A tetranuclear nickel(II) complex, [Ni_4(L)_4](ClO_4)_4·C_2H_3N·2H_2O, with an asymmetric Ni_4O_4 open-cubane-like core

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A tetranuclear complex with an open-cubane-like core structure was synthesized from 2-methoxy-6-(pyridin-2-yl-hydrazonomethyl)phenol (HL), namely, cyclo-tetrakis(µ-2-methoxy-6-[2-(pyridin-2-yl)hydrazin-1-ylidene]-methyl)phenolato]tetranickel(II) tetrakis(perchlorate) acetonitrile monosolvate dihydrate, [Ni_4(C_18H_12N_3O_2)_4](ClO_4)_4·C_2H_3N·2H_2O, and characterized using micro-analytical and spectroscopic techniques. The crystal-structure determination reveals the formation of a distorted Ni_4O_4 cubane-like core architecture encapsulated by four hydrazone Schiff base (HL) molecules. An open-cube tetranuclear architecture is created in which nickel(II) ions of the NiN_2O_3 unit are connected by µ_2-O anions of the phenolate moiety of HL. In this complex, each Ni centre has a slightly distorted square-pyramidal coordination environment. The supramolecular architectures are stabilized via the presence of various intermolecular hydrogen bonds and (aryl–aryl, aryl–chelate and chelate–chelate) stacking interactions.

1. Chemical context

Polynuclear metal(II) complexes have attracted much attention owing to their structural variety and significant applications in biology, catalysis, molecular recognition and magnetism (Alcantara et al., 2006; Powell, 2003). As such, complexes containing a tetranuclear cubane-like core have been an important class of compounds (Yang et al., 2005). The synthesis of such polynuclear metal complexes can often be promoted with the use of polydentate Schiff base ligands possessing nitrogen and oxygen donor atoms. Such Schiff bases are known to form high nuclearity complexes with interesting architectures, and the hydroxyl groups and other donor atoms are often suitable for the synthesis of polynuclear complexes (Gungor & Kara, 2015; Dutta et al., 2020; Shit et al., 2013). Several tetranuclear nickel(II) complexes have also been synthesized and their different electronic properties explored (Lin et al., 2011; Nihei et al., 2003; Zhang et al., 2012; Liu et al., 2012; Shit et al., 2013). As part of our study of polynuclear complexes, we have been interested in cubane-like structures to build complexes with high nuclearity (Ray et al., 2009; Chakraborthy et al., 2009; Sagar et al., 2017; Pouralimardan et al., 2007; Patel et al., 2019). In this article, the results were obtained with the Schiff base ligand (HL) 2-methoxy-6-(pyridin-2-yl-hydrazonomethyl)-phenol, which can bind one or two metal ions, simultaneously. The stoichiometric reaction of nickel(II) perchlorate hexahydrate with this ligand resulted the formation of Ni_4O_4 distorted cubane-like structure described herein.
2. Structural commentary

The hydrazone Schiff base (HL) was prepared by the reaction of 2-hydrazinopyridine and 2-hydroxy-3-methoxybenzaldehyde in a 1:1 ratio in ethanol. The reaction of nickel perchlorate hexahydrate and the HL ligand yielded a tetranuclear open-cubane-like complex with an Ni$_4$O$_4$ core-type architecture. The tetranuclear complex is formulated as [Ni$_4$(L)$_4$(ClO$_4$)$_4$]$_4$C$_2$H$_3$N$_2$H$_2$O (Fig. 1). Selected bond parameters are given in Table 1. The crystal-structure analysis reveals the formation of a distorted Ni$_4$O$_4$ cubane-like core. In this complex, four HL molecules coordinate to the four nickel centres as a pentadentate ligand (Fig. 2). The deprotonated Schiff base (L$^-$) ligand coordinates in a pentadentate mode ($\mu_2$-O$_{phenolate}$, $\eta^1$-N$_{imin}$, $\eta^1$-N$_{pyridin}$, $\eta^1$-O$_{methoxy}$), thus forming eight fused metal chelate rings (four five-membered and four six-membered rings). Such a coordination pattern results in a distorted square-pyramidal coordination sphere around each nickel(II) ion. The distortion in the square-pyramidal geometry is shown by the $\tau$ index ($\tau_\alpha$, with values of 0 for a perfect square pyramid and 1 for a perfect trigonal bipyramid; Addison et al., 1984). The values for each Ni$^{II}$ ion are 0.0383 for Ni1, 0.0050 for Ni2, 0.0033 for Ni3 and 0.0250 for Ni4. The fact that the $\tau$-values are very close to zero indicates that the

| Bond Distance or Angle | Value |
|------------------------|-------|

Table 1

Selected geometric parameters (Å, °).

Figure 1

Molecular structure of the tetranuclear nickel complex, [Ni$_4$(L)$_4$(ClO$_4$)$_4$]$_4$C$_2$H$_3$N$_2$H$_2$O. Displacement ellipsoids are drawn at the 30% probability level.

Figure 2

Ball-and-stick figure of the tetranuclear nickel complex, illustrating the coordination sphere about the nickel centres.

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geometries around each Ni centre are slightly distorted from a perfect square-pyramidal environment.

The hydroxyl group of each HL phenol is deprotonated and the oxygen atoms bridge two nickel centres. Similarly, the oxygen atom of the methoxy group coordinates to a second nickel centre in a $\mu_2$-mode. Each nickel centre is connected to the $\mu_2$-oxygen atoms, resulting in the construction of an Ni$_4$O$_4$ cubane-like core (Fig. 2). The basal plane of each nickel centre is constituted by one phenoxy oxygen, one methoxy oxygen, one azomethine nitrogen and one pyridine nitrogen atom. As a result of its weakly coordinating nature, each methoxy oxygen remains in an axial position. The Ni–N/O bond lengths are in the range 1.932 (7)–1.988 (5) Å and are very close to those reported for similar tetranuclear cubane-core-type complexes (Zhang et al., 2011, 2013; Yu et al., 2011; Tong et al., 2002; Mandal et al., 2008; Clemente-Juan et al., 2000; Li et al., 2006; Sun et al., 2011; Saha et al., 2014; Yang et al., 2006).

### 3. Supramolecular features

In the polynuclear crystal, intermolecular hydrogen-bonding interactions are detected involving C–H and N–H donors from the hydrazone Schiff base and acceptor oxygen atoms of perchlorate counter-ions and solvate water molecules (Fig. 3). The important hydrogen-bonding parameters are collected in Table 2. The two tetranuclear complexes are interconnected through intermolecular hydrogen bonding between C–H⋅⋅⋅O and N–H⋅⋅⋅O hydrogen bonds with the perchlorate ions, forming heterosynths (Fig. 3). Additionally, oxygen atoms of solvate water molecules also act as acceptor atoms for intermolecular hydrogen bonds. Furthermore, stabilization of the tetranuclear crystal lattice is facilitated by the presence of various weak (aryl–aryl, aryl–chelate and chelate–chelate) intramolecular stacking interactions (Fig. 4). The orthorhombic cell contains four formula units, and the packing is shown
in Fig. 5. The entire stacking pattern reveals that the intermolecular hydrogen bonds remain between perchlorate counter-ions and C–H/N–H moieties of the same molecule or adjacent molecules. Similarly, solvate water molecules also exert cooperative intermolecular hydrogen bonds from C—H/N—H moieties of the complex, and the crystal lattice is also stabilized via π–π stacking interactions [centroid–centroid distances = 3.343 (3)–3.668 (3) Å].

4. Database survey
A search of the Cambridge Structural Database (CSD; Groom et al., 2016) for 2-methoxy-6-(pyridin-2-yl-hydrazonomethyl)phenol gave no results. Several tetranuclear nickel complexes have been synthesized with several Schiff base ligands (Lin et al., 2011; Liu et al., 2012; Nihei et al., 2003; Saha et al., 2014; Shit et al., 2013; Zhang et al., 2012).

5. Synthesis and crystallization
A mixture of 2-hydrazinopyridine (0.327 g, 3.0 mmol) and 2-hydroxy-3-methoxybenzaldehyde (0.456 g, 3.0 mmol) in 30 mL of ethanol was refluxed for 3 h. The resulting light-yellow solution was cooled to room temperature. The obtained crystalline material was filtered off, washed with ethanol and kept in a CaCl₂ desiccator. Yield 80%. Analysis calculated for C₁₃H₁₂N₃O₂: C, 64.18; H, 5.38; N, 17.27%. Found: C, 64.11; H, 5.27; N, 17.18%. FTIR (KBr cm⁻¹): 1648, for (C≡N) and 3480 (–OH). The tetranuclear nickel complex was synthesized by taking an equimolar methanolic solution (10 ml) of the HL ligand (0.243 g, 1.0 mmol) and nickel perchlorate hexahydrate (0.365 g, 1.0 mmol). The resulting solution was stirred for 3 h.

