

catena-Poly[[triaqua(pyridine- κ N)-nickel(II)]- μ -sulfato- κ^2 O:O']

Yan-Fang Shi, Fu-Xing Li, Bo Geng, Yan-Cheng Liu and Zhen-Feng Chen*

Key Laboratory for the Chemistry and Molecular Engineering of Medicinal Resources (Ministry of Education of China), School of Chemistry & Chemical Engineering, Guangxi Normal University, Guilin 541004, People's Republic of China
Correspondence e-mail: chenzfgxnu@yahoo.com

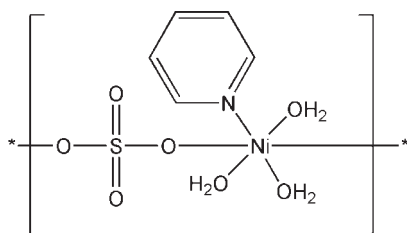
Received 2 November 2009; accepted 19 November 2009

Key indicators: single-crystal X-ray study; $T = 193$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.036; wR factor = 0.110; data-to-parameter ratio = 10.8.

The title compound, $[\text{Ni}(\text{SO}_4)(\text{C}_5\text{H}_5\text{N})(\text{H}_2\text{O})_3]_n$, was synthesized by the hydrothermal reaction of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, pyridine and water. The central Ni^{II} atom is coordinated in a distorted octahedral environment by a pyridine N atom, three aqua O atoms and two O atoms of bridging sulfate anions, yielding a zigzag chain. A three-dimensional network is generated *via* complex hydrogen bonds involving the sulfate and aqua ligands and a pyridine C—H group.

Related literature

For the structures of related nickel(II) complexes, see: Wang *et al.* (2006); Stein *et al.* (2007).



Experimental

Crystal data

$[\text{Ni}(\text{SO}_4)(\text{C}_5\text{H}_5\text{N})(\text{H}_2\text{O})_3]$
 $M_r = 287.92$
Monoclinic, $P2_1/c$
 $a = 11.868$ (3) Å
 $b = 7.5745$ (14) Å
 $c = 11.420$ (3) Å
 $\beta = 110.724$ (4)°

$V = 960.2$ (3) Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 2.26$ mm⁻¹
 $T = 193$ K
0.30 × 0.20 × 0.14 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
 $T_{\text{min}} = 0.465$, $T_{\text{max}} = 0.729$

8854 measured reflections
1746 independent reflections
1641 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.110$
 $S = 1.16$
1746 reflections
161 parameters
6 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.57$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.84$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O5}-\text{H5A}\cdots\text{O4}^{\text{i}}$	0.82 (3)	2.04 (3)	2.849 (3)	170 (4)
$\text{O5}-\text{H5B}\cdots\text{O1}^{\text{ii}}$	0.818 (10)	1.939 (12)	2.753 (3)	173 (4)
$\text{O6}-\text{H6A}\cdots\text{O3}^{\text{i}}$	0.821 (10)	1.949 (12)	2.764 (3)	172 (4)
$\text{O6}-\text{H6B}\cdots\text{O4}$	0.82 (3)	2.15 (3)	2.821 (3)	139 (4)
$\text{O7}-\text{H7A}\cdots\text{O2}^{\text{ii}}$	0.82 (3)	2.00 (3)	2.817 (3)	176 (4)
$\text{O7}-\text{H7B}\cdots\text{O4}^{\text{iii}}$	0.815 (10)	1.94 (2)	2.690 (3)	153 (4)
$\text{C4}-\text{H4}\cdots\text{O3}^{\text{iv}}$	0.95	2.57	3.304 (5)	135

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

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC & Rigaku, 2000); 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*.

The authors thank the National Natural Science Foundation of China (No. 20861002), the 973 Plan of China (2009CB526503), the Natural Science Foundation of Guangxi, China (Nos. 0991003, 0991012Z) and the Open Foundation of the Key Laboratory for the Chemistry and Molecular Engineering of Medicinal Resources (Ministry of Education of China) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2206).

