

Poly[[diaqua(μ_2 -4,4'-dipyridyl sulfide- κ^2 N:N')(4,4'-dipyridyl sulfide- κ N)-(2-hydroxy-5-sulfonatobenzoato- κ O¹)-nickel(II)] dihydrate]

Zhong-Xiang Du* and Jun-Xia Li

Department of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, Henan 471022, People's Republic of China

Correspondence e-mail: dzx6281@126.com

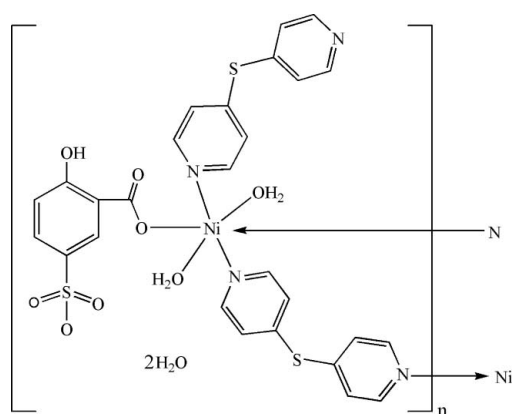
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; disorder in main residue; R factor = 0.058; wR factor = 0.179; data-to-parameter ratio = 14.2.

The asymmetric unit of the title helical coordination polymer, $\{[\text{Ni}(\text{C}_7\text{H}_4\text{O}_6\text{S})(\text{C}_{10}\text{H}_8\text{N}_2\text{S})_2(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}\}_n$, is comprised of an Ni^{II} ion, one 5-sulfosalicylic acid dianion (HSSA), two 4,4'-dipyridylsulfide (4,4'-dps) ligands, and two uncoordinated water molecules. The Ni^{II} ion is coordinated by two water molecules, one carboxylate O atom of the HSSA dianion and three N atoms from three 4,4'-dps ligands in a distorted octahedral environment. Half of the 4,4'-dps ligands are μ_2 -bridging ligands which link adjacent Ni^{II} centers, forming a one-dimensional helical structure along the b axis. This helical structure is further stabilized by O—H...O intra- and intermolecular hydrogen bonds.

Related literature

For related structures, see: Fujita *et al.* (1994); Hao & Zhang (2007); Hou *et al.* (2001); Jung *et al.* (1999, 2000); Niu *et al.* (2006); Vaganova *et al.* (2004); Wen *et al.* (2004); Zeng *et al.* (2006); Zheng & Vittal (2001); Zheng *et al.* (1999).



Experimental

Crystal data

$[\text{Ni}(\text{C}_7\text{H}_4\text{O}_6\text{S})(\text{C}_{10}\text{H}_8\text{N}_2\text{S})_2 \cdot (\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$
 $M_r = 723.42$
 Monoclinic, $P2_1/n$
 $a = 11.4649$ (10) Å
 $b = 13.9441$ (12) Å
 $c = 20.7051$ (18) Å

$\beta = 96.5520$ (10)°
 $V = 3288.5$ (5) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.84$ mm⁻¹
 $T = 291$ (2) K
 $0.44 \times 0.26 \times 0.18$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.709$, $T_{\text{max}} = 0.866$
 23823 measured reflections
 6054 independent reflections
 4328 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.058$
 $wR(F^2) = 0.178$
 $S = 1.02$
 6054 reflections
 426 parameters
 219 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.96$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.58$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|------------------------|-------------|-----------|-------------|
| Ni1—O9 | 1.981 (3) | Ni1—N3 | 2.055 (4) |
| Ni1—N4 ⁱ | 2.040 (3) | Ni1—O2 | 2.430 (3) |
| Ni1—N1 | 2.041 (4) | Ni1—O5 | 2.437 (3) |
| O9—Ni1—N4 ⁱ | 173.60 (14) | N1—Ni1—O2 | 87.06 (14) |
| O9—Ni1—N1 | 88.00 (13) | O9—Ni1—O5 | 80.75 (12) |
| O9—Ni1—N3 | 87.87 (13) | N1—Ni1—O5 | 93.08 (14) |
| N1—Ni1—N3 | 175.23 (14) | O2—Ni1—O5 | 173.73 (12) |
| O9—Ni1—O2 | 92.99 (12) | | |

Symmetry code: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$.

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|-----------------------------|-------|--------------|--------------|----------------|
| O11—H11...O10 | 0.82 | 1.84 | 2.535 (5) | 142 |
| O5—H10W...O3 ⁱⁱ | 0.83 | 1.98 | 2.797 (5) | 170 |
| O5—H9W...O4 ⁱⁱⁱ | 0.85 | 2.04 | 2.721 (8) | 136 |
| O4—H7W...O8 ^{iv} | 0.83 | 2.30 | 2.713 (9) | 112 |
| O3—H6W...O6 ^v | 0.83 | 2.10 | 2.811 (7) | 143 |
| O3—H5W...O6 ^{vi} | 0.83 | 2.24 | 2.765 (8) | 122 |
| O2—H4W...O10 | 0.83 | 1.95 | 2.690 (5) | 149 |
| O2—H3W...O7 ^v | 0.84 | 1.87 | 2.652 (7) | 155 |
| O1—H1W...O11 ^{vii} | 0.85 | 2.03 | 2.876 (7) | 180 |

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

Data collection: APEX2 (Bruker, 2004); cell refinement: APEX2; data reduction: SAINT (Bruker, 2004); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); 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: AT2628).

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supplementary materials

Acta Cryst. (2008). E64, m1278-m1279 [doi:10.1107/S1600536808029206]

Poly[[diaqua(μ_2 -4,4'-dipyridyl sulfide- $\kappa^2N:N'$)(4,4'-dipyridyl sulfide- κN)(2-hydroxy-5-sulfonato-benzoato- κO^1)nickel(II)] dihydrate]

Z.-X. Du and J.-X. Li

Comment

Recently, the use of bipyridyl based bridging ligands and transition metal centers in the preparation of various coordination compounds have attracted considerable interests not only because of their structural novelty but also for their potential properties in magnetism (Zheng *et al.*, 1999), nonlinear optics (Hou *et al.*, 2001), catalysts (Fujita *et al.*, 1994) and so on. 4,4'-dps possesses a magic angle (the angle of C—S—C almost equals to 100°) and conformational nonrigidity so it has some flexibility in contrast to linear rigid ligands such as simple 4,4'-bipyridine analogues. A number of metal complexes derived from 4,4'-dps have been reported previously. Among them, the 4,4'-dps has three kind of coordination modes and they are non-coordinate (Zeng *et al.*, 2006; Wen *et al.*, 2004; Vaganova *et al.*, 2004), μ_2 -bridging (Zheng & Vittal, 2001; Jung *et al.*, 2000; Hao & Zhang, 2007; Niu *et al.*, 2006), μ_2 and μ_3 together (Jung *et al.*, 1999). In this paper, we describe another new compound in which the 4,4'-dps is monodentate and μ_2 -bridging together, (I), (Fig. 1).