Figure 5
Crystal packing diagram viewed along a-axis of the complex.

| D—H · · · A  | D−H  | H · · · A  | D−A  | D−··· A  |
|-------------|-------|-----------|-------|----------|
| C7B—H7BB···N1S  | 0.98  | 2.60  | 3.523 (11) | 157    |
| C13C—H13B···O13  | 0.95  | 2.42  | 3.126 (9)  | 131    |
| C13C—H13C···N1S  | 0.95  | 2.59  | 3.348 (11) | 137    |
| N2D—H2DA···O2W  | 0.88  | 1.91  | 2.720 (9)  | 152    |
| C7D—H7DC···O12  | 0.98  | 2.56  | 3.389 (10) | 142    |
| C12S—H12G···O14  | 0.98  | 2.37  | 3.335 (12) | 168    |
| O2W—H2W1···O11  | 0.84 (3) | 2.12 (7) | 2.795 (8) | 138 (9) |
| O2W—H2W2···O14  | 0.83 (3) | 2.78 (3) | 3.599 (6) | 170 (9) |

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

Table 2
Hydrogen-bond geometry (Å).
FTIR (KBr, v, cm⁻¹): 1626 (C≡N), 1537 (C–O), 487 (Ni–O) and 421 (Ni–N).

6. Refinement
Crystallographic data and refinement details are presented in Table 3. H atoms were located in difference-Fourier maps and constrained to ride on their parent atoms with with C–H bond distances of 0.95 Å (aromatic H), 0.98 Å (methyl H) and 0.88 Å (N–H) and were refined as riding with isotropic displacement parameters 1.2 and 1.5 times those of the parent C/N atoms. Water H atoms were refined isotropically with \( U_{eq}(H) = 1.5 U_{eq}(O) \). Three of the four perchlorate anions are disordered over two orientations and were restrained to have tetrahedral geometries with occupancies of 0.57 (6)/0.43 (6), 0.412 (13)/0.488 (13), and 0.806 (12)/0.194 (12), respectively.

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Crystal data

[Ni₄(C₁₃H₁₂N₃O₂)₄](ClO₄)₄·C₂H₃N·2H₂O

M_r = 1678.75
Orthorhombic, Pna2₁
a = 23.5976 (6) Å
b = 11.8723 (3) Å
c = 22.2989 (6) Å
V = 6247.2 (3) Å³
Z = 4

D_x = 1.785 Mg m⁻³
Mo Kα radiation, λ = 0.71073 Å

Cell parameters from 9461 reflections
θ = 2.4–26.3°
µ = 1.46 mm⁻¹
T = 100 K
Needle, green

0.25 × 0.11 × 0.09 mm

14617 independent reflections
11629 reflections with I > 2σ(I)
R_factor = 0.068
θ_max = 28.3°, θ_min = 1.9°
h = −31→31
k = −15→15
l = −26→29

