 (9)°, respectively, indicating a significant contribution of $sp^2$ hybridization for these N atoms. The intermolecular N—H···O, C—H···O/F hydrogen bonds of the complex form a network structure, which looks like a seamless floral lace pattern.

## 1. Chemical context

A metal template condensation reaction with formaldehyde and appropriate amines is a useful method for the synthesis of saturated polyazamacrocyclic complexes. It often produces new macrocyclic complexes in one-pot reactions with high yield via selective routes (Salavati-Niasari & Davar, 2006; Salavati-Niasari & Najafian, 2003; Suh, 1996). The introduction of pendant arms into polyazamacrocyclic ligands has, sometimes, changed the structural and chemical properties of the complexes considerably (Hermann et al., 2008; Jee et al., 2003; Alexander, 1995; Kang et al., 1995). The information derived from polyazamacrocyclic complexes containing pendant arms helps in the understanding of apical effects in the biological behavior of tetraazamacrocyclic metalloenzymes having a square-planar geometry (Liang & Sadler, 2004; Costamagna et al., 2000). Furthermore, the donor atoms in the pendant arms of these macrocyclic complexes can be coordinated to another metal ion or participate in hydrogen bonding. Consequently, these complexes can be applied in the field of supramolecular chemistry or metal–organic frameworks. In the nickel(II) complex 8-(pyridin-4-ylmethyl)-1,3,6,8,10,13,15-heptaazatricyclo[13.1.1.1$^{13,15}$]octadecane, intermolecular hydrogen bonding between the nitrogen of the pendant pyridine and coordinated water produces a one-dimensional chain structure (Jee et al., 2003). In particular, many supramolecular

studies including metal–organic frameworks using complexes of 3,10-bis(alkyl)-1,3,5,8,10,12-hexaazacyclotetradecane-type ligands are available because they can be obtained by easy synthetic routes using metal template reactions (Min & Suh, 2001; Kang *et al.*, 1999; Suh *et al.*, 1994). The nickel(II) complex of 3,10-bis(2-cyanoethyl)-1,3,5,8,10,12-hexaazacyclotetradecane produces a coordination polymer with each nickel(II) ion in the macrocycle units coordinating to two nitrile pendant groups of neighboring macrocycles (Suh *et al.*, 1994). In the nickel(II) complex of 3,10-bis(pyridin-4-ylmethyl)-1,3,5,8,10,12-hexaazacyclotetradecane, hydrogen-bonding interactions between nitrogen atoms in pendant pyridine rings, structural water molecules and hydrogen atoms of the secondary amine of the macrocycle link the macrocyclic complexes, resulting in a two-dimensional network (Min & Suh, 2001). In addition, many studies on metal–organic frameworks have been performed using complexes of 3,10-bis(alkyl)-1,3,5,8,10,12-hexaazacyclotetradecane-type ligands (Jeoung *et al.*, 2019; Stackhouse & Ma, 2018; Lee & Moon, 2018; Lin *et al.*, 2014). In this communication, we report the preparation of a new nickel(II) complex [Ni*L*](ClO₄)₂, where *L* is a 3,10-bis(alkyl)-1,3,5,8,10,12-hexaazacyclotetradecane ligand having 4-fluorophenethyl pendant arms at positions 3 and 10, and its structural characterization by single-crystal X-ray crystallography.



## 2. Structural commentary

The molecular structure of the title compound is shown in Fig. 1. Both the complex and perchlorate anion display disorder. The Ni$^{II}$ ion lies close by a special position (twofold axis) and the [Ni*L*]$^{2+}$ complex occurs in two orientations with fixed occupancies of 0.50. The refinement of this whole-mol-
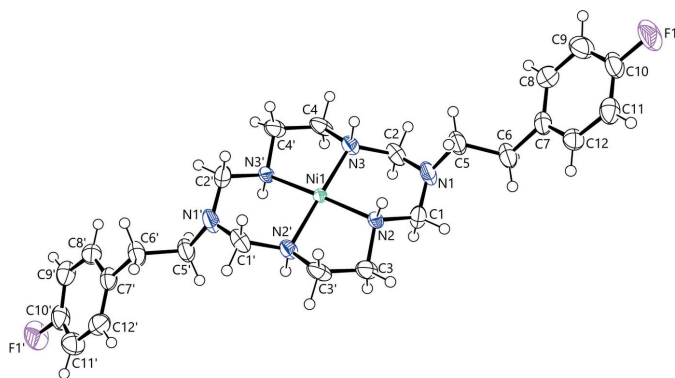


**Figure 1**
Molecular structure of one of the whole-molecule disorder component molecules of [Ni*L*]$^{2+}$ with displacement ellipsoids at 50% probability level. The second disorder component, generated by $(1 - x, y, \frac{1}{2} - z)$ is omitted for clarity.

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Ni1—N3 | 1.925 (7) | N1—C2 | 1.401 (11) |
| Ni1—N2 | 1.933 (9) | N1—C5 | 1.469 (10) |
| Ni1—N3′ | 1.934 (7) | N1′—C1′ | 1.400 (11) |
| Ni1—N2′ | 1.943 (9) | N1′—C2′ | 1.408 (12) |
| N1—C1 | 1.398 (11) | N1′—C5′ | 1.481 (10) |
| | | | |
| N2—Ni1—N3 | 93.7 (4) | C1—N1—C5 | 118.9 (9) |
| N3—Ni1—N3′ | 86.6 (3) | C2—N1—C5 | 119.1 (8) |
| N2—Ni1—N3′ | 176.4 (4) | C1′—N1′—C2′ | 115.6 (7) |
| N2—Ni1—N2′ | 86.5 (2) | C1′—N1′—C5′ | 120.1 (8) |
| C1—N1—C2 | 115.5 (7) | C2′—N1′—C5′ | 118.5 (9) |

ecule disorder needed additional restraints (see *Refinement* section). The occupancies of the disordered perchlorate ion are 0.795 (7) and 0.205 (7). The nickel(II) ion is coordinated to the four nitrogens N2, N3, N2′ and N3′, and the complex has a square-planar coordination geometry. The 14-membered ring skeleton adopts the thermodynamically most stable *trans*-III configuration with *R,R,S,S* chirality of the four coordinated nitrogen atoms (Barefield, 2010). The ligand *L* of the complex has two 4-fluorophenethyl pendant arms attached to the two uncoordinated nitrogens (N1 and N1′) of the 14-membered 1,3,5,8,10,12-hexaazacyclotetradecane ring skeleton. The 4-fluorophenethyl pendants are positioned above and below the square coordination plane. The six-membered chelate rings adopt a chair conformation and the five-membered chelate rings assume a *gauche* conformation.