References

- Jacobson, R. (1998). *REQAB*. Private communication to the Rigaku Corporation, Tokyo, Japan.
Rigaku (1999). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC & Rigaku (2000). *CrystalStructure*. Rigaku/MSC, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Stein, I., Speldrich, M., Schilder, H., Lueken, H. & Ruschewitz, U. (2007). *Z. Anorg. Allg. Chem.* **633**, 1382–1390.
Wang, Y., Su, Z.-M., Hao, X.-R., Shao, K.-Z. & Zhao, Y.-H. (2006). *Acta Cryst.* **E62**, m322–m324.

supplementary materials

Acta Cryst. (2009). E65, m1665 [doi:10.1107/S1600536809049605]

***catena*-Poly[[triaqua(pyridine- κ N)nickel(II)]- μ -sulfato- κ^2 O:O']**

Y.-F. Shi, F.-X. Li, B. Geng, Y.-C. Liu and Z.-F. Chen

Comment

The asymmetric unit contains one independent Ni atom, which is octahedrally coordinated by two sulfato anions, three aqua ligands and one pyridine molecule. The bond lengths and angles involving Ni—O(aqua), Ni—N are similar to those of other nickel-carboxylate coordination polymers with pyridine (Wang *et al.*, 2006; Stein *et al.*, 2007), with the Ni center displaying the typical distorted octahedral coordination, which can be viewed from the angles of N1—Ni1—O1 177.81 (10)°, N1—Ni1—O7 91.13 (11)°, O1—Ni1—O6 92.13 (9)°, O5—Ni1—O6 92.91 (10)° (Fig. 1). The SO₄²⁻ dianion acts as a μ_2 bridging ligand, linking two adjacent metal ions and generating a one-dimensional zigzag chain (Fig. 2). The aqua ligands, sulfato groups and C—H of pyridine form extensive hydrogen-bonding interactions (Table 1), resulting in a three-dimensional network (Fig. 3).

Experimental

Samples of NiSO₄·6H₂O (0.1 mmol) and pyridine (0.1 mmol) were placed in a thick-walled Pyrex tube (*ca* 20 cm long). After addition of H₂O (1 ml), the tube was frozen with liquid nitrogen, evacuated under vacuum and sealed with a torch. The tube was heated at 110°C for 2 days and then was slowly cooled down to room temperature, and light-green block-shaped crystals were obtained. Yield: 35%.

Refinement

The H atoms bonded to C atoms were positioned geometrically and refined using a riding model with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ (C—H = 0.95 Å). Water H positions were located in an electron-density difference map and refined freely.

Figures

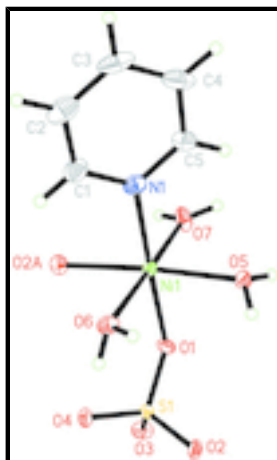


Fig. 1. The molecular structure showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

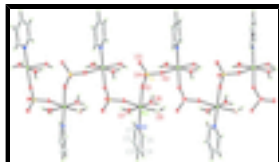


Fig. 2. A view of the one-dimensional chain structure that propagates along the *b* axis.

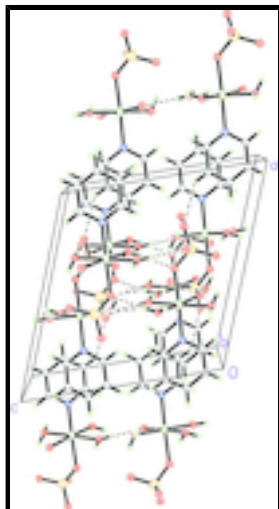


Fig. 3. A packing diagram viewed approximately down the *b* axis.

catena-poly[[triaqua(pyridine- κ N)nickel(II)]- μ -sulfato- κ^2 O:O']

Crystal data

[Ni(SO₄)(C₅H₅N)(H₂O)₃]

$M_r = 287.92$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 11.868$ (3) Å