Refinement

Refinement on F²
Least-squares matrix: full
R[F² > 2σ(F²)] = 0.048
wR(F²) = 0.125
S = 1.06
14617 reflections
1051 parameters
320 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
w = 1/[σ(F²)² + (0.0571P)² + 9.1783P]
where P = (F² + 2Fc²)/3
(Δ/σ)max = 0.001
Δρ_max = 0.84 e Å⁻³
Δρ_min = −1.30 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       | Uiso* | Ueq  | Occ. (<1) |
|----|---------|---------|---------|-------|------|-----------|
| Ni1| 0.73671 (3) | 0.62167 (6) | 0.50776 (4) | 0.01973 (18) |
| Ni2| 0.65087 (3) | 0.46794 (6) | 0.42646 (4) | 0.01955 (17) |
| Ni3| 0.58915 (3) | 0.60905 (7) | 0.53570 (4) | 0.02116 (18) |
| Ni4| 0.64341 (3) | 0.75702 (6) | 0.42586 (4) | 0.02228 (18) |
| O1A| 0.6789 (2) | 0.7401 (4) | 0.5065 (2) | 0.0261 (10) |
| O2A| 0.6144 (2) | 0.9102 (4) | 0.4796 (3) | 0.0319 (12) |
| N1A| 0.7519 (2) | 0.6456 (5) | 0.5919 (3) | 0.0282 (13) |
| N2A| 0.7897 (3) | 0.5687 (5) | 0.6153 (3) | 0.0315 (13) |
| H2AA| 0.797624 | 0.565969 | 0.653817 | 0.06 (3)* |
| N3A| 0.7945 (2) | 0.5026 (5) | 0.5191 (3) | 0.0251 (12) |
| C1A| 0.6699 (3) | 0.8175 (6) | 0.5507 (3) | 0.0259 (15) |
| C2A| 0.6349 (3) | 0.9109 (5) | 0.5375 (4) | 0.0284 (15) |
| C3A| 0.6212 (4) | 0.9884 (6) | 0.5812 (4) | 0.0383 (19) |
| H3AA| 0.597016 | 1.049895 | 0.571891 | 0.046* |
| C4A| 0.6423 (4) | 0.9769 (7) | 0.6381 (4) | 0.045 (2) |
| H4AA| 0.632679 | 1.030502 | 0.668040 | 0.054* |
| C5A| 0.6775 (4) | 0.8881 (7) | 0.6523 (4) | 0.0406 (19) |
| H5AA| 0.692022 | 0.881058 | 0.691864 | 0.049* |
| C6A| 0.6920 (3) | 0.8081 (6) | 0.6086 (3) | 0.0302 (16) |
| C7A| 0.5891 (4) | 1.0134 (6) | 0.4589 (4) | 0.044 (2) |
| H7AA| 0.612658 | 1.077293 | 0.471255 | 0.066* |
| H7AB| 0.551164 | 1.021250 | 0.476297 | 0.066* |
| H7AC| 0.586234 | 1.011904 | 0.415072 | 0.066* |
| C8A| 0.7308 (3) | 0.7216 (6) | 0.6278 (3) | 0.0311 (16) |
| H8AA| 0.741612 | 0.719868 | 0.668851 | 0.037* |
| C9A| 0.8139 (3) | 0.4970 (6) | 0.5750 (3) | 0.0265 (15) |
| C10A| 0.8565 (3) | 0.4202 (7) | 0.5921 (4) | 0.0326 (17) |
| H10A| 0.869548 | 0.417446 | 0.632430 | 0.039* |
| C11A| 0.8782 (3) | 0.3517 (7) | 0.5509 (4) | 0.0363 (18) |
| H11A| 0.906120 | 0.298033 | 0.562076 | 0.044* |
| C12A| 0.8601 (3) | 0.3578 (6) | 0.4908 (4) | 0.0342 (17) |
| H12A| 0.876312 | 0.310984 | 0.460768 | 0.041* |
| C13A| 0.8179 (3) | 0.4346 (6) | 0.4770 (4) | 0.0298 (15) |
| H13A| 0.804979 | 0.439766 | 0.436785 | 0.036* |
| O1B| 0.71137 (17) | 0.5813 (3) | 0.4250 (2) | 0.0231 (9) |
|     |     |     |     |     |     |     |
|-----|-----|-----|-----|-----|-----|-----|
| O2B | 0.80171 (19) | 0.7036 (4) | 0.4463 (2) | 0.0256 (10) |     |     |
| N1B | 0.6495 (2) | 0.4613 (5) | 0.3398 (3) | 0.0254 (13) |     |     |
| N2B | 0.6028 (3) | 0.4046 (5) | 0.3185 (3) | 0.0301 (13) |     |     |
| H2BA | 0.594259 | 0.403529 | 0.280135 | 0.07 (4)* |     |     |
| N3B | 0.5861 (2) | 0.3646 (4) | 0.4175 (3) | 0.0252 (12) |     |     |
| C1B | 0.7471 (3) | 0.6010 (5) | 0.3779 (3) | 0.0239 (14) |     |     |
| C2B | 0.7959 (3) | 0.6656 (5) | 0.3881 (3) | 0.0240 (14) |     |     |
| C3B | 0.8342 (3) | 0.6893 (6) | 0.3426 (3) | 0.0272 (15) |     |     |
| H3BA | 0.867365 | 0.732216 | 0.350462 | 0.033* |     |     |
| C4B | 0.8230 (3) | 0.6488 (6) | 0.2849 (3) | 0.0302 (15) |     |     |
| H4AB | 0.849159 | 0.663339 | 0.253437 | 0.036* |     |     |
| C5B | 0.7750 (3) | 0.5886 (6) | 0.2734 (3) | 0.0282 (15) |     |     |
| H5BA | 0.767612 | 0.563818 | 0.233670 | 0.034* |     |     |
| C6B | 0.7361 (3) | 0.5626 (6) | 0.3195 (3) | 0.0252 (14) |     |     |
| C7B | 0.8561 (3) | 0.7453 (7) | 0.4645 (4) | 0.0367 (18) |     |     |
| H7BA | 0.865425 | 0.812932 | 0.441373 | 0.055* |     |     |
| H7BB | 0.884944 | 0.687402 | 0.457407 | 0.055* |     |     |
| H7BC | 0.855123 | 0.763956 | 0.507341 | 0.055* |     |     |
| C8B | 0.6866 (3) | 0.4975 (6) | 0.3018 (3) | 0.0260 (14) |     |     |
| H8BA | 0.681397 | 0.480901 | 0.260520 | 0.031* |     |     |
| C9B | 0.5706 (3) | 0.3504 (6) | 0.3597 (4) | 0.0282 (15) |     |     |
| C10B | 0.5229 (3) | 0.2854 (6) | 0.3442 (4) | 0.0352 (17) |     |     |
| H10B | 0.511292 | 0.278466 | 0.303583 | 0.042* |     |     |
| C11B | 0.4938 (3) | 0.2330 (6) | 0.3886 (4) | 0.0369 (19) |     |     |
| H11B | 0.461388 | 0.188949 | 0.379192 | 0.044* |     |     |
| C12B | 0.5115 (3) | 0.2435 (6) | 0.4488 (4) | 0.0316 (16) |     |     |
| H12B | 0.492177 | 0.204730 | 0.479900 | 0.038* |     |     |
| C13B | 0.5568 (3) | 0.3103 (5) | 0.4614 (4) | 0.0298 (16) |     |     |
| H13B | 0.568312 | 0.319200 | 0.501973 | 0.036* |     |     |
| O1C | 0.64418 (19) | 0.4911 (4) | 0.5149 (2) | 0.0252 (10) |     |     |
| O2C | 0.7018 (2) | 0.3254 (4) | 0.4662 (2) | 0.0303 (11) |     |     |
| N1C | 0.6018 (2) | 0.5844 (5) | 0.6203 (3) | 0.0281 (13) |     |     |
| N2C | 0.5735 (3) | 0.6611 (5) | 0.6559 (3) | 0.0327 (14) |     |     |
| H2CA | 0.575273 | 0.658689 | 0.695341 | 0.14 (6)* |     |     |
| N3C | 0.5404 (2) | 0.7317 (5) | 0.5660 (3) | 0.0270 (12) |     |     |
| C1C | 0.6662 (3) | 0.4124 (6) | 0.5522 (3) | 0.0253 (15) |     |     |
| C2C | 0.6976 (3) | 0.3228 (6) | 0.5273 (4) | 0.0299 (16) |     |     |
| C3C | 0.7222 (3) | 0.2412 (6) | 0.5633 (4) | 0.0367 (18) |     |     |
| H3CA | 0.742965 | 0.181040 | 0.545793 | 0.044* |     |     |
| C4C | 0.7164 (3) | 0.2480 (7) | 0.6254 (4) | 0.0385 (19) |     |     |
| H4CA | 0.733433 | 0.192783 | 0.650417 | 0.046* |     |     |
| C5C | 0.6862 (3) | 0.3341 (7) | 0.6501 (4) | 0.0364 (18) |     |     |
| H5CA | 0.682536 | 0.337817 | 0.692492 | 0.044* |     |     |
| C6C | 0.6602 (3) | 0.4172 (6) | 0.6149 (4) | 0.0290 (15) |     |     |
| C7C | 0.7241 (3) | 0.2280 (6) | 0.4358 (4) | 0.041 (2) |     |     |
| H7CA | 0.764599 | 0.220712 | 0.444534 | 0.062* |     |     |
| H7CB | 0.718647 | 0.236542 | 0.392429 | 0.062* |     |     |
| H7CC | 0.704131 | 0.160439 | 0.449638 | 0.062* |     |     |
| Atom  | X      | Y      | Z      | Uiso  |
|-------|--------|--------|--------|-------|
| C8C   | 0.6291 (3) | 0.5033 (6) | 0.6458 (4) | 0.0329 (17) |
| H8CA  | 0.628574 | 0.500392 | 0.688326 | 0.040* |
| C9C   | 0.5425 (3) | 0.7411 (6) | 0.6262 (4) | 0.0315 (16) |
| C10C  | 0.5134 (3) | 0.8263 (7) | 0.6577 (4) | 0.0387 (18) |
| H10C  | 0.516037 | 0.832498 | 0.700031 | 0.046* |
| C11C  | 0.4814 (4) | 0.8989 (7) | 0.6252 (5) | 0.047 (2) |
| H11C  | 0.461404 | 0.957613 | 0.644892 | 0.056* |
| C12C  | 0.4776 (4) | 0.8880 (7) | 0.5629 (4) | 0.0383 (18) |
| H12C  | 0.454778 | 0.938126 | 0.540173 | 0.046* |
| C13C  | 0.5076 (3) | 0.8034 (6) | 0.5351 (4) | 0.0314 (15) |
| H13C  | 0.534959 | 0.795620 | 0.492831 | 0.038* |
| O1D   | 0.5854 (2) | 0.6484 (4) | 0.4489 (2) | 0.0273 (11) |
| O2D   | 0.5803 (2) | 0.5253 (4) | 0.5041 (3) | 0.0355 (11) |
| N1D   | 0.6176 (3) | 0.7490 (5) | 0.3430 (3) | 0.0319 (14) |
| N2D   | 0.6544 (3) | 0.7994 (6) | 0.3044 (3) | 0.0354 (15) |
| H2DA  | 0.650230 | 0.794969 | 0.265219 | 0.06 (3)* |
| N3D   | 0.7016 (3) | 0.8547 (5) | 0.3897 (3) | 0.0287 (13) |
| C1D   | 0.5384 (3) | 0.6181 (5) | 0.4174 (4) | 0.0278 (15) |
| C2D   | 0.4963 (3) | 0.5530 (5) | 0.4450 (3) | 0.0276 (15) |
| C3D   | 0.4487 (3) | 0.5165 (6) | 0.4145 (4) | 0.0330 (17) |
| H3DA  | 0.420861 | 0.472560 | 0.434478 | 0.040* |
| C4D   | 0.4418 (3) | 0.5441 (7) | 0.3551 (4) | 0.0381 (18) |
| H4DA  | 0.408760 | 0.520128 | 0.334346 | 0.046* |
| C5D   | 0.4820 (3) | 0.6059 (6) | 0.3257 (4) | 0.0365 (18) |
| H5DA  | 0.477119 | 0.623155 | 0.284384 | 0.044* |
| C6D   | 0.5309 (3) | 0.6444 (6) | 0.3562 (4) | 0.0319 (16) |
| C7D   | 0.4621 (4) | 0.4883 (7) | 0.5406 (4) | 0.045 (2) |
| H7DA  | 0.451192 | 0.411766 | 0.528798 | 0.067* |
| H7DB  | 0.429829 | 0.539144 | 0.535135 | 0.067* |
| H7DC  | 0.473691 | 0.488523 | 0.582776 | 0.067* |
| C8D   | 0.5718 (3) | 0.7045 (6) | 0.3210 (4) | 0.0324 (16) |
| H8DA  | 0.564968 | 0.712128 | 0.279187 | 0.039* |
| C9D   | 0.6979 (3) | 0.8568 (6) | 0.3297 (4) | 0.0333 (17) |
| C10D  | 0.7367 (4) | 0.9166 (7) | 0.2944 (4) | 0.0397 (19) |
| H10D  | 0.734936 | 0.914895 | 0.251865 | 0.048* |
| C11D  | 0.7779 (4) | 0.9786 (7) | 0.3244 (5) | 0.046 (2) |
| H11D  | 0.804852 | 1.026885 | 0.302010 | 0.056* |
| C12D  | 0.7801 (3) | 0.9795 (6) | 0.3863 (5) | 0.042 (2) |
| H12D  | 0.807932 | 1.023129 | 0.406559 | 0.050* |
| C13D  | 0.7415 (3) | 0.9164 (6) | 0.4182 (4) | 0.0342 (17) |
| H13D  | 0.742885 | 0.916202 | 0.460822 | 0.041* |
| C14   | 0.52002 (8) | 0.25270 (17) | 0.65520 (9) | 0.0359 (4) |
| O11   | 0.4683 (2) | 0.1969 (6) | 0.6705 (3) | 0.0526 (16) |
| O12   | 0.5136 (3) | 0.3696 (5) | 0.6678 (3) | 0.0580 (18) |
| O13   | 0.5308 (4) | 0.2377 (7) | 0.5934 (3) | 0.071 (2) |
| O14   | 0.5644 (3) | 0.2096 (7) | 0.6908 (3) | 0.072 (2) |
| C12   | 0.64516 (10) | 0.75422 (18) | 0.80261 (10) | 0.0462 (5) |
| O21   | 0.6408 (13) | 0.711 (2) | 0.8612 (9) | 0.044 (6) | 0.57 (6) |
### Atomic displacement parameters (Å²)