Selected bond distances and angles are listed in Table 1. The average Ni—N bond distance of 1.934 (9) Å is quite similar to those in square-planar nickel(II) complexes of various other related 14-membered polyaza macrocycles (Kang *et al.*, 1999; Suh *et al.*, 1998; Suh *et al.*, 1996). The bite angles of five-membered chelates are 86.5 (2)° for N2—Ni1—N2′ and 86.6 (3)° for N3—Ni1—N3′, respectively and those of six-membered chelates are 93.7 (4)° for N2—Ni1—N3 and 93.0 (4)° for N2′—Ni1—N3′, respectively. The four coordinating nitrogen atoms (N2, N3, N2′ and N3′) are almost co-planar (r.m.s. deviation 0.010 Å). The nickel(II) ion is located 0.051 (7) Å above this least-squares plane showing a slightly square-pyramidal distortion. The N—C bond distances involving the uncoordinated bridgehead nitrogens (N1 and N1′) range from 1.398 (11) Å (N1—C1) to 1.481 (10) Å (N1′—C5′) and the average N—C bond distance is 1.425 (12) Å, which is significantly shorter than the other N—C single bond distances. Furthermore, the C—N—C bond angles involving these bridgehead nitrogens range from 115.5 (7)° (C1—N1—C2) to 120.1 (8)° (C1′—N1′—C5′) and the average bond angle is 118.0 (9)°, which is distinctly larger than the ideal tetrahedral angle. These results indicate a significant contribution of $sp^2$ hybridization of the bridgehead nitrogen atoms (N1 and N1′) (Min & Suh, 2001; Kang *et al.*, 1999).

## 3. Supramolecular features

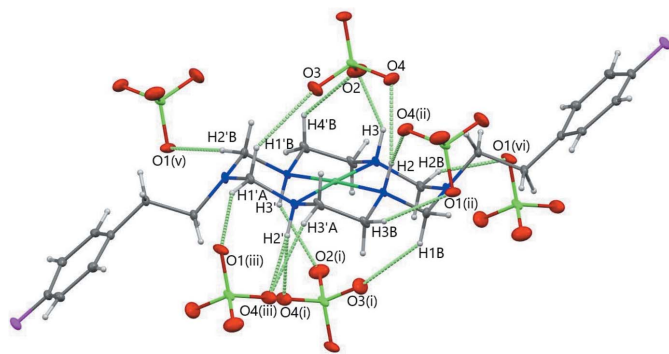There are several N—H⋯*A* (*A* = O) as well as C—H⋯*A* (*A* = O or F) hydrogen bonds in the crystal packing of

**Figure 2**
Hydrogen-bonding interactions involving the perchlorate anions in the crystal packing [Ni*L*](ClO₄)₂. Light-green dashed lines indicate N—H···O and C—H···O hydrogen-bonding interactions. Symmetry codes: (i) $1 - x, y, \frac{1}{2} - z$; (ii) $1 - x, 1 - y, 1 - z$; (iii) $x, -\frac{1}{2} + y, 1 - z$; (v) $\frac{3}{2} - x, \frac{1}{2} - y, 1 - z$; (vi) $-\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$. Only one of the whole-molecule disorder [Ni*L*]²⁺ components and the major component of the perchlorate anion are shown.

[Ni*L*](ClO₄)₂. Hydrogen-bonding interactions between N—H or C—H groups of the ligand *L* and perchlorate oxygen atoms are summarized in Table 2 and illustrated in Fig. 2. In addition, fluorine atom F1 in one of the pendant phenyl groups of the macrocycle is involved in an intermolecular interaction with hydrogen H4*A* of a neighboring molecule (Table 2 and Fig. 3). The other fluorine atom, F1′, takes part in a weaker hydrogen-bonding interaction with H4′*A* of a neighboring molecule [H4*A*···F1′ = 2.62 Å, C4′···F1′ = 3.312 (17) Å and C4′—H4′*A*···F1′ = 128.4 (8)°]. These interactions form a chain structure extending in the [\overline{1}01] direction (Fig. 3). All of these intermolecular hydrogen-bonding interactions lead to a network structure resembling a seamless floral lace pattern (Fig. 4).

## 4. Database survey

An Access Structures search of the Cambridge Structural Database (CSD, *via* CCDC Access Structures, December 2020; Groom *et al.*, 2016) resulted in 97 structures of complexes of 3,10-bis(alkyl)-1,3,5,8,10,12-hexaazacyclotetra-

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O4 | 0.98 | 2.51 | 3.355 (17) | 144 |
| N2—H2···O4ⁱ | 0.98 | 2.44 | 3.260 (16) | 141 |
| N2′—H2′···O4ⁱⁱ | 0.98 | 2.43 | 3.332 (18) | 152 |
| N2′—H2′···O4ⁱⁱⁱ | 0.98 | 2.36 | 3.089 (17) | 131 |
| N3—H3···O2 | 0.98 | 1.97 | 2.819 (11) | 144 |
| N3′—H3′···O2ⁱⁱ | 0.98 | 2.40 | 3.186 (11) | 137 |
| C1′—H1′*A*···O1ⁱⁱⁱ | 0.97 | 2.31 | 3.198 (13) | 151 |
| C1′—H1*B*···O3ⁱⁱ | 0.97 | 2.35 | 3.156 (16) | 140 |
| C1′—H1′*B*···O3 | 0.97 | 2.56 | 3.309 (15) | 134 |
| C2—H2*B*···O1ⁱᵛ | 0.97 | 2.50 | 3.394 (16) | 154 |
| C3—H3*B*···O1ⁱ | 0.97 | 2.48 | 3.35 (2) | 149 |
| C2′—H2′*B*···O1ᵛ | 0.97 | 2.58 | 3.551 (16) | 175 |
| C4—H4*A*···F1ᵛⁱ | 0.97 | 2.54 | 3.341 (19) | 140 |
| C3′—H3′*A*···O4ⁱⁱⁱ | 0.97 | 2.54 | 3.136 (17) | 119 |
| C4′—H4′*B*···O2 | 0.97 | 2.54 | 3.239 (14) | 129 |

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

decane derivatives and 13 structures of complexes of 1,8-bis(alkyl)-1,3,6,8,10,13-hexaazacyclotetradecane (different systematic name of the ligand). However, no results were found for the 3,10-bis(4-fluorophenethyl)-1,3,5,8,10,12-hexaazacyclotetradecane structure.

In addition, 92 structures containing the 1,3,5,8,10,12-hexaazacyclotetradecane skeleton were found during a SciFinder search, but again no results were found containing the title complex. Most are classified as octahedral complexes, while only a few cases are square-planar nickel(II) complexes. The Ni—N bond distances are 1.931 (2)–1.934 (3) Å in the nickel(II) complex of 3,10-bis(2-aminoethyl)-1,3,5,8,10,12-hexaazacyclotetradecane (Kang *et al.*, 1999), 1.934 Å in the nickel(II) complex of 3,10-dibenzyl-1,3,5,8,10,12-hexaazacyclotetradecane (Min & Suh, 2001), and 1.933 (3)–1.936 (3) Å in 1,8-dimethyl-1,3,6,8,10,13-hexaazacyclotetradecane (Benkada *et al.*, 2020), similar to those of the square-planar nickel(II) complexes of various other related 14-membered polyaza macrocycles. The Ni—N distances of 1.933 (4)–1.944 (4) Å in the nickel(II) complex of 1,8-dipentyl-1,3,6,8,10,13-hexaazacyclotetradecane (Park *et al.*, 2015) and the average Ni—N bond distance of 1.941 (6) Å in the nickel(II) complex of 3,10-bis(α-methylnaphthyl)-