Complex (I) is composed of $[\text{Ni}(\text{C}_{10}\text{H}_8\text{N}_2\text{S})_2(\text{C}_7\text{H}_4\text{O}_6\text{S})(\text{H}_2\text{O})_2] \cdot 2\text{H}_2\text{O}$ units, in which the Ni^{II} ion is six-coordinated in a distorted octahedral geometry (Table 1) formed by two coordinate water molecules, one carboxylate O atom of HSSA dianion, one N atoms from monodentate 4,4'-dps ligand and another two N atoms from another two μ_2 -bridging 4,4'-dps ligands. Half of the 4,4'-dps are monodentate and the other half are μ_2 -bridging. It is just through the μ_2 -bridging function that the adjacent Ni^{II} centers are joined to form a one-dimensional helix structure (Fig. 2, 3 & 4) along *b* axis in the monoclinic unit cell, with the $\text{Ni} \cdots \text{Ni}(1/2 - x, 1/2 + y, 3/2 - z)$ distance of 10.6096 (10) Å. The phenolic hydroxyl and carboxyl of HSSA dianion are involved in intramolecular hydrogen bonding (Table 2). Together with the other O—H \cdots O intermolecular hydrogen bonds with participation of water molecules, the helix structure are further stabilized.

Experimental

The ligand 4,4'-dps (0.5 mmol, 0.14 g), 5-sulfosalicylic acid (0.5 mmol, 0.13 g) and NaOH (1.0 mmol, 0.04 g) were dissolved in water and methanol mixed solvent (30 ml, *v/v* 1:1). To this solution, $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ (0.5 mmol, 0.13 g) was added and the resulting mixture was stirred and refluxed at 353 K for 2.5 h, then cooled to room temperature. After filtration and evaporation in air for 3 days, green block-shaped crystals were obtained in a yield of 32%. Analysis, found (%): C, 44.83; H 3.84, N 7.79, S 13.22. $\text{C}_{27}\text{H}_{28}\text{N}_4\text{NiO}_{10}\text{S}_3$ requires (%): C 44.78, H 3.87, N 7.74, S 13.27. [CCDC number 656224].

Refinement

H atoms bonded to C atoms were positioned geometrically with C—H distance of 0.93 Å, and treated as riding atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$. H atoms bonded to O atoms were located in a difference Fourier map and refined isotropically.

Figures

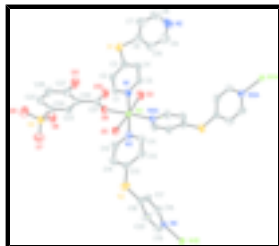


Fig. 1. The coordination environment of Ni^{II} ion in (I), with displacement ellipsoids shown at the 30% probability level. Uncoordinated water molecules and H atoms have been omitted [Symmetry codes: (A) $1/2 - x, 1/2 + y, 3/2 - z$; (B) $1/2 - x, -1/2 + y, 3/2 - z$].



Fig. 2. The helix structure for (I) along *b* axis. Uncoordinated water molecules and H atoms on C atoms have been omitted.

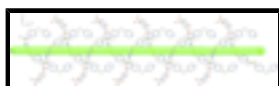


Fig. 3. The helix structure for (I) along *b* axis with a helix axis. Uncoordinated water molecules and H atoms have been omitted.

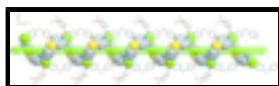


Fig. 4. The space filled diagram of the helix structure for (I) along *b* axis. Uncoordinated water molecules and H atoms have been omitted.

Poly[[diaqua(μ_2 -4,4'-dipyridyl sulfide- $\kappa^2N:N'$)(4,4'-dipyridyl sulfide- κN)(2-hydroxy-5-sulfonatobenzoato- κO^1)nickel(II)] dihydrate]

Crystal data

[Ni(C₇H₄O₆S)(C₁₀H₈N₂S)₂(H₂O)₂] \cdot 2H₂O

$M_r = 723.42$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 11.4649$ (10) Å

$b = 13.9441$ (12) Å

$c = 20.7051$ (18) Å

$\beta = 96.5520$ (10)°

$V = 3288.5$ (5) Å³

$Z = 4$

$F_{000} = 1496$

$D_x = 1.461$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4865 reflections

$\theta = 2.3$ – 21.6 °

$\mu = 0.84$ mm⁻¹

$T = 291$ (2) K

Block, green

$0.44 \times 0.26 \times 0.18$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 291$ (2) K

ϕ and ω scans

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

$T_{\min} = 0.709$, $T_{\max} = 0.866$

6054 independent reflections

4328 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\text{max}} = 25.5$ °

$\theta_{\text{min}} = 2.4$ °

$h = -13 \rightarrow 13$

$k = -16 \rightarrow 16$

23823 measured reflections

$l = -25 \rightarrow 25$

Refinement

Refinement on F^2

Secondary atom site location: difference Fourier map

Least-squares matrix: full

Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.058$

H-atom parameters constrained

$wR(F^2) = 0.178$

$$w = 1/[\sigma^2(F_o^2) + (0.0999P)^2 + 2.6202P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.03$

$(\Delta/\sigma)_{\max} = 0.001$

6054 reflections

$\Delta\rho_{\max} = 0.96 \text{ e } \text{\AA}^{-3}$

426 parameters

$\Delta\rho_{\min} = -0.58 \text{ e } \text{\AA}^{-3}$

219 restraints

Extinction correction: none

Primary atom site location: structure-invariant direct methods

Special details

Experimental. The sulfonic group of HSSA dianion is in disorder and has been refined but not satisfactory.