|     | $U^{11}$  | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|-----|-----------|-----------|-----------|-----------|-----------|-----------|
| Ni1 | 0.0225 (4) | 0.0185 (4) | 0.0181 (4) | 0.0000 (3) | 0.0010 (3) | −0.0011 (3) |
| Ni2 | 0.0213 (3) | 0.0167 (3) | 0.0207 (4) | −0.0003 (3) | 0.0006 (4) | 0.0004 (4)  |
| Ni3 | 0.0231 (4) | 0.0197 (4) | 0.0206 (4) | 0.0018 (3)  | 0.0008 (3) | 0.0027 (3)  |
| Ni4 | 0.0266 (4) | 0.0175 (4) | 0.0227 (4) | 0.0018 (3)  | 0.0022 (4) | 0.0019 (4)  |
| O1A | 0.031 (2)  | 0.018 (2)  | 0.029 (3)  | 0.0000 (18) | 0.005 (2) | −0.003 (2)  |
|          | O2A       | N1A       | N2A       | N3A       | C1A       | C2A       | C3A       | C4A       | C5A       | C6A       | C7A       | C8A       | C9A       | C10A      | C11A      | C12A      | C13A      | O1B       | O2B       | N1B       | N2B       | N3B       | C1B       | C2B       | C3B       | C4B       | C5B       | C6B       | C7B       | C8B       | C9B       | C10B      | C11B      | C12B      | C13B      | O1C       | O2C       | N1C       | N2C       | N3C       | C1C       | C2C       | C3C       | C4C       | C5C       | C6C       | C7C       | C8C       |
|----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|-----------|
| Atom | U11  | U22  | U33  | U12  | U13  | U23  |
|------|------|------|------|------|------|------|
| C9C  | 0.028 (3) | 0.031 (4) | 0.035 (5) | −0.004 (3) | 0.005 (3) | 0.004 (3) |
| C10C | 0.044 (4)  | 0.042 (4)  | 0.031 (4)  | 0.010 (4)  | 0.016 (4)  | 0.000 (3)  |
| C11C | 0.039 (4)  | 0.047 (5)  | 0.053 (6)  | 0.012 (4)  | 0.015 (4)  | −0.004 (4) |
| C12C | 0.037 (4)  | 0.035 (4)  | 0.043 (5)  | 0.007 (3)  | 0.012 (4)  | 0.003 (4)  |
| C13C | 0.033 (4)  | 0.030 (4)  | 0.032 (4)  | 0.005 (3)  | 0.004 (3)  | 0.006 (3)  |
| O1D  | 0.029 (2)  | 0.025 (2)  | 0.028 (3)  | 0.001 (2)  | −0.005 (2) | 0.002 (2)  |
| O2D  | 0.030 (2)  | 0.032 (3)  | 0.038 (3)  | −0.002 (2) | −0.002 (2) | 0.006 (2)  |
| N1D  | 0.038 (3)  | 0.028 (3)  | 0.030 (4)  | 0.004 (3)  | 0.006 (3)  | 0.006 (3)  |
| N2D  | 0.037 (4)  | 0.043 (4)  | 0.027 (4)  | 0.000 (3)  | 0.003 (3)  | 0.009 (3)  |
| N3D  | 0.039 (3)  | 0.018 (3)  | 0.030 (3)  | 0.004 (2)  | 0.004 (3)  | 0.001 (2)  |
| C11D | 0.049 (5)  | 0.032 (4)  | 0.058 (6)  | −0.001 (3) | 0.007 (4)  | −0.007 (4) |
| C12D | 0.037 (4)  | 0.024 (4)  | 0.066 (6)  | −0.001 (3) | 0.007 (4)  | −0.007 (4) |
| C13D | 0.033 (4)  | 0.022 (3)  | 0.047 (5)  | 0.001 (3)  | 0.005 (4)  | −0.004 (3) |
| C11  | 0.0321 (9) | 0.0463 (10) | 0.0294 (10) | 0.0015 (8) | 0.0001 (7) | 0.0039 (8) |
| O11  | 0.037 (3)  | 0.063 (4)  | 0.058 (4)  | −0.010 (3) | −0.002 (3) | 0.007 (3)  |
| O12  | 0.080 (5)  | 0.043 (3)  | 0.051 (4)  | −0.009 (3) | −0.002 (4) | 0.002 (3)  |
| O13  | 0.091 (5)  | 0.081 (5)  | 0.040 (4)  | 0.010 (4)  | 0.022 (4)  | −0.001 (4) |
| O14  | 0.039 (4)  | 0.099 (6)  | 0.076 (5)  | 0.015 (4)  | −0.010 (3) | 0.025 (4)  |
| O21  | 0.0553 (12) | 0.0398 (11) | 0.0434 (13) | 0.0063 (9) | 0.0047 (10) | 0.0041 (9) |
| O22  | 0.114 (18) | 0.054 (10)  | 0.049 (11)  | 0.039 (11)  | −0.045 (11) | −0.015 (8) |
| O23  | 0.064 (10) | 0.044 (9)   | 0.059 (11)  | 0.026 (7)   | 0.025 (8)   | 0.018 (7)  |
| O24  | 0.066 (11) | 0.115 (17)  | 0.110 (18)  | −0.021 (11) | 0.008 (10)  | 0.031 (14) |
| O21A | 0.057 (15) | 0.053 (16)  | 0.049 (16)  | −0.011 (11) | −0.001 (12) | 0.012 (13) |
| O22A | 0.088 (18) | 0.038 (10)  | 0.032 (10)  | 0.018 (10)  | 0.000 (11)  | 0.001 (7)  |
| O23A | 0.064 (13) | 0.12 (2)    | 0.062 (15)  | 0.035 (15)  | −0.011 (11) | 0.040 (16) |
| O24A | 0.15 (2)   | 0.074 (16)  | 0.056 (14)  | −0.063 (16) | −0.006 (15) | −0.003 (11) |
| C11  | 0.076 (2)  | 0.126 (3)   | 0.067 (2)   | −0.032 (2)  | −0.0087 (16) | 0.013 (2)  |
| O31  | 0.114 (8)  | 0.178 (10)  | 0.139 (9)   | −0.060 (8)  | 0.030 (7)   | −0.013 (8) |
| O32  | 0.078 (7)  | 0.074 (7)   | 0.040 (6)   | −0.002 (6)  | −0.023 (6)  | −0.005 (6) |
| O33  | 0.066 (7)  | 0.085 (8)   | 0.073 (8)   | 0.021 (7)   | −0.024 (7)  | −0.008 (7) |
| O34  | 0.061 (9)  | 0.085 (9)   | 0.062 (8)   | −0.005 (8)  | −0.043 (7)  | −0.002 (7) |
| O32A | 0.056 (7)  | 0.076 (8)   | 0.040 (7)   | −0.010 (6)  | −0.003 (6)  | 0.017 (6)  |
| O33A | 0.097 (8)  | 0.086 (8)   | 0.055 (7)   | −0.004 (7)  | −0.051 (6)  | 0.001 (6)  |
| O34A | 0.079 (10) | 0.080 (8)   | 0.045 (7)   | −0.005 (8)  | −0.028 (7)  | 0.007 (6)  |
| C14  | 0.0608 (15)| 0.081 (2)   | 0.074 (2)   | 0.0103 (14) | 0.0146 (14) | 0.0152 (16) |
| O41  | 0.096 (8)  | 0.088 (6)   | 0.046 (6)   | −0.016 (6)  | −0.020 (5)  | 0.040 (5)  |
| O42  | 0.087 (7)  | 0.044 (5)   | 0.060 (6)   | −0.010 (5)  | 0.012 (5)   | 0.009 (4)  |
Geometric parameters (Å, °)