$b = 7.5745$ (14) Å

$c = 11.420$ (3) Å

$\beta = 110.724$ (4)°

$V = 960.2$ (3) Å³

$Z = 4$

$F(000) = 592$

$D_x = 1.992$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å

Cell parameters from 3550 reflections

$\theta = 3.1$ – 25.3 °

$\mu = 2.26$ mm⁻¹

$T = 193$ K

Block, light-green

$0.30 \times 0.20 \times 0.14$ mm

Data collection

Rigaku Mercury CCD
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: 7.31 pixels mm⁻¹
 ω scans

Absorption correction: multi-scan
(REQAB; Jacobson, 1998)

$T_{\min} = 0.465$, $T_{\max} = 0.729$

8854 measured reflections

1746 independent reflections

1641 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\max} = 25.3$ °, $\theta_{\min} = 3.3$ °

$h = -14 \rightarrow 13$

$k = -9 \rightarrow 9$

$l = -13 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.036$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.110$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.16$	$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2 + 0.4274P]$
1746 reflections	where $P = (F_o^2 + 2F_c^2)/3$
161 parameters	$(\Delta/\sigma)_{\max} = 0.001$
6 restraints	$\Delta\rho_{\max} = 0.57 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -0.84 \text{ e } \text{\AA}^{-3}$

Special details

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

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.65136 (3)	0.59664 (5)	0.21049 (4)	0.0112 (2)
S1	0.37812 (7)	0.51034 (10)	0.20733 (7)	0.0111 (2)
O1	0.4632 (2)	0.5757 (3)	0.1473 (2)	0.0144 (5)
O2	0.3600 (2)	0.3178 (3)	0.1818 (2)	0.0148 (5)
O3	0.2651 (2)	0.6067 (3)	0.1535 (2)	0.0185 (6)
O4	0.4322 (2)	0.5392 (3)	0.3441 (2)	0.0163 (5)
O5	0.6504 (2)	0.3741 (3)	0.1062 (2)	0.0139 (5)
H5A	0.626 (3)	0.283 (3)	0.128 (3)	0.017 (10)*
H5B	0.619 (4)	0.381 (6)	0.0300 (11)	0.033 (13)*
O6	0.6708 (2)	0.4525 (3)	0.3691 (2)	0.0165 (5)
H6A	0.687 (4)	0.3472 (19)	0.367 (4)	0.037 (13)*
H6B	0.611 (2)	0.435 (5)	0.387 (4)	0.028 (12)*
O7	0.6283 (2)	0.7484 (3)	0.0563 (2)	0.0212 (6)
H7A	0.632 (4)	0.724 (5)	-0.0120 (19)	0.034 (12)*
H7B	0.609 (4)	0.850 (2)	0.064 (4)	0.031 (12)*
N1	0.8352 (3)	0.6199 (4)	0.2656 (3)	0.0179 (7)
C1	0.9028 (3)	0.6672 (5)	0.3822 (3)	0.0247 (8)