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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|--------------|---------------|--------------|----------------------------------|-----------|
| S3 | 1.11886 (11) | -0.14488 (10) | 0.89754 (7) | 0.0681 (4) | 0.622 (5) |
| O6 | 1.0657 (6) | -0.0994 (5) | 0.9516 (3) | 0.0971 (16) | 0.622 (5) |
| O7 | 1.0712 (6) | -0.2372 (4) | 0.8826 (3) | 0.0973 (17) | 0.622 (5) |
| O8 | 1.2456 (4) | -0.1450 (6) | 0.9124 (4) | 0.1026 (19) | 0.622 (5) |
| S3' | 1.11886 (11) | -0.14488 (10) | 0.89754 (7) | 0.0681 (4) | 0.378 (5) |
| O6' | 1.0085 (7) | -0.1624 (8) | 0.9241 (5) | 0.0971 (16) | 0.378 (5) |
| O7' | 1.1545 (9) | -0.2352 (6) | 0.8675 (5) | 0.0973 (17) | 0.378 (5) |
| O8' | 1.2068 (8) | -0.1005 (8) | 0.9383 (6) | 0.1026 (19) | 0.378 (5) |
| Ni1 | 0.59573 (4) | 0.10463 (4) | 0.80537 (3) | 0.04492 (19) | |
| S1 | 0.86708 (13) | 0.45268 (11) | 0.97477 (9) | 0.0867 (5) | |
| S2 | 0.42135 (12) | -0.26440 (11) | 0.61663 (7) | 0.0762 (4) | |
| O1 | 0.2056 (7) | 0.1245 (5) | 0.6004 (4) | 0.081 (2) | 0.50 |
| H1W | 0.1451 | 0.1176 | 0.6202 | 0.122* | 0.50 |
| H2W | 0.2395 | 0.1543 | 0.6298 | 0.122* | 0.50 |
| O2 | 0.5916 (3) | 0.2119 (3) | 0.71261 (17) | 0.0762 (10) | |

supplementary materials

| | | | | | |
|------|------------|-------------|--------------|-------------|------|
| H3W | 0.5535 | 0.2426 | 0.6827 | 0.114* | |
| H4W | 0.6615 | 0.2079 | 0.7065 | 0.114* | |
| O3 | 0.6648 (4) | 0.4534 (3) | 0.5299 (2) | 0.0938 (12) | |
| H5W | 0.6367 | 0.4593 | 0.4912 | 0.141* | |
| H6W | 0.6116 | 0.4399 | 0.5528 | 0.141* | |
| O4 | 0.4748 (7) | 0.8801 (5) | 0.9582 (4) | 0.084 (2) | 0.50 |
| H7W | 0.4223 | 0.8558 | 0.9771 | 0.127* | 0.50 |
| H8W | 0.4646 | 0.8593 | 0.9178 | 0.127* | 0.50 |
| O5 | 0.6211 (3) | -0.0084 (3) | 0.89567 (18) | 0.0763 (10) | |
| H9W | 0.5833 | -0.0606 | 0.8975 | 0.114* | |
| H10W | 0.6840 | -0.0261 | 0.9156 | 0.114* | |
| O9 | 0.7577 (2) | 0.0611 (2) | 0.79683 (15) | 0.0566 (7) | |
| O10 | 0.8024 (3) | 0.1301 (3) | 0.70624 (19) | 0.0765 (10) | |
| O11 | 1.0017 (3) | 0.1011 (3) | 0.6679 (2) | 0.0915 (13) | |
| H11 | 0.9311 | 0.1125 | 0.6621 | 0.137* | |
| N1 | 0.6667 (3) | 0.2157 (3) | 0.86085 (17) | 0.0531 (9) | |
| N2 | 0.6301 (7) | 0.7173 (4) | 0.9694 (4) | 0.119 (2) | |
| N3 | 0.5365 (3) | -0.0069 (3) | 0.74554 (18) | 0.0545 (9) | |
| N4 | 0.0690 (3) | -0.3576 (3) | 0.67586 (18) | 0.0512 (8) | |
| C1 | 0.7555 (4) | 0.1994 (3) | 0.9076 (2) | 0.0577 (11) | |
| H1 | 0.7793 | 0.1363 | 0.9159 | 0.069* | |
| C2 | 0.8125 (4) | 0.2702 (4) | 0.9436 (2) | 0.0609 (12) | |
| H2 | 0.8734 | 0.2553 | 0.9756 | 0.073* | |
| C3 | 0.7792 (4) | 0.3647 (3) | 0.9322 (2) | 0.0591 (11) | |
| C4 | 0.6873 (5) | 0.3829 (4) | 0.8847 (3) | 0.0680 (13) | |
| H4 | 0.6615 | 0.4454 | 0.8760 | 0.082* | |
| C5 | 0.6349 (4) | 0.3076 (3) | 0.8506 (2) | 0.0628 (12) | |
| H5 | 0.5736 | 0.3208 | 0.8184 | 0.075* | |
| C6 | 0.7725 (5) | 0.5558 (4) | 0.9719 (3) | 0.0777 (15) | |
| C7 | 0.8082 (7) | 0.6385 (5) | 0.9465 (3) | 0.097 (2) | |
| H7 | 0.8803 | 0.6426 | 0.9302 | 0.116* | |
| C8 | 0.7340 (9) | 0.7170 (5) | 0.9456 (4) | 0.113 (2) | |
| H8 | 0.7579 | 0.7735 | 0.9272 | 0.135* | |
| C9 | 0.5986 (7) | 0.6361 (6) | 0.9958 (4) | 0.115 (2) | |
| H9 | 0.5272 | 0.6343 | 1.0130 | 0.138* | |
| C10 | 0.6680 (6) | 0.5523 (5) | 0.9989 (4) | 0.0945 (19) | |
| H10 | 0.6442 | 0.4967 | 1.0185 | 0.113* | |
| C11 | 0.4525 (4) | 0.0005 (4) | 0.6954 (2) | 0.0617 (12) | |
| H11A | 0.4178 | 0.0600 | 0.6866 | 0.074* | |
| C12 | 0.4151 (4) | -0.0753 (4) | 0.6564 (2) | 0.0647 (12) | |
| H12 | 0.3560 | -0.0664 | 0.6222 | 0.078* | |
| C13 | 0.4639 (4) | -0.1637 (3) | 0.6674 (2) | 0.0592 (11) | |
| C14 | 0.5518 (5) | -0.1722 (4) | 0.7173 (3) | 0.0743 (14) | |
| H14 | 0.5884 | -0.2311 | 0.7258 | 0.089* | |
| C15 | 0.5863 (5) | -0.0938 (4) | 0.7548 (3) | 0.0733 (14) | |
| H15 | 0.6471 | -0.1012 | 0.7882 | 0.088* | |
| C16 | 0.2851 (4) | -0.2967 (3) | 0.6419 (2) | 0.0548 (10) | |
| C17 | 0.2466 (4) | -0.2696 (4) | 0.7005 (2) | 0.0635 (12) | |
| H17 | 0.2928 | -0.2305 | 0.7295 | 0.076* | |