| Bond                  | Distance (Å) | Angle (°) |
|-----------------------|--------------|-----------|
| Ni1—N1A               | 1.932 (7)    | 1.406 (11)|
| Ni1—O1A               | 1.960 (5)    | 1.410 (10)|
| Ni1—N3A               | 1.980 (6)    | 1.386 (10)|
| Ni1—O1B               | 1.998 (5)    | 1.393 (12)|
| Ni1—O2B               | 2.276 (4)    | 0.9500    |
| Ni2—N1B               | 1.935 (6)    | 1.363 (12)|
| Ni2—O1B               | 1.962 (4)    | 0.9500    |
| Ni2—N3B               | 1.971 (6)    | 1.401 (10)|
| Ni2—O1C               | 1.997 (5)    | 0.9500    |
| Ni2—O2C               | 2.257 (5)    | 1.434 (11)|
| Ni3—N1C               | 1.932 (7)    | 0.9800    |
| Ni3—O1C               | 1.965 (5)    | 0.9800    |
| Ni3—N3C               | 1.975 (6)    | 0.9800    |
| Ni3—O1D               | 1.993 (5)    | 0.9500    |
| Ni3—O2D               | 2.264 (5)    | 1.409 (11)|
| Ni4—N1D               | 1.948 (7)    | 1.355 (13)|
| Ni4—O1D               | 1.950 (5)    | 0.9500    |
| Ni4—N3D               | 1.969 (6)    | 1.397 (13)|
| Ni4—O1A               | 1.993 (5)    | 0.9500    |
| Ni4—O2A               | 2.283 (5)    | 1.375 (10)|
| O1A—C1A               | 1.365 (8)    | 0.9500    |
| O2A—C2A               | 1.378 (10)   | 0.9500    |
| O2A—C7A               | 1.439 (8)    | 1.360 (9) |
| N1A—C8A               | 1.305 (9)    | 1.387 (9) |
| N1A—N2A               | 1.377 (9)    | 1.429 (9) |
| N2A—C9A               | 1.364 (10)   | 1.300 (10)|
| N2A—H2AA              | 0.8800       | 1.362 (9) |
| N3A—C9A               | 1.330 (9)    | 1.355 (11)|
| N3A—C13A              | 1.356 (9)    | 0.8800    |
| C1A—C6A               | 1.396 (11)   | 1.341 (10)|
| C1A—C2A               | 1.414 (10)   | 1.351 (10)|
| C2A—C3A               | 1.379 (10)   | 1.403 (10)|
| C3A—C4A               | 1.370 (13)   | 1.410 (11)|
| Bond                  | Length  | Bond                  | Length  | Bond                  | Length  |
|----------------------|---------|----------------------|---------|----------------------|---------|
| C3A—H3AA             | 0.9500  | C2D—C3D              | 1.383 (10) | C2D—C3D              | 1.383 (10) |
| C4A—C5A              | 1.378 (13) | C3D—C4D              | 1.373 (12) | C3D—C4D              | 1.373 (12) |
| C4A—H4AA             | 0.9500  | C4D—C5D              | 1.368 (12) | C4D—C5D              | 1.368 (12) |
| C5A—C6A              | 1.403 (10) | C4D—H4DA             | 0.9500  | C4D—H4DA             | 0.9500  |
| C5A—H5AA             | 0.9500  | C5D—C6D              | 1.416 (11) | C5D—C6D              | 1.416 (11) |
| C6A—C8A              | 1.441 (11) | C5D—H5DA             | 0.9500  | C5D—H5DA             | 0.9500  |
| C7A—H7AA             | 0.9800  | C6D—C8D              | 1.433 (11) | C6D—C8D              | 1.433 (11) |
| C7A—H7AB             | 0.9800  | C7D—H7DA             | 0.9800  | C7D—H7DA             | 0.9800  |
| C7A—H7AC             | 0.9800  | C7D—H7DB             | 0.9800  | C7D—H7DB             | 0.9800  |
| C8A—H8AA             | 0.9500  | C8D—H8DA             | 0.9500  | C8D—H8DA             | 0.9500  |
| C9A—C10A             | 1.411 (10) | C8D—H8DA             | 0.9500  | C8D—H8DA             | 0.9500  |
| C10A—C11A            | 1.330 (12) | C9D—C10D             | 1.401 (11) | C9D—C10D             | 1.401 (11) |
| C10A—H10A            | 0.9500  | C10D—C11D            | 1.390 (13) | C10D—C11D            | 1.390 (13) |
| C11A—C12A            | 1.408 (12) | C10D—H10D            | 0.9500  | C10D—H10D            | 0.9500  |
| C11A—H11A            | 0.9500  | C11D—C12D            | 1.381 (14) | C11D—C12D            | 1.381 (14) |
| C12A—C13A            | 1.384 (10) | C11D—H11D            | 0.9500  | C11D—H11D            | 0.9500  |
| C12A—H12A            | 0.9500  | C12D—C13D            | 1.380 (11) | C12D—C13D            | 1.380 (11) |
| C13A—H13A            | 0.9500  | C12D—H12D            | 0.9500  | C12D—H12D            | 0.9500  |
| O1B—C1B              | 1.367 (8)  | C13D—H13D            | 0.9500  | C13D—H13D            | 0.9500  |
| O2B—C2B              | 1.380 (8)  | CI1—O14              | 1.409 (6)  | CI1—O14              | 1.409 (6)  |
| N1B—C8B              | 1.292 (9)  | CI1—O13              | 1.413 (7)  | CI1—O13              | 1.413 (7)  |
| N1B—N2B              | 1.377 (8)  | CI1—O12              | 1.424 (6)  | CI1—O12              | 1.424 (6)  |
| N2B—C9B              | 1.353 (10) | CI1—O11              | 1.429 (6)  | CI1—O11              | 1.429 (6)  |
| N2B—H2BA             | 0.8800  | CI2—O24A             | 1.378 (18) | CI2—O24A             | 1.378 (18) |
| N3B—C9B              | 1.351 (10) | CI2—O21A             | 1.397 (18) | CI2—O21A             | 1.397 (18) |
| N3B—C13B             | 1.360 (9)  | CI2—O23              | 1.399 (14) | CI2—O23              | 1.399 (14) |
| C1B—C2B              | 1.402 (9)  | CI2—O21              | 1.405 (15) | CI2—O21              | 1.405 (15) |
| C1B—C6B              | 1.404 (10) | CI2—O22              | 1.430 (14) | CI2—O22              | 1.430 (14) |
| C2B—C3B              | 1.387 (10) | CI2—O22A             | 1.437 (17) | CI2—O22A             | 1.437 (17) |
| C3B—C4B              | 1.398 (10) | CI2—O24              | 1.438 (14) | CI2—O24              | 1.438 (14) |
| C3B—H3BA             | 0.9500  | CI2—O23A             | 1.449 (17) | CI2—O23A             | 1.449 (17) |
| C4B—C5B              | 1.364 (10) | CI3—O33A             | 1.248 (12) | CI3—O33A             | 1.248 (12) |
| C4B—H4BA             | 0.9500  | CI3—O34A             | 1.326 (15) | CI3—O34A             | 1.326 (15) |
| C5B—C6B              | 1.414 (10) | CI3—O32              | 1.336 (12) | CI3—O32              | 1.336 (12) |
| C5B—H5BA             | 0.9500  | CI3—O34              | 1.377 (15) | CI3—O34              | 1.377 (15) |
| C6B—C8B              | 1.453 (10) | CI3—O31              | 1.448 (9)  | CI3—O31              | 1.448 (9)  |
| C7B—H7BA             | 0.9800  | CI3—O33              | 1.517 (12) | CI3—O33              | 1.517 (12) |
| C7B—H7BB             | 0.9800  | CI3—O32A             | 1.677 (14) | CI3—O32A             | 1.677 (14) |
| C7B—H7BC             | 0.9800  | CI4—O41              | 1.363 (9)  | CI4—O41              | 1.363 (9)  |
| C8B—H8BA             | 0.9500  | CI4—O44              | 1.368 (9)  | CI4—O44              | 1.368 (9)  |
| C9B—C10B             | 1.408 (10) | CI4—O44A             | 1.38 (2)   | CI4—O44A             | 1.38 (2)   |
| C10B—C11B            | 1.356 (12) | CI4—O41A             | 1.40 (2)   | CI4—O41A             | 1.40 (2)   |
| C10B—H10B            | 0.9500  | CI4—O43A             | 1.40 (2)   | CI4—O43A             | 1.40 (2)   |
| C11B—C12B            | 1.411 (12) | CI4—O42              | 1.420 (9)  | CI4—O42              | 1.420 (9)  |
| C11B—H11B            | 0.9500  | CI4—O43              | 1.457 (10) | CI4—O43              | 1.457 (10) |
| C12B—C13B            | 1.360 (10) | CI4—O42A             | 1.47 (2)   | CI4—O42A             | 1.47 (2)   |
| C12B—H12B            | 0.9500  | N1S—C11S             | 1.117 (13) | N1S—C11S             | 1.117 (13) |
C13B—H13B 0.9500 C11S—C12S 1.462 (15)
O1C—C1C 1.355 (8) C12S—H12E 0.9800
O2C—C2C 1.368 (9) C12S—H12F 0.9800
O2C—C7C 1.439 (9) C12S—H12G 0.9800
N1C—C8C 1.290 (9) O1W—H1W1 0.80 (3)
N1C—N2C 1.381 (9) O1W—H1W2 0.82 (3)
N2C—C9C 1.371 (10) O1WA—H1W3 0.82 (3)
N2C—H2CA 0.8800 O1WA—H1W4 0.82 (3)
N3C—C13C 1.341 (9) O2W—H2W1 0.84 (3)
N3C—C9C 1.347 (10) O2W—H2W2 0.83 (3)