**Figure 3**
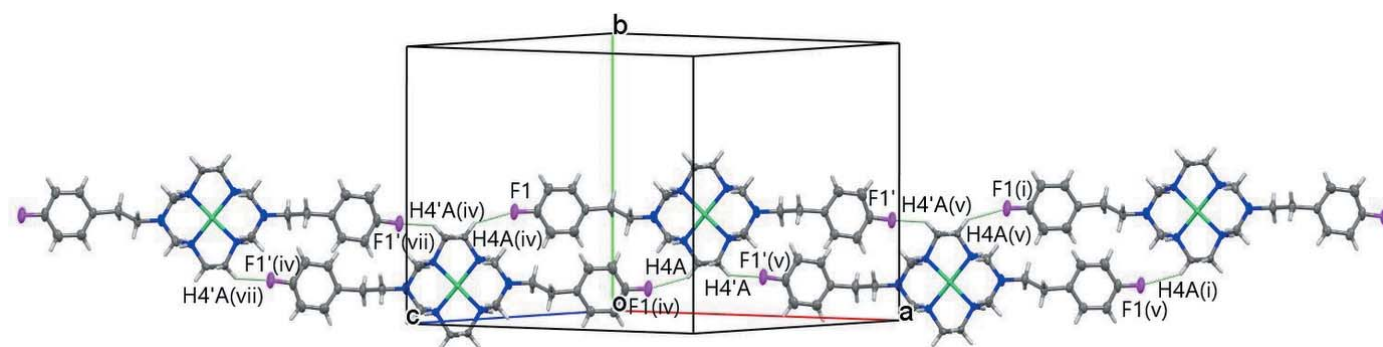A view showing the one-dimensional chain propagation of rings formed by the intermolecular hydrogen bonding between F1···H4*A* and F1′···H4′*A* in [Ni*L*]²⁺. Symmetry codes: (i) $1 - x, y, \frac{1}{2} - z$; (iv) $\frac{1}{2} - x, \frac{1}{2} - y, 1 - z$; (v) $\frac{3}{2} - x, \frac{1}{2} - y, -z$; (vii) $-1 + x, y, 1 + z$. Only one of the whole-molecule disorder [Ni*L*]²⁺ components is shown.
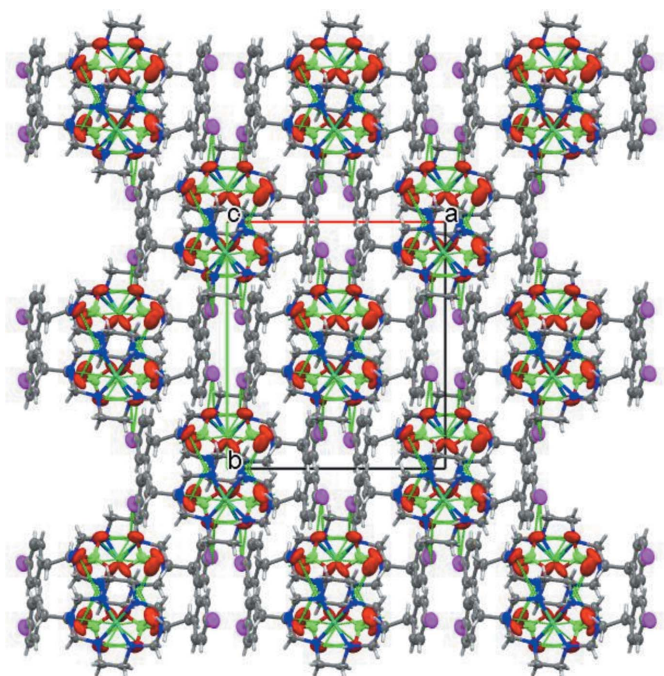
**Figure 4**
A view of the crystal packing of [Ni*L*](ClO4)2, which resembles a seamless floral lace pattern. Light-green dashed lines indicate hydrogen-bonding interactions.

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | [Ni(C24H36F2N6)](ClO4)2 |
| $M_r$ | 704.20 |
| Crystal system, space group | Monoclinic, *C*2/*c* |
| Temperature (K) | 173 |
| *a*, *b*, *c* (Å) | 16.9910 (12), 15.5187 (11), 13.8864 (9) |
| $\beta$ (°) | 126.189 (1) |
| *V* (Å³) | 2955.1 (4) |
| *Z* | 4 |
| Radiation type | Mo *K*α |
| $\mu$ (mm⁻¹) | 0.91 |
| Crystal size (mm) | 0.40 × 0.35 × 0.20 |
| | |
| Data collection | |
| Diffractometer | Bruker SMART CCD area detector |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 9357, 3400, 2737 |
| $R_{int}$ | 0.083 |
| $(\sin \theta/\lambda)_{max}$ (Å⁻¹) | 0.667 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S* | 0.068, 0.159, 1.13 |
| No. of reflections | 3400 |
| No. of parameters | 282 |
| No. of restraints | 492 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å⁻³) | 0.88, −0.63 |

Computer programs: *SMART* and *SAINT* (Bruker, 2002), *SHELXS97* (Sheldrick, 2008), *SHELXL2018/3* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012), and *Mercury* (Macrae *et al.*, 2020).

1,3,5,8,10,12-hexaazacyclotetradecane (Min *et al.*, 2013) are a little longer than those of analogous complexes. However, the Ni—N distances of 1.927 (4)–1.932 (4) Å in the nickel(II) complex of 3,10-bis(2-thiophenemethyl)-1,3,5,8,10,12-hexaazacyclotetradecane (Su *et al.*, 2007) and 1.926 (1)–1.928 (1) Å in that of 3,10-bis(2-hydroxyethyl)-1,3,5,8,10,12-hexaazacyclotetradecane (Kim *et al.*, 2002) are somewhat shorter than those of analogous complexes. In all these nickel(II) complexes of 3,10-bis(alkyl)-1,3,5,8,10,12-hexaazacyclotetradecane analogues, the nickel(II) ion is situated on an inversion center, except for the nickel(II) complex of 3,10-bis($\alpha$-methylnaphthyl)-1,3,5,8,10,12-hexaazacyclotetradecane, which does not have an inversion center due to the chiral pendants of the macrocyclic ligand (Min *et al.*, 2013). The nickel (II) ion is exactly in the least-squares plane through the four coordinating nitrogen atoms.

## 5. Synthesis and crystallization

A well-known one-pot reaction of template condensation was used for the preparation of the title complex (Salavati-Niasari & Rezai-Adaryani, 2004; Min & Suh, 2001; Kang *et al.*, 1999). 98% Ethylenediamine (1.1 ml, 16mmol), 99% 4-fluorophenethylamine (2.1 ml, 16 mmol), and 95% paraformaldehyde (1.44 g, 48 mmol) were slowly added to a stirred solution of 98% nickel(II) acetate tetrahydrate (2.0 g, 8.0 mmol) in 50 ml of methanol. The solution was heated under reflux for 24 h and then cooled to room temperature. The solution was filtered, concentrated HClO4 was added to the filtrate, adjusting pH of the solution to 4, and it was kept in a refrigerator until a yellow-colored precipitate was formed. The product was filtered, washed with methanol, and dried in air. Single crystals for X-ray crystallography were obtained by recrystallization from hot water.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were positioned geometrically and allowed to ride on their respective parent atoms [C—H = 0.93 Å (CH, aromatic), 0.97 Å (CH2) and N—H = 0.98 Å (NH2), and $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.2U_{eq}(N)$].