| | | | | |
|-----|------------|-------------|------------|-------------|
| C18 | 0.1396 (4) | -0.3014 (4) | 0.7149 (2) | 0.0614 (12) |
| H18 | 0.1148 | -0.2827 | 0.7542 | 0.074* |
| C19 | 0.1080 (4) | -0.3847 (4) | 0.6199 (2) | 0.0628 (12) |
| H19 | 0.0610 | -0.4249 | 0.5921 | 0.075* |
| C20 | 0.2136 (4) | -0.3559 (4) | 0.6018 (2) | 0.0645 (12) |
| H20 | 0.2369 | -0.3763 | 0.5625 | 0.077* |
| C21 | 0.8262 (4) | 0.0772 (3) | 0.7551 (2) | 0.0487 (10) |
| C22 | 0.9428 (4) | 0.0302 (3) | 0.7648 (2) | 0.0500 (10) |
| C23 | 0.9732 (4) | -0.0280 (3) | 0.8182 (2) | 0.0520 (10) |
| H23 | 0.9185 | -0.0395 | 0.8471 | 0.062* |
| C24 | 1.0833 (4) | -0.0692 (3) | 0.8294 (2) | 0.0558 (11) |
| C25 | 1.1637 (4) | -0.0526 (4) | 0.7859 (3) | 0.0692 (13) |
| H25 | 1.2382 | -0.0795 | 0.7934 | 0.083* |
| C26 | 1.1358 (4) | 0.0022 (4) | 0.7325 (3) | 0.0786 (16) |
| H26 | 1.1907 | 0.0115 | 0.7033 | 0.094* |
| C27 | 1.0251 (4) | 0.0452 (4) | 0.7209 (2) | 0.0638 (12) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| S3 | 0.0600 (7) | 0.0674 (8) | 0.0720 (8) | 0.0162 (6) | -0.0135 (6) | -0.0046 (6) |
| O6 | 0.097 (3) | 0.101 (4) | 0.091 (3) | 0.027 (3) | 0.001 (3) | 0.014 (3) |
| O7 | 0.114 (4) | 0.080 (3) | 0.090 (3) | -0.011 (3) | -0.024 (3) | 0.010 (2) |
| O8 | 0.074 (3) | 0.118 (4) | 0.108 (4) | 0.007 (3) | -0.026 (3) | 0.012 (3) |
| S3' | 0.0600 (7) | 0.0674 (8) | 0.0720 (8) | 0.0162 (6) | -0.0135 (6) | -0.0046 (6) |
| O6' | 0.097 (3) | 0.101 (4) | 0.091 (3) | 0.027 (3) | 0.001 (3) | 0.014 (3) |
| O7' | 0.114 (4) | 0.080 (3) | 0.090 (3) | -0.011 (3) | -0.024 (3) | 0.010 (2) |
| O8' | 0.074 (3) | 0.118 (4) | 0.108 (4) | 0.007 (3) | -0.026 (3) | 0.012 (3) |
| Ni1 | 0.0342 (3) | 0.0437 (3) | 0.0563 (3) | 0.0050 (2) | 0.0026 (2) | -0.0027 (2) |
| S1 | 0.0655 (8) | 0.0828 (10) | 0.1068 (12) | -0.0091 (7) | -0.0121 (8) | -0.0239 (8) |
| S2 | 0.0628 (8) | 0.0861 (9) | 0.0849 (9) | -0.0234 (7) | 0.0313 (7) | -0.0296 (7) |
| O1 | 0.090 (5) | 0.062 (4) | 0.103 (5) | -0.020 (3) | 0.061 (4) | -0.011 (3) |
| O2 | 0.068 (2) | 0.088 (2) | 0.073 (2) | 0.0191 (18) | 0.0125 (17) | 0.0177 (19) |
| O3 | 0.081 (3) | 0.122 (3) | 0.076 (2) | 0.007 (2) | -0.003 (2) | -0.007 (2) |
| O4 | 0.069 (4) | 0.059 (4) | 0.127 (6) | 0.001 (3) | 0.020 (4) | 0.031 (4) |
| O5 | 0.067 (2) | 0.072 (2) | 0.088 (2) | 0.0028 (17) | 0.0020 (18) | 0.0209 (19) |
| O9 | 0.0428 (16) | 0.0611 (18) | 0.0661 (19) | 0.0083 (14) | 0.0075 (14) | 0.0037 (15) |
| O10 | 0.059 (2) | 0.089 (2) | 0.082 (2) | 0.0182 (18) | 0.0104 (17) | 0.026 (2) |
| O11 | 0.068 (2) | 0.119 (3) | 0.092 (3) | 0.016 (2) | 0.027 (2) | 0.039 (2) |
| N1 | 0.0474 (19) | 0.053 (2) | 0.058 (2) | 0.0062 (16) | 0.0000 (16) | 0.0026 (16) |
| N2 | 0.142 (6) | 0.066 (4) | 0.140 (6) | 0.004 (4) | -0.027 (5) | -0.021 (4) |
| N3 | 0.0425 (18) | 0.057 (2) | 0.063 (2) | 0.0050 (16) | 0.0035 (16) | 0.0009 (18) |
| N4 | 0.0455 (19) | 0.051 (2) | 0.057 (2) | -0.0061 (16) | 0.0047 (16) | -0.0015 (17) |
| C1 | 0.054 (3) | 0.053 (3) | 0.064 (3) | 0.006 (2) | -0.003 (2) | 0.008 (2) |
| C2 | 0.054 (3) | 0.069 (3) | 0.057 (3) | 0.004 (2) | -0.005 (2) | 0.007 (2) |
| C3 | 0.056 (3) | 0.061 (3) | 0.059 (3) | -0.004 (2) | 0.004 (2) | -0.009 (2) |
| C4 | 0.071 (3) | 0.055 (3) | 0.073 (3) | 0.006 (2) | -0.014 (3) | -0.001 (2) |
| C5 | 0.058 (3) | 0.057 (3) | 0.069 (3) | 0.012 (2) | -0.014 (2) | -0.001 (2) |