N1A—Ni1—O1A 92.2 (2) C8C—N1C—N2C 118.7 (7)
N1A—Ni1—N3A 81.6 (2) C8C—N1C—Ni3 128.3 (6)
O1A—Ni1—N3A 173.5 (2) N2C—N1C—Ni3 112.8 (5)
N1A—Ni1—O1B 171.2 (2) C9C—N2C—Ni1C 115.9 (7)
O1A—Ni1—O1B 87.1 (2) C9C—N2C—H12CA 122.1
N3A—Ni1—O1B 98.8 (2) N1C—N2C—H12CA 122.1
N1A—Ni1—O2B 113.4 (2) C13C—N3C—C9C 118.6 (6)
O1A—Ni1—O2B 98.83 (19) C13C—N3C—Ni3 129.0 (5)
N3A—Ni1—O2B 85.3 (2) C9C—N3C—Ni3 112.4 (5)
O1B—Ni1—O2B 75.39 (17) O1C—C1C—C6C 123.0 (6)
N1B—Ni2—O1B 91.3 (2) O1C—C1C—C2C 118.7 (7)
N1B—Ni2—N3B 82.0 (2) C6C—C1C—C2C 118.3 (6)
O1B—Ni2—N3B 171.8 (2) O2C—C2C—C3C 124.2 (7)
N1B—Ni2—O1C 172.1 (2) O2C—C2C—C1C 114.4 (6)
O1B—Ni2—O1C 88.8 (2) C3C—C2C—C1C 121.3 (8)
N3B—Ni2—O1C 97.1 (2) C2C—C3C—C4C 119.6 (7)
N1B—Ni2—O2C 111.7 (2) C2C—C3C—H3CA 120.2
O1B—Ni2—O2C 97.69 (18) C4C—C3C—H3CA 120.2
N3B—Ni2—O2C 89.2 (2) C5C—C4C—C3C 119.8 (7)
O1C—Ni3—O1C 76.02 (19) C5C—C4C—H4CA 120.1
N1C—Ni3—O1C 91.2 (2) C3C—C4C—H4CA 120.1
N1C—Ni3—N3C 82.4 (3) C4C—C5C—C6C 122.0 (8)
O1C—Ni3—N3C 172.3 (2) C4C—C5C—H5CA 119.0
N1C—Ni3—O1D 172.1 (2) C6C—C5C—H5CA 119.0
O1C—Ni3—O1D 88.1 (2) C5C—C6C—C1C 119.0 (7)
N3C—Ni3—O1D 97.7 (2) C5C—C6C—C8C 117.2 (8)
N1C—Ni3—O2D 111.5 (2) C1C—C6C—C8C 123.8 (7)
O1C—Ni3—O2D 99.81 (19) O2C—C7C—H7CA 109.5
N3C—Ni3—O2D 86.5 (2) O2C—C7C—H7CB 109.5
O1D—Ni3—O2D 76.3 (2) H7CA—C7C—H7CB 109.5
N1D—Ni4—O1D 89.9 (2) O2C—C7C—H7CC 109.5
N1D—Ni4—N3D 81.9 (3) H7CA—C7C—H7CC 109.5
O1D—Ni4—N3D 170.6 (2) H7CB—C7C—H7CC 109.5
N1D—Ni4—O1A 169.1 (2) N1C—C8C—C6C 125.2 (7)
O1D—Ni4—O1A 89.5 (2) N1C—C8C—H8CA 117.4
N3D—Ni4—O1A 97.8 (2) C6C—C8C—H8CA 117.4
N1D—Ni4—O2A 116.3 (2) N3C—C9C—N2C 116.4 (7)
| Bond                  | Angle (°) (E) | Bond                  | Angle (°) (E) |
|-----------------------|--------------|-----------------------|--------------|
| O1D—Ni4—O2A          | 100.3 (2)    | N3C—C9C—C10C         | 122.5 (7)    |
| N3D—Ni4—O2A          | 87.4 (2)     | N2C—C9C—C10C         | 121.1 (7)    |
| O1A—Ni4—O2A          | 74.5 (2)     | C11C—C10C—C9C        | 117.5 (8)    |
| C1A—O1A—Ni1          | 125.5 (4)    | C11C—C10C—H10C       | 121.3        |
| C1A—O1A—Ni4          | 121.3 (4)    | C9C—C10C—H10C        | 121.3        |
| Ni1—O1A—Ni4          | 112.2 (2)    | C10C—C11C—C12C       | 120.5 (8)    |
| C2A—O2A—C7A          | 116.2 (6)    | C10C—C11C—H11C       | 119.7        |
| C2A—O2A—Ni4          | 113.0 (4)    | C12C—C11C—H11C       | 119.7        |
| C7A—O2A—Ni4          | 129.4 (5)    | C13C—C12C—C11C       | 118.8 (8)    |
| C8A—N1A—N2A          | 118.3 (7)    | C13C—C12C—H12C       | 120.6        |
| C8A—N1A—Ni1          | 128.8 (5)    | C11C—C12C—H12C       | 120.6        |
| N2A—N1A—Ni1          | 112.9 (5)    | N3C—C13C—C12C        | 122.0 (8)    |
| C9A—N2A—N1A          | 115.8 (6)    | N3C—C13C—H13C        | 119.0        |
| C9A—N2A—H2AA         | 122.1        | C12C—C13C—H13C       | 119.0        |
| N1A—N2A—H2AA         | 122.1        | C1D—O1D—Ni4          | 127.6 (5)    |
| C9A—N3A—C13A         | 118.5 (6)    | C1D—O1D—Ni3          | 118.5 (4)    |
| C9A—N3A—Ni1          | 113.1 (5)    | Ni4—O1D—Ni3          | 112.3 (2)    |
| C13A—N3A—Ni1         | 128.2 (5)    | C2D—O2D—Ni3          | 117.2 (6)    |
| O1A—C1A—C6A          | 123.8 (6)    | C2D—O2D—C7D          | 111.3 (4)    |
| O1A—C1A—C2A          | 117.9 (7)    | C7D—O2D—Ni3          | 126.9 (5)    |
| C6A—C1A—C2A          | 118.3 (6)    | C8D—N1D—N2D          | 118.1 (7)    |
| O2A—C2A—C3A          | 125.7 (7)    | C8D—N1D—Ni4          | 129.7 (5)    |
| O2A—C2A—C1A          | 113.3 (6)    | N2D—N1D—Ni4          | 112.2 (5)    |
| C3A—C2A—C1A          | 120.8 (8)    | C9D—N2D—N1D          | 116.1 (7)    |
| C4A—C3A—C2A          | 120.2 (8)    | C9D—N2D—H2DA         | 121.9        |
| C4A—C3A—H3AA         | 119.9        | N1D—N2D—H2DA         | 121.9        |
| C2A—C3A—H3AA         | 119.9        | C9D—N3D—C13D         | 120.3 (7)    |
| C3A—C4A—C5A          | 120.5 (8)    | C9D—N3D—Ni4          | 112.0 (5)    |
| C3A—C4A—H4AA         | 119.7        | C13D—N3D—Ni4         | 127.7 (6)    |
| C5A—C4A—H4AA         | 119.7        | O1D—C1D—C2D          | 119.7 (7)    |
| C4A—C5A—C6A          | 120.3 (9)    | O1D—C1D—C6D          | 122.9 (6)    |
| C4A—C5A—H5AA         | 119.8        | C2D—C1D—C6D          | 117.2 (7)    |
| C6A—C5A—H5AA         | 119.8        | C3D—C2D—O2D          | 124.0 (6)    |
| C1A—C6A—C5A          | 119.8 (7)    | C3D—C2D—C1D          | 122.2 (7)    |
| C1A—C6A—C8A          | 124.7 (6)    | O2D—C2D—C1D          | 113.8 (6)    |
| C5A—C6A—C8A          | 115.5 (7)    | C4D—C3D—C2D          | 119.8 (7)    |
| O2A—C7A—H7AA         | 109.5        | C4D—C3D—H3DA         | 120.1        |
| O2A—C7A—H7AB         | 109.5        | C2D—C3D—H3DA         | 120.1        |
| H7AA—C7A—H7AB        | 109.5        | C5D—C4D—C3D          | 120.5 (7)    |
| O2A—C7A—H7AC         | 109.5        | C5D—C4D—H4DA         | 119.7        |
| H7AA—C7A—H7AC        | 109.5        | C3D—C4D—H4DA         | 119.7        |
| H7AB—C7A—H7AC        | 109.5        | C4D—C5D—C6D          | 120.5 (8)    |
| N1A—C8A—C6A          | 123.6 (7)    | C4D—C5D—H5DA         | 119.7        |
| N1A—C8A—H8AA         | 118.2        | C6D—C5D—H5DA         | 119.7        |
| C6A—C8A—H8AA         | 118.2        | C1D—C6D—C5D          | 119.8 (7)    |
| N3A—C9A—N2A          | 116.2 (6)    | C1D—C6D—C8D          | 123.8 (7)    |
| N3A—C9A—C10A         | 122.2 (7)    | C5D—C6D—C8D          | 116.4 (7)    |
| N2A—C9A—C10A         | 121.6 (7)    | O2D—C7D—H7DA         | 109.5        |