The refinement of the whole-molecule disorder employed the following constraints and restraints in *SHELXL*: (1) occupancy factors were set at 0.50, (2) the two chemically equivalent halves of the complex were restrained to be similar using the 'SAME' command, (3) the fluorinated benzene rings were given a weak 'FLAT' restraint, (4) Ni1 required a strong 'ISOR' restraint and (5) displacement factors for atom pairs related about the special position were constrained to be equal (EADP).

The perchlorate anion is disordered over two sets of atomic sites with occupancy ratios of 0.795 (7):0.205 (7).

# research communications

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# Crystal structure of [3,10-bis(4-fluorophenethyl)-1,3,5,8,10,12-hexaazacyclotetradecane]nickel(II) diperchlorate

## Chee-Hun Kwak and Mee Chang

**Computing details**

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2018/3* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012), *Mercury* (Macrae *et al.*, 2020); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

**[3,10-Bis(4-fluorophenethyl)-1,3,5,8,10,12-hexaazacyclotetradecane]nickel(II) bis(perchlorate)**

*Crystal data*

| | |
|---|---|
| [Ni($C_{24}H_{36}F_2N_6$)]($ClO_4$)$_2$ | $F(000) = 1464$ |
| $M_r = 704.20$ | $D_x = 1.583$ Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 16.9910$ (12) Å | Cell parameters from 3600 reflections |
| $b = 15.5187$ (11) Å | $\theta = 1.8–28.3°$ |
| $c = 13.8864$ (9) Å | $\mu = 0.91$ mm$^{-1}$ |
| $\beta = 126.189$ (1)° | $T = 173$ K |
| $V = 2955.1$ (4) Å$^3$ | Block, yellow |
| $Z = 4$ | $0.40 \times 0.35 \times 0.20$ mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area detector diffractometer | $R_{int} = 0.083$ |
| phi and $\omega$ scans | $\theta_{max} = 28.3°$, $\theta_{min} = 2.0°$ |
| 9357 measured reflections | $h = -21 \rightarrow 19$ |
| 3400 independent reflections | $k = -19 \rightarrow 18$ |
| 2737 reflections with $I > 2\sigma(I)$ | $l = -18 \rightarrow 15$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.068$ | H-atom parameters constrained |
| $wR(F^2) = 0.159$ | $w = 1/[\sigma^2(F_o^2) + (0.0449P)^2 + 12.0119P]$ |
| $S = 1.13$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3400 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 282 parameters | $\Delta\rho_{max} = 0.88$ e Å$^{-3}$ |
| 492 restraints | $\Delta\rho_{min} = -0.63$ e Å$^{-3}$ |

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Ni1 | 0.50831 (10) | 0.37625 (4) | 0.25924 (13) | 0.0174 (2)* | 0.5 |
| F1 | 0.0633 (8) | 0.3851 (12) | 0.5651 (8) | 0.071 (3) | 0.5 |
| N1 | 0.2930 (6) | 0.3576 (6) | 0.2078 (8) | 0.0457 (18) | 0.5 |
| N2 | 0.4195 (8) | 0.4599 (6) | 0.2494 (14) | 0.0285 (16) | 0.5 |
| H2 | 0.440274 | 0.471036 | 0.330822 | 0.034* | 0.5 |
| N3 | 0.4422 (6) | 0.2796 (5) | 0.2689 (8) | 0.0315 (17) | 0.5 |
| H3 | 0.464913 | 0.275912 | 0.352038 | 0.038* | 0.5 |
| C1 | 0.3129 (7) | 0.4353 (7) | 0.1751 (11) | 0.0438 (19) | 0.5 |
| H1A | 0.276898 | 0.481023 | 0.180846 | 0.053* | 0.5 |
| H1B | 0.289042 | 0.431370 | 0.092254 | 0.053* | 0.5 |
| C2 | 0.3322 (7) | 0.2838 (6) | 0.1930 (9) | 0.0407 (18) | 0.5 |
| H2A | 0.307538 | 0.280024 | 0.109821 | 0.049* | 0.5 |
| H2B | 0.308648 | 0.233687 | 0.210625 | 0.049* | 0.5 |
| C3 | 0.4317 (10) | 0.5416 (7) | 0.2027 (12) | 0.043 (2) | 0.5 |
| H3A | 0.398839 | 0.537629 | 0.117278 | 0.052* | 0.5 |
| H3B | 0.404620 | 0.589837 | 0.218621 | 0.052* | 0.5 |
| C4 | 0.4734 (8) | 0.1987 (5) | 0.2428 (11) | 0.042 (2) | 0.5 |
| H4A | 0.462776 | 0.149576 | 0.277068 | 0.050* | 0.5 |
| H4B | 0.436522 | 0.190069 | 0.157356 | 0.050* | 0.5 |
| C5 | 0.2772 (7) | 0.3584 (7) | 0.3011 (9) | 0.043 (2) | 0.5 |
| H5A | 0.324764 | 0.396071 | 0.365491 | 0.051* | 0.5 |
| H5B | 0.286668 | 0.300782 | 0.333287 | 0.051* | 0.5 |
| C6 | 0.1754 (10) | 0.3892 (15) | 0.2521 (13) | 0.0436 (17) | 0.5 |
| H6A | 0.169116 | 0.448576 | 0.226476 | 0.052* | 0.5 |
| H6B | 0.129079 | 0.355294 | 0.181870 | 0.052* | 0.5 |
| C7 | 0.1472 (8) | 0.3846 (10) | 0.3366 (10) | 0.0368 (12) | 0.5 |
| C8 | 0.1429 (10) | 0.3083 (10) | 0.3863 (11) | 0.048 (3) | 0.5 |
| H8 | 0.157393 | 0.256297 | 0.366611 | 0.057* | 0.5 |
| C9 | 0.1173 (10) | 0.3085 (10) | 0.4650 (11) | 0.054 (3) | 0.5 |
| H9 | 0.119223 | 0.257617 | 0.501639 | 0.065* | 0.5 |
| C10 | 0.0891 (15) | 0.3847 (12) | 0.4885 (15) | 0.0519 (18) | 0.5 |
| C11 | 0.0973 (19) | 0.4610 (12) | 0.447 (2) | 0.046 (2) | 0.5 |
| H11 | 0.084266 | 0.512687 | 0.469261 | 0.055* | 0.5 |
| C12 | 0.126 (2) | 0.4599 (11) | 0.371 (2) | 0.039 (2) | 0.5 |
| H12 | 0.130349 | 0.512186 | 0.342290 | 0.047* | 0.5 |
| F1′ | 0.9281 (8) | 0.3557 (11) | −0.0646 (8) | 0.071 (3) | 0.5 |
| N1′ | 0.7183 (6) | 0.3953 (6) | 0.3006 (8) | 0.0457 (18) | 0.5 |
| N2′ | 0.5718 (8) | 0.4730 (6) | 0.2422 (15) | 0.0315 (17) | 0.5 |
| H2′ | 0.548837 | 0.474605 | 0.158805 | 0.038* | 0.5 |