supplementary materials

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|-----|-----------|-----------|-----------|--------------|--------------|-------------|
| C6 | 0.081 (4) | 0.072 (4) | 0.075 (3) | -0.015 (3) | -0.014 (3) | -0.017 (3) |
| C7 | 0.125 (6) | 0.084 (4) | 0.079 (4) | -0.019 (4) | -0.001 (4) | -0.005 (3) |
| C8 | 0.148 (8) | 0.073 (5) | 0.111 (6) | -0.006 (5) | -0.012 (5) | 0.001 (4) |
| C9 | 0.109 (6) | 0.098 (6) | 0.135 (7) | 0.002 (5) | -0.002 (5) | -0.027 (5) |
| C10 | 0.085 (4) | 0.072 (4) | 0.124 (5) | -0.005 (3) | 0.001 (4) | -0.014 (4) |
| C11 | 0.050 (2) | 0.064 (3) | 0.071 (3) | 0.008 (2) | 0.002 (2) | 0.001 (2) |
| C12 | 0.055 (3) | 0.073 (3) | 0.064 (3) | -0.006 (2) | 0.000 (2) | -0.005 (2) |
| C13 | 0.047 (2) | 0.062 (3) | 0.071 (3) | -0.010 (2) | 0.015 (2) | -0.009 (2) |
| C14 | 0.081 (4) | 0.051 (3) | 0.088 (4) | 0.002 (2) | -0.003 (3) | -0.006 (3) |
| C15 | 0.074 (3) | 0.061 (3) | 0.079 (3) | 0.013 (3) | -0.018 (3) | -0.002 (3) |
| C16 | 0.049 (2) | 0.059 (3) | 0.057 (3) | -0.003 (2) | 0.0089 (19) | -0.002 (2) |
| C17 | 0.052 (3) | 0.074 (3) | 0.066 (3) | -0.020 (2) | 0.012 (2) | -0.024 (2) |
| C18 | 0.056 (3) | 0.071 (3) | 0.059 (3) | -0.013 (2) | 0.013 (2) | -0.012 (2) |
| C19 | 0.060 (3) | 0.067 (3) | 0.061 (3) | -0.015 (2) | 0.006 (2) | -0.011 (2) |
| C20 | 0.067 (3) | 0.072 (3) | 0.057 (3) | -0.017 (2) | 0.016 (2) | -0.015 (2) |
| C21 | 0.044 (2) | 0.045 (2) | 0.056 (2) | -0.0004 (17) | -0.0016 (19) | 0.0009 (19) |
| C22 | 0.041 (2) | 0.047 (2) | 0.062 (3) | 0.0018 (17) | 0.0029 (19) | -0.008 (2) |
| C23 | 0.047 (2) | 0.050 (2) | 0.057 (2) | 0.0039 (18) | -0.0016 (19) | -0.004 (2) |
| C24 | 0.044 (2) | 0.051 (2) | 0.070 (3) | 0.0082 (19) | -0.003 (2) | -0.010 (2) |
| C25 | 0.048 (3) | 0.069 (3) | 0.090 (4) | 0.013 (2) | 0.005 (3) | -0.007 (3) |
| C26 | 0.051 (3) | 0.097 (4) | 0.091 (4) | 0.017 (3) | 0.023 (3) | 0.006 (3) |
| C27 | 0.051 (3) | 0.075 (3) | 0.066 (3) | 0.000 (2) | 0.007 (2) | 0.008 (2) |

Geometric parameters (Å, °)

| | | | |
|---------------------|-----------|----------|------------|
| S3—O7 | 1.418 (5) | C2—H2 | 0.9300 |
| S3—O8 | 1.450 (5) | C3—C4 | 1.381 (7) |
| S3—O6 | 1.477 (5) | C4—C5 | 1.365 (7) |
| S3—C24 | 1.771 (5) | C4—H4 | 0.9300 |
| Ni1—O9 | 1.981 (3) | C5—H5 | 0.9300 |
| Ni1—N4 ⁱ | 2.040 (3) | C6—C7 | 1.349 (8) |
| Ni1—N1 | 2.041 (4) | C6—C10 | 1.379 (9) |
| Ni1—N3 | 2.055 (4) | C7—C8 | 1.385 (11) |
| Ni1—O2 | 2.430 (3) | C7—H7 | 0.9300 |
| Ni1—O5 | 2.437 (3) | C8—H8 | 0.9300 |
| S1—C3 | 1.759 (5) | C9—C10 | 1.411 (9) |
| S1—C6 | 1.798 (6) | C9—H9 | 0.9300 |
| S2—C16 | 1.762 (4) | C10—H10 | 0.9300 |
| S2—C13 | 1.789 (5) | C11—C12 | 1.369 (7) |
| O1—H1W | 0.8504 | C11—H11A | 0.9300 |
| O1—H2W | 0.8000 | C12—C13 | 1.362 (7) |
| O2—H3W | 0.8350 | C12—H12 | 0.9300 |
| O2—H4W | 0.8278 | C13—C14 | 1.363 (7) |
| O3—H5W | 0.8338 | C14—C15 | 1.373 (7) |
| O3—H6W | 0.8347 | C14—H14 | 0.9300 |
| O4—H7W | 0.8256 | C15—H15 | 0.9300 |
| O4—H8W | 0.8821 | C16—C20 | 1.375 (6) |
| O5—H9W | 0.8501 | C16—C17 | 1.390 (6) |
| O5—H10W | 0.8267 | C17—C18 | 1.369 (6) |