| Bond                        | Bond Angle (°) | Bond Angle (°) |
|-----------------------------|----------------|----------------|
| C11A—C10A—C9A              | 118.8 (8)      | O2D—C7D—H7DB  | 109.5 |
| C11A—C10A—H10A             | 120.6          | H7DA—C7D—H7DB | 109.5 |
| C9A—C10A—H10A              | 120.6          | O2D—C7D—H7DC  | 109.5 |
| C10A—C11A—C12A             | 120.7 (7)      | H7DA—C7D—H7DC | 109.5 |
| C10A—C11A—H11A             | 119.6          | H7DB—C7D—H7DC | 109.5 |
| C12A—C11A—H11A             | 119.6          | N1D—C8D—C6D   | 123.7 (7) |
| C13A—C12A—C11A             | 117.5 (7)      | N1D—C8D—H8DA  | 118.2 |
| C13A—C12A—H12A             | 121.2          | C6D—C8D—H8DA  | 118.2 |
| C11A—C12A—H12A             | 121.2          | N3D—C9D—N2D   | 117.1 (7) |
| N3A—C13A—C12A              | 122.2 (7)      | N3D—C9D—C10D  | 121.9 (8) |
| N3A—C13A—H13A              | 118.9          | N2D—C9D—C10D  | 121.0 (8) |
| C12A—C13A—H13A             | 118.9          | C11D—C10D—C9D | 117.1 (8) |
| C1B—O1B—Ni2                | 125.4 (4)      | C11D—C10D—H10D| 121.5 |
| C1B—O1B—Ni1                | 118.9 (4)      | C9D—C10D—H10D | 121.5 |
| Ni2—O1B—Ni1                | 111.5 (2)      | C12D—C11D—C10D| 120.7 (8) |
| C2B—O2B—C7B                | 117.9 (5)      | C12D—C11D—H11D| 119.6 |
| C2B—O2B—Ni1                | 111.1 (4)      | C10D—C11D—H11D| 119.6 |
| C7B—O2B—Ni1                | 125.4 (4)      | C13D—C12D—C11D| 119.1 (8) |
| C8B—N1B—N2B                | 118.7 (6)      | C13D—C12D—H12D| 120.4 |
| C8B—N1B—Ni2                | 129.1 (5)      | C11D—C12D—H12D| 120.4 |
| N2B—N1B—Ni2                | 121.2 (4)      | N3D—C13D—C12D | 120.8 (9) |
| C9B—N2B—N1B                | 116.7 (6)      | N3D—C13D—H13D | 119.6 |
| C9B—N2B—H2BA               | 121.7          | C12D—C13D—H13D| 119.6 |
| N1B—N2B—H2BA               | 121.7          | O14—Cl1—O13   | 111.7 (5) |
| C9B—N3B—C13B               | 119.4 (6)      | O14—Cl1—O12   | 108.8 (5) |
| C9B—N3B—Ni2                | 112.6 (5)      | O13—Cl1—O12   | 109.5 (4) |
| C13B—N3B—Ni2               | 128.0 (5)      | O14—Cl1—O11   | 109.3 (4) |
| O1B—C1B—C2B                | 118.5 (6)      | O13—Cl1—O11   | 109.1 (5) |
| O1B—C1B—C6B                | 122.7 (6)      | O12—Cl1—O11   | 108.3 (4) |
| C2B—C1B—C6B                | 118.8 (6)      | O24A—Cl2—O21A | 112.5 (17) |
| O2B—C2B—C3B                | 123.8 (6)      | O23—Cl2—O21   | 111.8 (11) |
| O2B—C2B—C1B                | 114.3 (6)      | O23—Cl2—O22   | 107.8 (11) |
| C3B—C2B—C1B                | 121.8 (7)      | O21—Cl2—O22   | 108.6 (13) |
| C2B—C3B—C4B                | 118.7 (7)      | O24A—Cl2—O22A | 111.1 (14) |
| C2B—C3B—H3BA               | 120.6          | O21A—Cl2—O22A | 108.6 (16) |
| C4B—C3B—H3BA               | 120.6          | O23—Cl2—O24   | 112.1 (12) |
| C5B—C4B—C3B                | 120.7 (7)      | O21—Cl2—O24   | 106.6 (13) |
| C5B—C4B—H4BA               | 119.7          | O22—Cl2—O24   | 109.9 (12) |
| C3B—C4B—H4BA               | 119.7          | O24A—Cl2—O23A | 111.2 (15) |
| C4B—C5B—C6B                | 121.2 (7)      | O21A—Cl2—O23A | 109.4 (17) |
| C4B—C5B—H5BA               | 119.4          | O22A—Cl2—O23A | 103.7 (15) |
| C6B—C5B—H5BA               | 119.4          | O33A—Cl3—O34A | 131.5 (15) |
| C1B—C6B—C5B                | 118.8 (6)      | O32—Cl3—O34   | 117.6 (13) |
| C1B—C6B—C8B                | 125.1 (6)      | O33A—Cl3—O31  | 109.2 (10) |
| C5B—C6B—C8B                | 116.1 (6)      | O34A—Cl3—O31  | 110.4 (13) |
| O2B—C7B—H7BA               | 109.5          | O32—Cl3—O31   | 111.5 (9) |
| O2B—C7B—H7BB               | 109.5          | O34—Cl3—O31   | 103.8 (12) |
| H7BA—C7B—H7BB              | 109.5          | O32—Cl3—O33   | 108.8 (9) |
| Bond          | Angle (°) | Bond          | Angle (°) | Bond          | Angle (°) |
|---------------|-----------|---------------|-----------|---------------|-----------|
| O2B—C7B—H7BC | 109.5     | O34—Cl3—O33  | 109.1     |
| H7BA—C7B—H7BC| 109.5     | O31—Cl3—O33  | 105.3     |
| H7BB—C7B—H7BC| 109.5     | O33A—Cl3—O32A| 100.3     |
| N1B—C8B—C6B  | 122.9     | O34A—Cl3—O32A| 102.7     |
| N1B—C8B—H8BA | 118.6     | O31—Cl3—O32A | 95.4      |
| C6B—C8B—H8BA | 118.6     | O41—Cl4—O44  | 120.0     |
| N3B—C9B—N2B  | 115.9     | O44A—Cl4—O41A| 114       |
| N3B—C9B—C10B | 121.2     | O44A—Cl4—O43A| 111.8     |
| N2B—C9B—C10B | 122.9     | O41A—Cl4—O43A| 111       |
| C11B—C10B—C9B| 118.5     | O41—Cl4—O42  | 108.1     |
| C11B—C10B—H10B| 120.7   | O44—Cl4—O42  | 109.7     |
| C9B—C10B—H10B| 120.7     | O41—Cl4—O43  | 101.2     |
| C10B—C11B—C12B| 120.2   | O44—Cl4—O43  | 104.3     |
| C10B—C11B—H11B| 119.9    | O42—Cl4—O43  | 113.3     |
| C12B—C11B—H11B| 119.9    | O44A—Cl4—O42A| 110.2     |
| C13B—C12B—C11B| 118.8    | O41A—Cl4—O42A| 104.0     |
| C13B—C12B—H12B| 120.6    | O43A—Cl4—O42A| 105.6     |
| C11B—C12B—H12B| 120.6    | N1S—Cl1S—Cl12S| 177.7   |
| C12B—C13B—N3B | 121.7   | C11S—Cl12S—H12E| 109.5  |
| C12B—C13B—H13B| 119.1    | C11S—Cl12S—H12F| 109.5   |
| N3B—C13B—H13B| 119.1    | H12E—Cl12S—H12F| 109.5   |
| C1C—O1C—Ni3  | 126.9    | C11S—Cl12S—H12G| 109.5  |
| C1C—O1C—Ni2  | 118.8    | H12E—Cl12S—H12G| 109.5   |
| Ni3—O1C—Ni2  | 112.6    | H12F—Cl12S—H12G| 109.5   |
| C2C—O2C—C7C  | 118.5    | H1W1—O1W—H1W2 | 107 (5)  |
| C2C—O2C—Ni2  | 111.7    | H1W3—O1WA—H1W4 | 108 (5)  |
| C7C—O2C—Ni2  | 127.8    | H2W1—O2W—H2W2 | 102 (4)  |