| | | | | | |
|---|---|---|---|---|---|
| N3′ | 0.5922 (6) | 0.2927 (5) | 0.2582 (8) | 0.0285 (16) | 0.5 |
| H3′ | 0.570787 | 0.286557 | 0.175710 | 0.034* | 0.5 |
| C1′ | 0.6818 (7) | 0.4693 (6) | 0.3192 (10) | 0.0407 (18) | 0.5 |
| H1′A | 0.706114 | 0.519692 | 0.303050 | 0.049* | 0.5 |
| H1′B | 0.706349 | 0.471549 | 0.402388 | 0.049* | 0.5 |
| C2′ | 0.6995 (7) | 0.3171 (7) | 0.3348 (10) | 0.0438 (19) | 0.5 |
| H2′A | 0.722435 | 0.321528 | 0.417275 | 0.053* | 0.5 |
| H2′B | 0.736204 | 0.271471 | 0.330180 | 0.053* | 0.5 |
| C3′ | 0.5385 (10) | 0.5534 (7) | 0.2658 (12) | 0.042 (2) | 0.5 |
| H3′A | 0.551526 | 0.603098 | 0.234804 | 0.050* | 0.5 |
| H3′B | 0.571198 | 0.561228 | 0.350715 | 0.050* | 0.5 |
| C4′ | 0.5786 (8) | 0.2082 (5) | 0.2969 (10) | 0.043 (2) | 0.5 |
| H4′A | 0.600078 | 0.161811 | 0.270542 | 0.052* | 0.5 |
| H4′B | 0.616559 | 0.206027 | 0.383173 | 0.052* | 0.5 |
| C5′ | 0.7323 (7) | 0.3938 (7) | 0.2050 (9) | 0.043 (2) | 0.5 |
| H5′A | 0.695637 | 0.440331 | 0.149100 | 0.051* | 0.5 |
| H5′B | 0.708406 | 0.339732 | 0.161773 | 0.051* | 0.5 |
| C6′ | 0.8384 (11) | 0.4037 (15) | 0.2587 (14) | 0.0436 (17) | 0.5 |
| H6′A | 0.874819 | 0.360440 | 0.320403 | 0.052* | 0.5 |
| H6′B | 0.860033 | 0.459795 | 0.296500 | 0.052* | 0.5 |
| C7′ | 0.8622 (8) | 0.3952 (10) | 0.1701 (10) | 0.0368 (12) | 0.5 |
| C8′ | 0.8807 (9) | 0.3129 (9) | 0.1490 (10) | 0.039 (2) | 0.5 |
| H8′ | 0.875962 | 0.266112 | 0.187145 | 0.047* | 0.5 |
| C9′ | 0.9059 (10) | 0.2994 (10) | 0.0724 (11) | 0.046 (2) | 0.5 |
| H9′ | 0.923553 | 0.245074 | 0.062804 | 0.055* | 0.5 |
| C10′ | 0.9039 (15) | 0.3693 (11) | 0.0113 (15) | 0.0519 (18) | 0.5 |
| C11′ | 0.893 (2) | 0.4515 (12) | 0.034 (2) | 0.054 (3) | 0.5 |
| H11′ | 0.902900 | 0.497780 | −0.000650 | 0.065* | 0.5 |
| C12′ | 0.868 (2) | 0.4641 (12) | 0.112 (2) | 0.048 (3) | 0.5 |
| H12′ | 0.854488 | 0.519427 | 0.124343 | 0.057* | 0.5 |
| Cl1 | 0.6115 (4) | 0.3604 (3) | 0.5699 (5) | 0.0469 (4) | 0.795 (7) |
| O1 | 0.6801 (6) | 0.3549 (5) | 0.6978 (5) | 0.082 (3) | 0.795 (7) |
| O2 | 0.5813 (5) | 0.2776 (4) | 0.5188 (5) | 0.102 (2) | 0.795 (7) |
| O3 | 0.6555 (5) | 0.3981 (6) | 0.5233 (6) | 0.113 (3) | 0.795 (7) |
| O4 | 0.5284 (5) | 0.4091 (5) | 0.5368 (6) | 0.089 (2) | 0.795 (7) |
| Cl1′ | 0.6086 (16) | 0.3675 (13) | 0.5680 (19) | 0.0469 (4) | 0.205 (7) |
| O1′ | 0.6497 (18) | 0.3241 (14) | 0.6781 (19) | 0.049 (4) | 0.205 (7) |
| O2′ | 0.6325 (17) | 0.3288 (16) | 0.4980 (16) | 0.074 (4) | 0.205 (7) |
| O3′ | 0.6267 (19) | 0.4551 (13) | 0.574 (2) | 0.097 (6) | 0.205 (7) |
| O4′ | 0.5058 (15) | 0.3669 (16) | 0.5057 (19) | 0.067 (4) | 0.205 (7) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.074 (3) | 0.112 (11) | 0.060 (2) | 0.010 (4) | 0.0571 (19) | 0.004 (3) |
| N1 | 0.035 (2) | 0.063 (6) | 0.054 (3) | −0.001 (2) | 0.034 (2) | 0.005 (3) |
| N2 | 0.034 (3) | 0.030 (3) | 0.034 (3) | 0.007 (2) | 0.028 (3) | 0.006 (3) |
| N3 | 0.037 (3) | 0.031 (3) | 0.039 (4) | −0.003 (3) | 0.029 (3) | −0.002 (3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C1 | 0.031 (3) | 0.057 (5) | 0.049 (4) | 0.012 (3) | 0.026 (3) | 0.011 (4) |
| C2 | 0.038 (3) | 0.050 (4) | 0.043 (4) | −0.014 (3) | 0.029 (3) | −0.007 (3) |
| C3 | 0.067 (6) | 0.031 (3) | 0.060 (6) | 0.013 (3) | 0.054 (5) | 0.010 (3) |
| C4 | 0.073 (7) | 0.022 (3) | 0.062 (6) | −0.003 (3) | 0.057 (6) | −0.001 (3) |
| C5 | 0.033 (3) | 0.061 (8) | 0.045 (3) | −0.001 (3) | 0.028 (2) | 0.002 (3) |
| C6 | 0.032 (4) | 0.066 (6) | 0.040 (3) | −0.003 (4) | 0.025 (3) | −0.003 (5) |
| C7 | 0.029 (2) | 0.052 (3) | 0.033 (2) | 0.003 (5) | 0.020 (2) | −0.002 (4) |
| C8 | 0.045 (6) | 0.053 (4) | 0.046 (7) | 0.009 (4) | 0.027 (6) | 0.010 (4) |
| C9 | 0.051 (7) | 0.073 (5) | 0.041 (7) | 0.000 (5) | 0.029 (6) | 0.011 (5) |
| C10 | 0.041 (3) | 0.086 (6) | 0.041 (2) | 0.010 (6) | 0.031 (2) | 0.008 (5) |
| C11 | 0.036 (5) | 0.068 (4) | 0.032 (5) | 0.007 (4) | 0.020 (5) | −0.003 (4) |
| C12 | 0.036 (5) | 0.052 (4) | 0.028 (5) | 0.003 (4) | 0.019 (4) | 0.003 (4) |
| F1′ | 0.074 (3) | 0.112 (11) | 0.060 (2) | 0.010 (4) | 0.0571 (19) | 0.004 (3) |
| N1′ | 0.035 (2) | 0.063 (6) | 0.054 (3) | −0.001 (2) | 0.034 (2) | 0.005 (3) |
| N2′ | 0.037 (3) | 0.031 (3) | 0.039 (4) | −0.003 (3) | 0.029 (3) | −0.002 (3) |
| N3′ | 0.034 (3) | 0.030 (3) | 0.034 (3) | 0.007 (2) | 0.028 (3) | 0.006 (3) |
| C1′ | 0.038 (3) | 0.050 (4) | 0.043 (4) | −0.014 (3) | 0.029 (3) | −0.007 (3) |
| C2′ | 0.031 (3) | 0.057 (5) | 0.049 (4) | 0.012 (3) | 0.026 (3) | 0.011 (4) |
| C3′ | 0.073 (7) | 0.022 (3) | 0.062 (6) | −0.003 (3) | 0.057 (6) | −0.001 (3) |
| C4′ | 0.067 (6) | 0.031 (3) | 0.060 (6) | 0.013 (3) | 0.054 (5) | 0.010 (3) |
| C5′ | 0.033 (3) | 0.061 (8) | 0.045 (3) | −0.001 (3) | 0.028 (2) | 0.002 (3) |
| C6′ | 0.032 (4) | 0.066 (6) | 0.040 (3) | −0.003 (4) | 0.025 (3) | −0.003 (5) |
| C7′ | 0.029 (2) | 0.052 (3) | 0.033 (2) | 0.003 (5) | 0.020 (2) | −0.002 (4) |
| C8′ | 0.036 (5) | 0.052 (4) | 0.028 (5) | 0.003 (4) | 0.019 (4) | 0.003 (4) |
| C9′ | 0.036 (5) | 0.068 (4) | 0.032 (5) | 0.007 (4) | 0.020 (5) | −0.003 (4) |
| C10′ | 0.041 (3) | 0.086 (6) | 0.041 (2) | 0.010 (6) | 0.031 (2) | 0.008 (5) |
| C11′ | 0.051 (7) | 0.073 (5) | 0.041 (7) | 0.000 (5) | 0.029 (6) | 0.011 (5) |
| C12′ | 0.045 (6) | 0.053 (4) | 0.046 (7) | 0.009 (4) | 0.027 (6) | 0.010 (4) |
| Cl1 | 0.0583 (8) | 0.0582 (11) | 0.0380 (6) | 0.0229 (7) | 0.0361 (6) | 0.0149 (7) |
| O1 | 0.089 (5) | 0.116 (6) | 0.038 (3) | 0.058 (4) | 0.035 (3) | 0.014 (3) |
| O2 | 0.119 (5) | 0.063 (3) | 0.077 (4) | 0.015 (3) | 0.032 (3) | 0.012 (3) |
| O3 | 0.113 (5) | 0.162 (6) | 0.094 (4) | −0.035 (4) | 0.077 (4) | 0.015 (4) |
| O4 | 0.081 (4) | 0.108 (5) | 0.062 (4) | 0.056 (4) | 0.033 (3) | 0.010 (3) |
| Cl1′ | 0.0583 (8) | 0.0582 (11) | 0.0380 (6) | 0.0229 (7) | 0.0361 (6) | 0.0149 (7) |
| O1′ | 0.068 (8) | 0.052 (8) | 0.050 (7) | 0.028 (6) | 0.047 (6) | 0.017 (6) |
| O2′ | 0.093 (8) | 0.095 (9) | 0.039 (6) | 0.040 (8) | 0.042 (6) | 0.016 (7) |
| O3′ | 0.108 (9) | 0.081 (8) | 0.084 (8) | 0.002 (7) | 0.046 (7) | 0.013 (6) |
| O4′ | 0.073 (7) | 0.093 (9) | 0.050 (7) | 0.007 (7) | 0.044 (5) | 0.008 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Ni1—N3 | 1.925 (7) | N1′—C1′ | 1.400 (11) |
| Ni1—N2 | 1.933 (9) | N1′—C2′ | 1.408 (12) |
| Ni1—N3′ | 1.934 (7) | N1′—C5′ | 1.481 (10) |
| Ni1—N2′ | 1.943 (9) | N2′—C3′ | 1.484 (10) |
| F1—C10 | 1.370 (10) | N2′—C1′ | 1.510 (11) |
| N1—C1 | 1.398 (11) | N2′—H2′ | 0.9800 |
| N1—C2 | 1.401 (11) | N3′—C4′ | 1.486 (10) |