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|-------------------------|-------------|--------------|-----------|
| O9—C21 | 1.252 (5) | C17—H17 | 0.9300 |
| O10—C21 | 1.256 (5) | C18—H18 | 0.9300 |
| O11—C27 | 1.348 (6) | C19—C20 | 1.367 (7) |
| O11—H11 | 0.8200 | C19—H19 | 0.9300 |
| N1—C1 | 1.342 (5) | C20—H20 | 0.9300 |
| N1—C5 | 1.343 (6) | C21—C22 | 1.482 (6) |
| N2—C9 | 1.324 (10) | C22—C23 | 1.384 (6) |
| N2—C8 | 1.340 (11) | C22—C27 | 1.397 (6) |
| N3—C11 | 1.337 (6) | C23—C24 | 1.382 (6) |
| N3—C15 | 1.344 (6) | C23—H23 | 0.9300 |
| N4—C18 | 1.332 (6) | C24—C25 | 1.380 (7) |
| N4—C19 | 1.344 (6) | C25—C26 | 1.352 (8) |
| N4—Ni1 ⁱⁱ | 2.040 (3) | C25—H25 | 0.9300 |
| C1—C2 | 1.358 (6) | C26—C27 | 1.399 (7) |
| C1—H1 | 0.9300 | C26—H26 | 0.9300 |
| C2—C3 | 1.385 (7) | | |
| O7—S3—O8 | 113.4 (4) | C6—C7—H7 | 121.1 |
| O7—S3—O6 | 111.9 (4) | C8—C7—H7 | 121.1 |
| O8—S3—O6 | 109.2 (4) | N2—C8—C7 | 124.5 (8) |
| O7—S3—C24 | 108.6 (3) | N2—C8—H8 | 117.7 |
| O8—S3—C24 | 107.7 (4) | C7—C8—H8 | 117.7 |
| O6—S3—C24 | 105.7 (3) | N2—C9—C10 | 123.2 (8) |
| O9—Ni1—N4 ⁱ | 173.60 (14) | N2—C9—H9 | 118.4 |
| O9—Ni1—N1 | 88.00 (13) | C10—C9—H9 | 118.4 |
| N4 ⁱ —Ni1—N1 | 90.87 (14) | C6—C10—C9 | 117.6 (7) |
| O9—Ni1—N3 | 87.87 (13) | C6—C10—H10 | 121.2 |
| N4 ⁱ —Ni1—N3 | 93.50 (14) | C9—C10—H10 | 121.2 |
| N1—Ni1—N3 | 175.23 (14) | N3—C11—C12 | 123.3 (5) |
| O9—Ni1—O2 | 92.99 (12) | N3—C11—H11A | 118.4 |
| N4 ⁱ —Ni1—O2 | 93.24 (13) | C12—C11—H11A | 118.4 |
| N1—Ni1—O2 | 87.06 (14) | C13—C12—C11 | 120.3 (5) |
| N3—Ni1—O2 | 90.79 (14) | C13—C12—H12 | 119.9 |
| O9—Ni1—O5 | 80.75 (12) | C11—C12—H12 | 119.9 |
| N4 ⁱ —Ni1—O5 | 93.03 (13) | C12—C13—C14 | 117.4 (4) |
| N1—Ni1—O5 | 93.08 (14) | C12—C13—S2 | 122.2 (4) |
| N3—Ni1—O5 | 88.60 (14) | C14—C13—S2 | 120.3 (4) |
| O2—Ni1—O5 | 173.73 (12) | C13—C14—C15 | 120.0 (5) |
| C3—S1—C6 | 103.4 (2) | C13—C14—H14 | 120.0 |
| C16—S2—C13 | 102.6 (2) | C15—C14—H14 | 120.0 |
| H1W—O1—H2W | 92.5 | N3—C15—C14 | 123.1 (5) |
| Ni1—O2—H3W | 149.8 | N3—C15—H15 | 118.4 |
| Ni1—O2—H4W | 98.5 | C14—C15—H15 | 118.4 |
| H3W—O2—H4W | 110.7 | C20—C16—C17 | 117.7 (4) |
| H5W—O3—H6W | 109.9 | C20—C16—S2 | 117.5 (3) |
| H7W—O4—H8W | 106.8 | C17—C16—S2 | 124.8 (3) |
| Ni1—O5—H9W | 124.9 | C18—C17—C16 | 118.9 (4) |
| Ni1—O5—H10W | 126.7 | C18—C17—H17 | 120.6 |