supplementary materials

H1	0.8637	0.6950	0.4394	0.030*
C2	1.0269 (3)	0.6774 (6)	0.4232 (4)	0.0325 (9)
H2	1.0718	0.7112	0.5069	0.039*
C3	1.0846 (3)	0.6379 (6)	0.3407 (4)	0.0345 (10)
H3	1.1699	0.6441	0.3664	0.041*
C4	1.0165 (4)	0.5894 (5)	0.2207 (5)	0.0331 (10)
H4	1.0539	0.5612	0.1620	0.040*
C5	0.8929 (3)	0.5822 (5)	0.1867 (4)	0.0255 (9)
H5C	0.8464	0.5490	0.1034	0.031*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0115 (3)	0.0107 (3)	0.0109 (3)	-0.00030 (14)	0.0035 (2)	0.00009 (15)
S1	0.0122 (4)	0.0103 (4)	0.0114 (4)	-0.0005 (3)	0.0050 (3)	-0.0009 (3)
O1	0.0123 (12)	0.0198 (13)	0.0129 (12)	-0.0013 (9)	0.0066 (10)	0.0014 (9)
O2	0.0222 (13)	0.0097 (12)	0.0127 (11)	-0.0012 (9)	0.0064 (10)	-0.0036 (10)
O3	0.0172 (13)	0.0147 (13)	0.0259 (14)	0.0034 (9)	0.0103 (11)	0.0035 (10)
O4	0.0226 (13)	0.0160 (12)	0.0113 (12)	-0.0048 (10)	0.0071 (10)	-0.0043 (10)
O5	0.0188 (13)	0.0144 (12)	0.0081 (12)	-0.0028 (10)	0.0042 (10)	0.0010 (10)
O6	0.0158 (13)	0.0135 (13)	0.0198 (13)	-0.0005 (11)	0.0058 (10)	0.0022 (11)
O7	0.0361 (15)	0.0135 (14)	0.0175 (14)	0.0053 (11)	0.0139 (12)	0.0009 (11)
N1	0.0154 (15)	0.0150 (15)	0.0223 (16)	-0.0002 (11)	0.0053 (12)	0.0016 (12)
C1	0.0198 (19)	0.029 (2)	0.0217 (19)	-0.0036 (16)	0.0025 (15)	-0.0012 (17)
C2	0.020 (2)	0.035 (2)	0.033 (2)	-0.0033 (17)	-0.0025 (17)	0.002 (2)
C3	0.0139 (19)	0.028 (2)	0.057 (3)	-0.0045 (16)	0.007 (2)	0.004 (2)
C4	0.024 (2)	0.028 (2)	0.056 (3)	-0.0004 (17)	0.025 (2)	0.002 (2)
C5	0.0198 (19)	0.029 (2)	0.030 (2)	-0.0035 (15)	0.0124 (17)	-0.0038 (17)

Geometric parameters (\AA , $^\circ$)

Ni1—O7	2.039 (2)	O6—H6B	0.82 (3)
Ni1—N1	2.053 (3)	O7—H7A	0.82 (3)
Ni1—O6	2.056 (2)	O7—H7B	0.815 (10)
Ni1—O5	2.062 (2)	N1—C1	1.337 (5)
Ni1—O1	2.096 (2)	N1—C5	1.342 (5)
Ni1—O2 ⁱ	2.110 (2)	C1—C2	1.380 (5)
S1—O3	1.458 (2)	C1—H1	0.9500
S1—O4	1.479 (2)	C2—C3	1.380 (6)
S1—O2	1.488 (2)	C2—H2	0.9500
S1—O1	1.491 (2)	C3—C4	1.372 (6)
O2—Ni1 ⁱⁱ	2.110 (2)	C3—H3	0.9500
O5—H5A	0.82 (3)	C4—C5	1.379 (6)
O5—H5B	0.818 (10)	C4—H4	0.9500
O6—H6A	0.821 (10)	C5—H5C	0.9500
O7—Ni1—N1	91.13 (11)	H5A—O5—H5B	108 (4)
O7—Ni1—O6	177.36 (10)	Ni1—O6—H6A	117 (3)
N1—Ni1—O6	89.97 (11)	Ni1—O6—H6B	118 (3)