| | | | |
|---|---|---|---|
| N1—C5 | 1.469 (10) | N3′—C2′ | 1.519 (12) |
| N2—C3 | 1.495 (11) | N3′—H3′ | 0.9800 |
| N2—C1 | 1.511 (11) | C1′—H1′A | 0.9700 |
| N2—H2 | 0.9800 | C1′—H1′B | 0.9700 |
| N3—C4 | 1.488 (9) | C2′—H2′A | 0.9700 |
| N3—C2 | 1.511 (12) | C2′—H2′B | 0.9700 |
| N3—H3 | 0.9800 | C3′—H3′A | 0.9700 |
| C1—H1A | 0.9700 | C3′—H3′B | 0.9700 |
| C1—H1B | 0.9700 | C4′—H4′A | 0.9700 |
| C2—H2A | 0.9700 | C4′—H4′B | 0.9700 |
| C2—H2B | 0.9700 | C5′—C6′ | 1.498 (12) |
| C3—C3′ | 1.489 (13) | C5′—H5′A | 0.9700 |
| C3—H3A | 0.9700 | C5′—H5′B | 0.9700 |
| C3—H3B | 0.9700 | C6′—C7′ | 1.510 (10) |
| C4—C4′ | 1.482 (17) | C6′—H6′A | 0.9700 |
| C4—H4A | 0.9700 | C6′—H6′B | 0.9700 |
| C4—H4B | 0.9700 | C7′—C12′ | 1.380 (11) |
| C5—C6 | 1.515 (11) | C7′—C8′ | 1.387 (11) |
| C5—H5A | 0.9700 | C8′—C9′ | 1.377 (11) |
| C5—H5B | 0.9700 | C8′—H8′ | 0.9300 |
| C6—C7 | 1.509 (10) | C9′—C10′ | 1.366 (12) |
| C6—H6A | 0.9700 | C9′—H9′ | 0.9300 |
| C6—H6B | 0.9700 | C10′—C11′ | 1.351 (12) |
| C7—C8 | 1.393 (11) | C11′—C12′ | 1.395 (12) |
| C7—C12 | 1.394 (11) | C11′—H11′ | 0.9300 |
| C8—C9 | 1.394 (12) | C12′—H12′ | 0.9300 |
| C8—H8 | 0.9300 | Cl1—O3 | 1.376 (7) |
| C9—C10 | 1.385 (12) | Cl1—O2 | 1.410 (7) |
| C9—H9 | 0.9300 | Cl1—O4 | 1.418 (6) |
| C10—C11 | 1.357 (12) | Cl1—O1 | 1.440 (6) |
| C11—C12 | 1.396 (11) | Cl1′—O3′ | 1.386 (17) |
| C11—H11 | 0.9300 | Cl1′—O2′ | 1.392 (17) |
| C12—H12 | 0.9300 | Cl1′—O4′ | 1.420 (17) |
| F1′—C10′ | 1.356 (10) | Cl1′—O1′ | 1.421 (16) |
| | | | |
| N2—Ni1—N3 | 93.7 (4) | C3′—N2′—C1′ | 110.4 (9) |
| N3—Ni1—N3′ | 86.6 (3) | C3′—N2′—Ni1 | 108.1 (8) |
| N2—Ni1—N3′ | 176.4 (4) | C1′—N2′—Ni1 | 115.4 (8) |
| N3—Ni1—N2′ | 177.5 (5) | C3′—N2′—H2′ | 107.5 |
| N2—Ni1—N2′ | 86.5 (2) | C1′—N2′—H2′ | 107.5 |
| N3′—Ni1—N2′ | 93.0 (4) | Ni1—N2′—H2′ | 107.5 |
| C1—N1—C2 | 115.5 (7) | C4′—N3′—C2′ | 110.2 (8) |
| C1—N1—C5 | 118.9 (9) | C4′—N3′—Ni1 | 108.5 (6) |
| C2—N1—C5 | 119.1 (8) | C2′—N3′—Ni1 | 114.4 (6) |
| C3—N2—C1 | 109.6 (9) | C4′—N3′—H3′ | 107.9 |
| C3—N2—Ni1 | 107.3 (7) | C2′—N3′—H3′ | 107.9 |
| C1—N2—Ni1 | 116.7 (7) | Ni1—N3′—H3′ | 107.9 |
| C3—N2—H2 | 107.6 | N1′—C1′—N2′ | 113.4 (8) |