| Bond          | Angle (°) | Bond          | Angle (°) | Bond          | Angle (°) |
|---------------|-----------|---------------|-----------|---------------|-----------|
| C8A—N1A—N2A—C9A| −174.9    | C8C—N1C—N2C—C9C| −176.2   |
| Ni1—N1A—N2A—C9A| 6.2       | Ni3—N1C—N2C—C9C| −0.8     |
| Ni1—O1A—C1A—C6A| −14.8     | Ni3—O1C—C1C—C6C| −12.1    |
| Ni4—O1A—C1A—C6A| 177.7     | Ni2—O1C—C1C—C6C| −175.5   |
| Ni1—O1A—C1A—C2A| 167.0     | Ni3—O1C—C1C—C2C| 169.2    |
| Ni4—O1A—C1A—C2A| −0.5      | Ni2—O1C—C1C—C2C| 5.8      |
| C7A—O2A—C2A—C3A| 17.2      | C7C—O2C—C2C—C3C| 11.8     |
| Ni4—O2A—C2A—C3A| −175.2    | Ni2—O2C—C2C—C3C| 176.9    |
| C7A—O2A—C2A—C1A| −166.9    | C7C—O2C—C2C—C1C| −169.2   |
| Ni4—O2A—C2A—C1A| 0.8       | Ni2—O2C—C2C—C1C| −4.1     |
| O1A—C1A—C2A—O2A| −0.2      | O1C—C1C—C2C—O2C| −0.5     |
| C6A—C1A—C2A—O2A| −178.6    | C6C—C1C—C2C—O2C| −179.3   |
| O1A—C1A—C2A—C3A| 175.9     | O1C—C1C—C2C—C3C| 178.5    |
| C6A—C1A—C2A—C3A| −2.4      | C6C—C1C—C2C—C3C| −0.3     |
| O2A—C2A—C3A—C4A| 176.9     | O2C—C2C—C3C—C4C| 178.5    |
| C1A—C2A—C3A—C4A| 1.2       | C1C—C2C—C3C—C4C| −0.5     |
| C2A—C3A—C4A—C5A| 0.1       | C2C—C3C—C4C—C5C| 0.6      |
| C3A—C4A—C5A—C6A| −0.2      | C3C—C4C—C5C—C6C| 0.1      |
| O1A—C1A—C6A—C5A| −175.9    | C4C—C5C—C6C—C1C| −0.8     |
| C2A—C1A—C6A—C5A| 2.3       | C4C—C5C—C6C—C8C| 179.5    |
| Bond                        | Value (deg) | Bond                        | Value (deg)  |
|-----------------------------|-------------|-----------------------------|--------------|
| O1A—C1A—C6A—C8A            | 5.5 (11)    | O1C—C1C—C6C—C5C            | -177.8 (6)   |
| C2A—C1A—C6A—C8A           | -176.2 (6)  | C2C—C1C—C6C—C5C            | 0.9 (10)     |
| C4A—C5A—C6A—C1A           | -1.1 (11)   | O1C—C1C—C6C—C8C            | 1.8 (10)     |
| C4A—C5A—C6A—C8A           | 177.6 (7)   | C2C—C1C—C6C—C8C            | -179.5 (6)   |
| N2A—N1A—C8A—C6A           | 178.6 (6)   | N2C—N1C—C8C—C6C            | 179.8 (6)    |
| Ni1—N1A—C8A—C6A           | -2.7 (11)   | Ni3—N1C—C8C—C6C            | 5.2 (10)     |
| C1A—C6A—C8A—N1A           | 3.8 (11)    | C5C—C6C—C8C—N1C            | -178.4 (7)   |
| C5A—C6A—C8A—N1A           | -174.8 (7)  | C1C—C6C—C8C—N1C            | 1.9 (11)     |
| C13A—N3A—C9A—N2A          | 178.7 (6)   | C13C—N3C—C9C—N2C           | 175.7 (6)    |
| Ni1—N3A—C9A—C6A           | 2.9 (7)     | Ni3—N3C—C9C—N2C            | -4.9 (8)     |
| C13A—N3A—C9A—C10A         | -2.2 (10)   | C13C—N3C—C9C—C10C          | -2.6 (10)    |
| Ni1—N3A—C9A—C10A          | -178.0 (5)  | Ni3—N3C—C9C—C10C           | 176.8 (6)    |
| N1A—N2A—C9A—N3A           | -6.0 (9)    | Ni3—N2C—C9C—N3C            | 3.9 (9)      |
| N1A—N2A—C9A—C10A          | 174.9 (6)   | N1C—N2C—C9C—C10C           | -177.8 (7)   |
| N3A—C3A—C10A—C11A         | 0.4 (11)    | N3C—C9C—C10C—C11C          | 1.4 (12)     |
| N2A—C9A—C10A—C11A         | 179.4 (7)   | N2C—C9C—C10C—C11C          | -176.8 (7)   |
| C9A—C10A—C11A—C12A        | 1.9 (11)    | C9C—C10C—C11C—C12C         | 0.4 (13)     |
| C10A—C11A—C12A—C13A       | -2.2 (11)   | C10C—C11C—C12C—C13C        | -1.0 (13)    |
| C9A—N3A—C13A—C12A         | 1.8 (10)    | C9C—N3C—C13C—C12C          | 2.0 (10)     |
| Ni1—N3A—C13A—C12A         | 176.9 (5)   | Ni3—N3C—C13C—C12C          | -177.3 (6)   |
| C11A—C12A—C13A—N3A        | 0.4 (11)    | C11C—C12C—C13C—N3C         | -0.3 (12)    |
| C8B—N1B—N2B—C9B           | 168.2 (6)   | C8D—N1D—N2D—C9D            | 172.1 (6)    |
| Ni2—N1B—N2B—C9B           | -8.9 (7)    | Ni4—N1D—N2D—C9D            | -7.5 (8)     |
| Ni2—O1B—C1B—C2B           | -165.5 (4)  | Ni4—O1D—C1D—C2D            | -169.2 (4)   |
| Ni1—O1B—C1B—C2B           | -10.6 (7)   | Ni3—O1D—C1D—C2D            | -5.0 (8)     |
| Ni2—O1B—C1B—C6B           | 16.7 (9)    | Ni4—O1D—C1D—C6D            | 14.1 (9)     |
| Ni1—O1B—C1B—C6B           | 171.7 (5)   | Ni3—O1D—C1D—C6D            | 178.3 (5)    |
| C7B—O2B—C2B—C3B           | -16.1 (10)  | C7D—O2D—C2D—C3D            | -20.3 (10)   |
| Ni1—O2B—C2B—C3B           | -171.2 (5)  | Ni3—O2D—C2D—C3D            | -178.1 (5)   |
| C7B—O2B—C2B—C1B           | 164.7 (6)   | C7D—O2D—C2D—C1D            | 162.5 (6)    |
| Ni1—O2B—C2B—C1B           | 9.6 (7)     | Ni3—O2D—C2D—C1D            | 4.7 (7)      |
| O1B—C1B—C2B—O2B           | -0.7 (9)    | O1D—C1D—C2D—C3D            | -177.7 (6)   |
| C6B—C1B—C2B—O2B           | 177.2 (6)   | C6D—C1D—C2D—C3D            | -0.8 (9)     |
| O1B—C1B—C2B—C3B           | -179.9 (6)  | O1D—C1D—C2D—O2D            | -0.4 (9)     |
| C6B—C1B—C2B—C3B           | -2.1 (10)   | C6D—C1D—C2D—O2D            | 176.5 (6)    |
| O2B—C2B—C3B—C4B           | -178.2 (6)  | O2D—C2D—C3D—C4D            | -177.0 (7)   |
| C1B—C2B—C3B—C4B           | 1.0 (10)    | C1D—C2D—C3D—C4D            | 0.0 (10)     |
| C2B—C3B—C4B—C5B           | 1.0 (11)    | C2D—C3D—C4D—C5D            | 1.0 (11)     |
| C3B—C4B—C5B—C6B           | -1.9 (11)   | C3D—C4D—C5D—C6D            | -1.3 (12)    |
| O1B—C1B—C6B—C5B           | 178.9 (6)   | O1D—C1D—C6D—C5D            | 177.3 (6)    |
| C2B—C1B—C6B—C5B           | 1.1 (10)    | C2D—C1D—C6D—C5D            | 0.5 (10)     |
| O1B—C1B—C6B—C8B           | -0.5 (10)   | O1D—C1D—C6D—C8D            | 0.6 (11)     |
| C2B—C1B—C6B—C8B           | -178.3 (6)  | C2D—C1D—C6D—C8D            | -176.2 (6)   |
| C4B—C5B—C6B—C1B           | 0.8 (11)    | C4D—C5D—C6D—C1D            | 0.5 (11)     |
| C4B—C5B—C6B—C8B           | -179.7 (7)  | C4D—C5D—C6D—C8D            | 177.5 (7)    |
| N2B—N1B—C8B—C6B           | 177.2 (6)   | N2D—N1D—C8D—C6D            | 177.0 (7)    |
| Ni2—N1B—C8B—C6B           | -6.3 (10)   | Ni4—N1D—C8D—C6D            | -3.5 (11)    |
| C1B—C6B—C8B—N1B           | -5.2 (11)   | C1D—C6D—C8D—N1D            | -6.1 (11)    |
| Bond                     | Angle (°) | Error   |
|-------------------------|-----------|---------|
| C5B—C6B—C8B—N1B        | 175.3 (6) |         |
| C5D—C6D—C8D—N1D        | 177.1 (7) |         |
| C13B—N3B—C9B—N2B      | -178.2 (6)|         |
| C13D—N3D—C9D—N2D      | -175.4 (6)|         |
| Ni2—N3B—C9B—N2B      | 2.5 (7)   |         |
| Ni4—N3D—C9D—N2D      | 3.8 (8)   |         |
| C13B—N3B—C9B—C10B    | 3.5 (10)  |         |
| C13D—N3D—C9D—C10D    | 3.8 (10)  |         |
| Ni2—N3B—C9B—C10B     | -175.8 (5)|         |
| Ni4—N3D—C9D—C10D     | -177.0 (6)|         |
| N1B—N2B—C9B—N3B      | 4.2 (9)   |         |
| N1D—N2D—C9D—N3D      | 2.5 (10)  |         |
| N1B—N2B—C9B—C10B     | -177.5 (6)|         |
| N1D—N2D—C9D—C10D     | -176.8 (7)|         |
| N3B—C9B—C10B—C11B   | -2.8 (11) |         |
| N3D—C9D—C10D—C11D   | -3.0 (11) |         |
| N2B—C9B—C10B—C11B   | 179.1 (7) |         |
| N2D—C9D—C10D—C11D   | 176.2 (7) |         |
| C9B—C10B—C11B—C12B  | -0.2 (11) |         |
| C9D—C10D—C11D—C12D  | 0.4 (12)  |         |
| C10B—C11B—C12B—C13B | 2.4 (11)  |         |
| C10D—C11D—C12D—C13D | 1.2 (12)  |         |
| C11B—C12B—C13B—N3B  | -1.6 (10) |         |
| C9D—N3D—C13D—C12D  | -2.1 (10) |         |
| C9B—N3B—C13B—C12B  | -1.3 (9)  |         |
| Ni4—N3D—C13D—C12D  | 178.9 (5) |         |
| Ni2—N3B—C13B—C12B | 177.9 (5) |         |
|                     |           |         |

Hydrogen-bond geometry (Å, °)

| Bond                      | D—H | H···A | D···A | D—H···A |
|--------------------------|-----|-------|-------|---------|
| C7B—H7BB···N1S           | 0.98| 2.60  | 3.523 (11) | 157     |
| C13B—H13B···O13          | 0.95| 2.42  | 3.126 (9)   | 131     |
| C13C—H13C···N1S          | 0.95| 2.59  | 3.348 (11) | 137     |
| N2B—H2DA···O2W           | 0.88| 1.91  | 2.720 (9)   | 152     |
| C7D—H7DC···O12           | 0.98| 2.56  | 3.389 (10) | 142     |
| C12S—H12G···O14          | 0.98| 2.37  | 3.335 (12) | 168     |
| O2W···H2W1···O11         | 0.84 (3)| 2.12 (7) | 2.795 (8) | 138 (9) |
| O2W···H2W2···C14         | 0.83 (3)| 2.78 (3) | 3.599 (6) | 170 (9) |

Symmetry codes: (i) x+1/2, -y+3/2, z; (ii) -x+1, -y+1, z-1/2.