supplementary materials

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| H9W—O5—H10W | 98.2 | C16—C17—H17 | 120.6 |
| C21—O9—Ni1 | 132.4 (3) | N4—C18—C17 | 123.8 (4) |
| C27—O11—H11 | 109.5 | N4—C18—H18 | 118.1 |
| C1—N1—C5 | 116.5 (4) | C17—C18—H18 | 118.1 |
| C1—N1—Ni1 | 119.9 (3) | N4—C19—C20 | 123.1 (4) |
| C5—N1—Ni1 | 123.5 (3) | N4—C19—H19 | 118.5 |
| C9—N2—C8 | 116.5 (7) | C20—C19—H19 | 118.5 |
| C11—N3—C15 | 115.9 (4) | C19—C20—C16 | 119.7 (4) |
| C11—N3—Ni1 | 124.7 (3) | C19—C20—H20 | 120.1 |
| C15—N3—Ni1 | 119.4 (3) | C16—C20—H20 | 120.1 |
| C18—N4—C19 | 116.7 (4) | O9—C21—O10 | 124.4 (4) |
| C18—N4—Ni1 ⁱⁱ | 123.2 (3) | O9—C21—C22 | 117.0 (4) |
| C19—N4—Ni1 ⁱⁱ | 119.9 (3) | O10—C21—C22 | 118.6 (4) |
| N1—C1—C2 | 123.3 (4) | C23—C22—C27 | 118.7 (4) |
| N1—C1—H1 | 118.3 | C23—C22—C21 | 120.5 (4) |
| C2—C1—H1 | 118.3 | C27—C22—C21 | 120.8 (4) |
| C1—C2—C3 | 119.6 (4) | C24—C23—C22 | 121.3 (4) |
| C1—C2—H2 | 120.2 | C24—C23—H23 | 119.3 |
| C3—C2—H2 | 120.2 | C22—C23—H23 | 119.3 |
| C4—C3—C2 | 118.0 (4) | C25—C24—C23 | 119.0 (5) |
| C4—C3—S1 | 125.2 (4) | C25—C24—S3 | 120.6 (3) |
| C2—C3—S1 | 116.6 (4) | C23—C24—S3 | 120.3 (4) |
| C5—C4—C3 | 118.8 (4) | C26—C25—C24 | 121.0 (4) |
| C5—C4—H4 | 120.6 | C26—C25—H25 | 119.5 |
| C3—C4—H4 | 120.6 | C24—C25—H25 | 119.5 |
| N1—C5—C4 | 123.9 (4) | C25—C26—C27 | 120.5 (5) |
| N1—C5—H5 | 118.1 | C25—C26—H26 | 119.8 |
| C4—C5—H5 | 118.1 | C27—C26—H26 | 119.8 |
| C7—C6—C10 | 120.3 (7) | O11—C27—C22 | 121.9 (4) |
| C7—C6—S1 | 119.2 (6) | O11—C27—C26 | 118.7 (5) |
| C10—C6—S1 | 120.4 (5) | C22—C27—C26 | 119.4 (5) |
| C6—C7—C8 | 117.9 (8) | | |
| N4 ⁱ —Ni1—O9—C21 | -172.7 (11) | Ni1—N3—C11—C12 | 180.0 (4) |
| N1—Ni1—O9—C21 | -92.8 (4) | N3—C11—C12—C13 | -0.3 (8) |
| N3—Ni1—O9—C21 | 84.9 (4) | C11—C12—C13—C14 | -1.4 (7) |
| O2—Ni1—O9—C21 | -5.8 (4) | C11—C12—C13—S2 | -178.2 (4) |
| O5—Ni1—O9—C21 | 173.8 (4) | C16—S2—C13—C12 | -76.2 (4) |
| O9—Ni1—N1—C1 | -45.1 (3) | C16—S2—C13—C14 | 107.0 (4) |
| N4 ⁱ —Ni1—N1—C1 | 128.6 (4) | C12—C13—C14—C15 | 1.2 (8) |
| N3—Ni1—N1—C1 | -74.9 (18) | S2—C13—C14—C15 | 178.0 (4) |
| O2—Ni1—N1—C1 | -138.2 (3) | C11—N3—C15—C14 | -2.3 (8) |
| O5—Ni1—N1—C1 | 35.6 (4) | Ni1—N3—C15—C14 | 179.7 (5) |
| O9—Ni1—N1—C5 | 130.2 (4) | C13—C14—C15—N3 | 0.7 (9) |
| N4 ⁱ —Ni1—N1—C5 | -56.1 (4) | C13—S2—C16—C20 | 163.6 (4) |
| N3—Ni1—N1—C5 | 100.3 (17) | C13—S2—C16—C17 | -18.9 (5) |
| O2—Ni1—N1—C5 | 37.1 (4) | C20—C16—C17—C18 | -1.1 (8) |
| O5—Ni1—N1—C5 | -149.2 (4) | S2—C16—C17—C18 | -178.6 (4) |
| O9—Ni1—N3—C11 | -134.4 (4) | C19—N4—C18—C17 | 1.0 (7) |

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|-----------------------------|-------------|-------------------------------|------------|
| N4 ⁱ —Ni1—N3—C11 | 51.8 (4) | Ni1 ⁱⁱ —N4—C18—C17 | -174.8 (4) |
| N1—Ni1—N3—C11 | -104.5 (17) | C16—C17—C18—N4 | 0.1 (8) |
| O2—Ni1—N3—C11 | -41.5 (4) | C18—N4—C19—C20 | -1.1 (7) |
| O5—Ni1—N3—C11 | 144.8 (4) | Ni1 ⁱⁱ —N4—C19—C20 | 174.8 (4) |
| O9—Ni1—N3—C15 | 43.4 (4) | N4—C19—C20—C16 | 0.2 (8) |
| N4 ⁱ —Ni1—N3—C15 | -130.3 (4) | C17—C16—C20—C19 | 0.9 (8) |
| N1—Ni1—N3—C15 | 73.3 (18) | S2—C16—C20—C19 | 178.6 (4) |
| O2—Ni1—N3—C15 | 136.4 (4) | Ni1—O9—C21—O10 | 0.8 (7) |
| O5—Ni1—N3—C15 | -37.4 (4) | Ni1—O9—C21—C22 | -179.4 (3) |
| C5—N1—C1—C2 | -0.1 (7) | O9—C21—C22—C23 | -0.4 (6) |
| Ni1—N1—C1—C2 | 175.4 (4) | O10—C21—C22—C23 | 179.4 (4) |
| N1—C1—C2—C3 | -0.3 (8) | O9—C21—C22—C27 | -179.2 (4) |
| C1—C2—C3—C4 | 0.8 (7) | O10—C21—C22—C27 | 0.6 (6) |
| C1—C2—C3—S1 | -173.6 (4) | C27—C22—C23—C24 | 1.5 (6) |
| C6—S1—C3—C4 | 26.3 (5) | C21—C22—C23—C24 | -177.4 (4) |
| C6—S1—C3—C2 | -159.8 (4) | C22—C23—C24—C25 | -0.7 (7) |
| C2—C3—C4—C5 | -1.0 (8) | C22—C23—C24—S3 | -179.0 (3) |
| S1—C3—C4—C5 | 172.8 (4) | O7—S3—C24—C25 | -98.3 (5) |
| C1—N1—C5—C4 | -0.1 (8) | O8—S3—C24—C25 | 24.9 (5) |
| Ni1—N1—C5—C4 | -175.5 (4) | O6—S3—C24—C25 | 141.5 (5) |
| C3—C4—C5—N1 | 0.7 (8) | O7—S3—C24—C23 | 80.1 (5) |
| C3—S1—C6—C7 | -123.6 (5) | O8—S3—C24—C23 | -156.8 (5) |
| C3—S1—C6—C10 | 60.5 (5) | O6—S3—C24—C23 | -40.1 (5) |
| C10—C6—C7—C8 | -3.4 (9) | C23—C24—C25—C26 | -0.8 (7) |
| S1—C6—C7—C8 | -179.3 (5) | S3—C24—C25—C26 | 177.6 (4) |
| C9—N2—C8—C7 | 0.4 (12) | C24—C25—C26—C27 | 1.4 (9) |
| C6—C7—C8—N2 | 1.5 (11) | C23—C22—C27—O11 | -179.9 (5) |
| C8—N2—C9—C10 | -0.5 (12) | C21—C22—C27—O11 | -1.0 (7) |
| C7—C6—C10—C9 | 3.3 (9) | C23—C22—C27—C26 | -0.8 (7) |
| S1—C6—C10—C9 | 179.2 (5) | C21—C22—C27—C26 | 178.0 (5) |
| N2—C9—C10—C6 | -1.3 (11) | C25—C26—C27—O11 | 178.5 (5) |
| C15—N3—C11—C12 | 2.1 (7) | C25—C26—C27—C22 | -0.6 (8) |