| | | | |
|---|---|---|---|
| C1—N2—H2 | 107.6 | N1′—C1′—H1′A | 108.9 |
| Ni1—N2—H2 | 107.6 | N2′—C1′—H1′A | 108.9 |
| C4—N3—C2 | 109.7 (7) | N1′—C1′—H1′B | 108.9 |
| C4—N3—Ni1 | 109.6 (5) | N2′—C1′—H1′B | 108.9 |
| C2—N3—Ni1 | 116.8 (6) | H1′A—C1′—H1′B | 107.7 |
| C4—N3—H3 | 106.7 | N1′—C2′—N3′ | 113.3 (7) |
| C2—N3—H3 | 106.7 | N1′—C2′—H2′A | 108.9 |
| Ni1—N3—H3 | 106.7 | N3′—C2′—H2′A | 108.9 |
| N1—C1—N2 | 114.7 (8) | N1′—C2′—H2′B | 108.9 |
| N1—C1—H1A | 108.6 | N3′—C2′—H2′B | 108.9 |
| N2—C1—H1A | 108.6 | H2′A—C2′—H2′B | 107.7 |
| N1—C1—H1B | 108.6 | N2′—C3′—C3 | 105.0 (11) |
| N2—C1—H1B | 108.6 | N2′—C3′—H3′A | 110.8 |
| H1A—C1—H1B | 107.6 | C3—C3′—H3′A | 110.8 |
| N1—C2—N3 | 115.7 (7) | N2′—C3′—H3′B | 110.8 |
| N1—C2—H2A | 108.4 | C3—C3′—H3′B | 110.8 |
| N3—C2—H2A | 108.4 | H3′A—C3′—H3′B | 108.8 |
| N1—C2—H2B | 108.4 | C4—C4′—N3′ | 107.6 (8) |
| N3—C2—H2B | 108.4 | C4—C4′—H4′A | 110.2 |
| H2A—C2—H2B | 107.4 | N3′—C4′—H4′A | 110.2 |
| C3′—C3—N2 | 106.4 (10) | C4—C4′—H4′B | 110.2 |
| C3′—C3—H3A | 110.4 | N3′—C4′—H4′B | 110.2 |
| N2—C3—H3A | 110.4 | H4′A—C4′—H4′B | 108.5 |
| C3′—C3—H3B | 110.4 | N1′—C5′—C6′ | 109.6 (8) |
| N2—C3—H3B | 110.4 | N1′—C5′—H5′A | 109.8 |
| H3A—C3—H3B | 108.6 | C6′—C5′—H5′A | 109.8 |
| C4′—C4—N3 | 106.8 (8) | N1′—C5′—H5′B | 109.8 |
| C4′—C4—H4A | 110.4 | C6′—C5′—H5′B | 109.8 |
| N3—C4—H4A | 110.4 | H5′A—C5′—H5′B | 108.2 |
| C4′—C4—H4B | 110.4 | C5′—C6′—C7′ | 114.0 (8) |
| N3—C4—H4B | 110.4 | C5′—C6′—H6′A | 108.7 |
| H4A—C4—H4B | 108.6 | C7′—C6′—H6′A | 108.7 |
| N1—C5—C6 | 111.1 (8) | C5′—C6′—H6′B | 108.7 |
| N1—C5—H5A | 109.4 | C7′—C6′—H6′B | 108.7 |
| C6—C5—H5A | 109.4 | H6′A—C6′—H6′B | 107.6 |
| N1—C5—H5B | 109.4 | C12′—C7′—C8′ | 119.1 (9) |
| C6—C5—H5B | 109.4 | C12′—C7′—C6′ | 123.8 (11) |
| H5A—C5—H5B | 108.0 | C8′—C7′—C6′ | 117.1 (11) |
| C7—C6—C5 | 116.0 (8) | C9′—C8′—C7′ | 121.1 (10) |
| C7—C6—H6A | 108.3 | C9′—C8′—H8′ | 119.4 |
| C5—C6—H6A | 108.3 | C7′—C8′—H8′ | 119.4 |
| C7—C6—H6B | 108.3 | C10′—C9′—C8′ | 117.0 (10) |
| C5—C6—H6B | 108.3 | C10′—C9′—H9′ | 121.5 |
| H6A—C6—H6B | 107.4 | C8′—C9′—H9′ | 121.5 |
| C8—C7—C12 | 116.1 (9) | C11′—C10′—F1′ | 118.1 (11) |
| C8—C7—C6 | 124.0 (11) | C11′—C10′—C9′ | 124.3 (10) |
| C12—C7—C6 | 119.9 (11) | F1′—C10′—C9′ | 116.8 (12) |
| C7—C8—C9 | 121.1 (11) | C10′—C11′—C12′ | 117.2 (13) |