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-------------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| O11—H11 \cdots O10 | 0.82 | 1.84 | 2.535 (5) | 142 |
| O5—H10W \cdots O3 ⁱⁱⁱ | 0.83 | 1.98 | 2.797 (5) | 170 |
| O5—H9W \cdots O4 ^{iv} | 0.85 | 2.04 | 2.721 (8) | 136 |
| O4—H7W \cdots O8 ^v | 0.83 | 2.30 | 2.713 (9) | 112 |
| O3—H6W \cdots O6 ^{vi} | 0.83 | 2.10 | 2.811 (7) | 143 |
| O3—H5W \cdots O6 ^{vii} | 0.83 | 2.24 | 2.765 (8) | 122 |
| O2—H4W \cdots O10 | 0.83 | 1.95 | 2.690 (5) | 149 |
| O2—H3W \cdots O7 ^{vi} | 0.84 | 1.87 | 2.652 (7) | 155 |
| O1—H1W \cdots O11 ^{viii} | 0.85 | 2.03 | 2.876 (7) | 180 |

supplementary materials

Symmetry codes: (iii) $-x+3/2, y-1/2, -z+3/2$; (iv) $x, y-1, z$; (v) $x-1, y+1, z$; (vi) $-x+3/2, y+1/2, -z+3/2$; (vii) $x-1/2, -y+1/2, z-1/2$; (viii) $x-1, y, z$.

Fig. 1

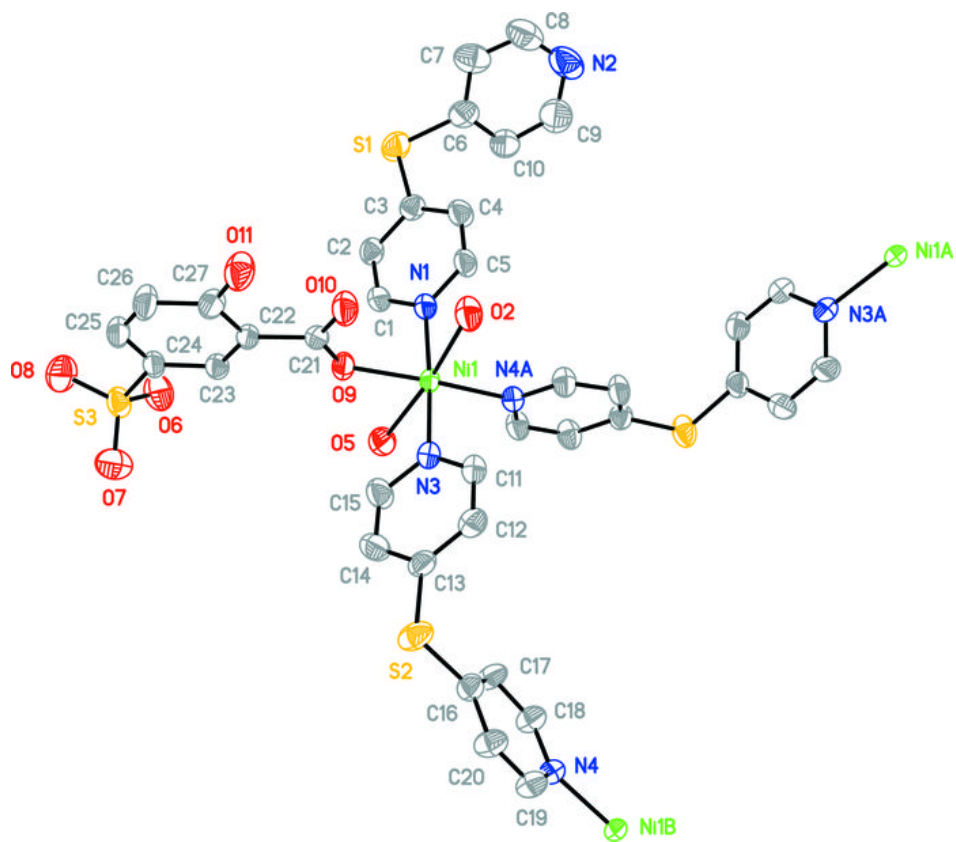


Fig. 2

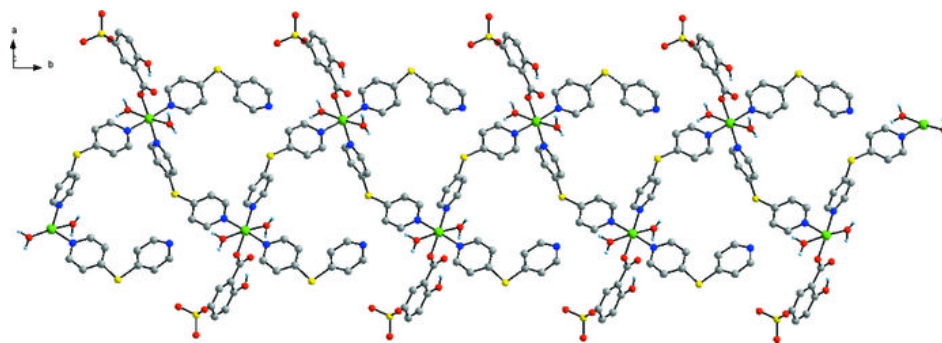


Fig. 3

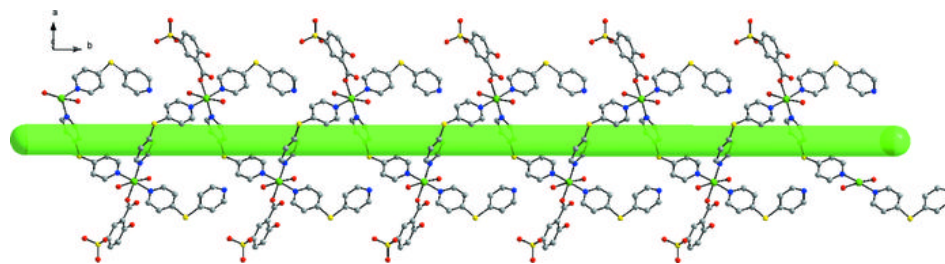


Fig. 4