| | | | |
|---|---|---|---|
| C7—C8—H8 | 119.4 | C10′—C11′—H11′ | 121.4 |
| C9—C8—H8 | 119.4 | C12′—C11′—H11′ | 121.4 |
| C10—C9—C8 | 119.9 (11) | C7′—C12′—C11′ | 120.5 (13) |
| C10—C9—H9 | 120.0 | C7′—C12′—H12′ | 119.8 |
| C8—C9—H9 | 120.0 | C11′—C12′—H12′ | 119.8 |
| C11—C10—F1 | 118.8 (12) | O3—Cl1—O2 | 106.7 (6) |
| C11—C10—C9 | 120.7 (10) | O3—Cl1—O4 | 109.5 (6) |
| F1—C10—C9 | 120.1 (11) | O2—Cl1—O4 | 109.0 (6) |
| C10—C11—C12 | 118.4 (12) | O3—Cl1—O1 | 109.6 (6) |
| C10—C11—H11 | 120.8 | O2—Cl1—O1 | 110.9 (5) |
| C12—C11—H11 | 120.8 | O4—Cl1—O1 | 110.9 (5) |
| C7—C12—C11 | 123.4 (12) | O3′—Cl1′—O2′ | 108.4 (19) |
| C7—C12—H12 | 118.3 | O3′—Cl1′—O4′ | 101.1 (17) |
| C11—C12—H12 | 118.3 | O2′—Cl1′—O4′ | 109.5 (18) |
| C1′—N1′—C2′ | 115.6 (7) | O3′—Cl1′—O1′ | 116.9 (18) |
| C1′—N1′—C5′ | 120.1 (8) | O2′—Cl1′—O1′ | 112.8 (18) |
| C2′—N1′—C5′ | 118.5 (9) | O4′—Cl1′—O1′ | 107.4 (18) |
| | | | |
| C2—N1—C1—N2 | −64.3 (13) | C5′—N1′—C1′—N2′ | 86.6 (11) |
| C5—N1—C1—N2 | 87.3 (12) | C3′—N2′—C1′—N1′ | −179.4 (10) |
| C3—N2—C1—N1 | 177.6 (10) | Ni1—N2′—C1′—N1′ | 57.6 (13) |
| Ni1—N2—C1—N1 | 55.4 (13) | C1′—N1′—C2′—N3′ | 67.6 (12) |
| C1—N1—C2—N3 | 63.7 (12) | C5′—N1′—C2′—N3′ | −85.8 (10) |
| C5—N1—C2—N3 | −87.7 (11) | C4′—N3′—C2′—N1′ | 178.0 (8) |
| C4—N3—C2—N1 | −179.3 (8) | Ni1—N3′—C2′—N1′ | −59.5 (10) |
| Ni1—N3—C2—N1 | −53.9 (10) | C1′—N2′—C3′—C3 | −169.8 (8) |
| C1—N2—C3—C3′ | −170.4 (8) | Ni1—N2′—C3′—C3 | −42.7 (11) |
| Ni1—N2—C3—C3′ | −42.7 (11) | N2—C3—C3′—N2′ | 55.7 (7) |
| C2—N3—C4—C4′ | 166.7 (7) | N3—C4—C4′—N3′ | −49.6 (8) |
| Ni1—N3—C4—C4′ | 37.3 (9) | C2′—N3′—C4′—C4 | 165.1 (7) |
| C1—N1—C5—C6 | 78.6 (14) | Ni1—N3′—C4′—C4 | 39.3 (9) |
| C2—N1—C5—C6 | −130.9 (14) | C1′—N1′—C5′—C6′ | 103.5 (13) |
| N1—C5—C6—C7 | 174.2 (12) | C2′—N1′—C5′—C6′ | −104.4 (13) |
| C5—C6—C7—C8 | −61.5 (16) | N1′—C5′—C6′—C7′ | 174.9 (12) |
| C5—C6—C7—C12 | 117 (2) | C5′—C6′—C7′—C12′ | 93 (2) |
| C12—C7—C8—C9 | 0.7 (16) | C5′—C6′—C7′—C8′ | −88.0 (15) |
| C6—C7—C8—C9 | 179.3 (10) | C12′—C7′—C8′—C9′ | 1.0 (16) |
| C7—C8—C9—C10 | 4 (2) | C6′—C7′—C8′—C9′ | −178.0 (10) |
| C8—C9—C10—C11 | −8 (2) | C7′—C8′—C9′—C10′ | −5.3 (19) |
| C8—C9—C10—F1 | 179.6 (14) | C8′—C9′—C10′—C11′ | 10 (3) |
| F1—C10—C11—C12 | 179 (2) | C8′—C9′—C10′—F1′ | −179.6 (13) |
| C9—C10—C11—C12 | 6 (3) | F1′—C10′—C11′—C12′ | 180 (2) |
| C8—C7—C12—C11 | −3 (3) | C9′—C10′—C11′—C12′ | −11 (3) |
| C6—C7—C12—C11 | 178.8 (18) | C8′—C7′—C12′—C11′ | −1 (3) |
| C10—C11—C12—C7 | −1 (4) | C6′—C7′—C12′—C11′ | 177.8 (18) |
| C2′—N1′—C1′—N2′ | −66.3 (12) | C10′—C11′—C12′—C7′ | 6 (4) |

*Hydrogen-bond geometry (Å, º)*

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O4 | 0.98 | 2.51 | 3.355 (17) | 144 |
| N2—H2···O4$^{i}$ | 0.98 | 2.44 | 3.260 (16) | 141 |
| N2′—H2′···O4$^{ii}$ | 0.98 | 2.43 | 3.332 (18) | 152 |
| N2′—H2′···O4$^{iii}$ | 0.98 | 2.36 | 3.089 (17) | 131 |
| N3—H3···O2 | 0.98 | 1.97 | 2.819 (11) | 144 |
| N3′—H3′···O2$^{ii}$ | 0.98 | 2.40 | 3.186 (11) | 137 |
| C1′—H1′$A$···O1$^{iii}$ | 0.97 | 2.31 | 3.198 (13) | 151 |
| C1—H1$B$···O3$^{ii}$ | 0.97 | 2.35 | 3.156 (16) | 140 |
| C1′—H1′$B$···O3 | 0.97 | 2.56 | 3.309 (15) | 134 |
| C2—H2$B$···O1$^{iv}$ | 0.97 | 2.50 | 3.394 (16) | 154 |
| C3—H3$B$···O1$^{i}$ | 0.97 | 2.48 | 3.35 (2) | 149 |
| C2′—H2′$B$···O1$^{v}$ | 0.97 | 2.58 | 3.551 (16) | 175 |
| C4—H4$A$···F1$^{vi}$ | 0.97 | 2.54 | 3.341 (19) | 140 |
| C3′—H3′$A$···O4$^{iii}$ | 0.97 | 2.54 | 3.136 (17) | 119 |
| C4′—H4′$B$···O2 | 0.97 | 2.54 | 3.239 (14) | 129 |

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