O7—Ni1—O5	89.45 (9)	H6A—O6—H6B	94 (4)
N1—Ni1—O5	92.04 (11)	Ni1—O7—H7A	131 (3)
O6—Ni1—O5	92.91 (10)	Ni1—O7—H7B	113 (3)
O7—Ni1—O1	86.80 (10)	H7A—O7—H7B	115 (4)
N1—Ni1—O1	177.81 (10)	C1—N1—C5	117.1 (3)
O6—Ni1—O1	92.13 (9)	C1—N1—Ni1	121.8 (2)
O5—Ni1—O1	87.23 (9)	C5—N1—Ni1	121.1 (3)
O7—Ni1—O2 ⁱ	92.22 (9)	N1—C1—C2	123.1 (4)
N1—Ni1—O2 ⁱ	91.89 (10)	N1—C1—H1	118.5
O6—Ni1—O2 ⁱ	85.33 (9)	C2—C1—H1	118.5
O5—Ni1—O2 ⁱ	175.69 (9)	C3—C2—C1	118.9 (4)
O1—Ni1—O2 ⁱ	88.90 (9)	C3—C2—H2	120.5
O3—S1—O4	111.21 (14)	C1—C2—H2	120.5
O3—S1—O2	111.13 (13)	C4—C3—C2	118.8 (4)
O4—S1—O2	109.26 (13)	C4—C3—H3	120.6
O3—S1—O1	108.13 (14)	C2—C3—H3	120.6
O4—S1—O1	108.95 (13)	C3—C4—C5	118.9 (4)
O2—S1—O1	108.08 (12)	C3—C4—H4	120.6
S1—O1—Ni1	132.76 (14)	C5—C4—H4	120.6
S1—O2—Ni1 ⁱⁱ	134.32 (13)	N1—C5—C4	123.2 (4)
Ni1—O5—H5A	116 (3)	N1—C5—H5C	118.4
Ni1—O5—H5B	118 (3)	C4—C5—H5C	118.4
O3—S1—O1—Ni1	150.13 (18)	O2 ⁱ —Ni1—N1—C1	37.9 (3)
O4—S1—O1—Ni1	29.1 (2)	O7—Ni1—N1—C5	-52.1 (3)
O2—S1—O1—Ni1	-89.5 (2)	O6—Ni1—N1—C5	130.3 (3)
O7—Ni1—O1—S1	-167.8 (2)	O5—Ni1—N1—C5	37.4 (3)
O6—Ni1—O1—S1	9.8 (2)	O2 ⁱ —Ni1—N1—C5	-144.4 (3)
O5—Ni1—O1—S1	102.64 (19)	C5—N1—C1—C2	-0.3 (5)
O2 ⁱ —Ni1—O1—S1	-75.47 (19)	Ni1—N1—C1—C2	177.5 (3)
O3—S1—O2—Ni1 ⁱⁱ	-103.2 (2)	N1—C1—C2—C3	0.2 (6)
O4—S1—O2—Ni1 ⁱⁱ	19.9 (2)	C1—C2—C3—C4	-0.1 (6)
O1—S1—O2—Ni1 ⁱⁱ	138.29 (18)	C2—C3—C4—C5	0.1 (6)
O7—Ni1—N1—C1	130.2 (3)	C1—N1—C5—C4	0.3 (5)
O6—Ni1—N1—C1	-47.4 (3)	Ni1—N1—C5—C4	-177.5 (3)
O5—Ni1—N1—C1	-140.3 (3)	C3—C4—C5—N1	-0.3 (6)

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

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>
O5—H5A \cdots O4 ⁱⁱ	0.82 (3)	2.04 (3)	2.849 (3)	170 (4)
O5—H5A \cdots S1 ⁱⁱ	0.82 (3)	2.81 (2)	3.571 (3)	157 (3)
O5—H5B \cdots O1 ⁱⁱⁱ	0.82 (1)	1.94 (1)	2.753 (3)	173 (4)
O5—H5B \cdots S1 ⁱⁱⁱ	0.82 (1)	2.85 (2)	3.584 (3)	151 (4)
O6—H6A \cdots O3 ⁱⁱ	0.82 (1)	1.95 (1)	2.764 (3)	172 (4)

supplementary materials

O6—H6A…S1 ⁱⁱ	0.82 (1)	2.71 (2)	3.458 (3)	152 (4)
O6—H6B…O4	0.82 (3)	2.15 (3)	2.821 (3)	139 (4)
O6—H6B…S1	0.82 (3)	2.85 (4)	3.336 (3)	120 (3)
O7—H7A…O2 ⁱⁱⁱ	0.82 (3)	2.00 (3)	2.817 (3)	176 (4)
O7—H7A…S1 ⁱⁱⁱ	0.82 (3)	2.82 (2)	3.571 (3)	154 (4)
O7—H7B…O4 ⁱ	0.82 (1)	1.94 (2)	2.690 (3)	153 (4)
O7—H7B…S1 ⁱ	0.82 (1)	2.84 (4)	3.372 (3)	125 (4)
C4—H4…O3 ^{iv}	0.95	2.57	3.304 (5)	135.

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

Fig. 1

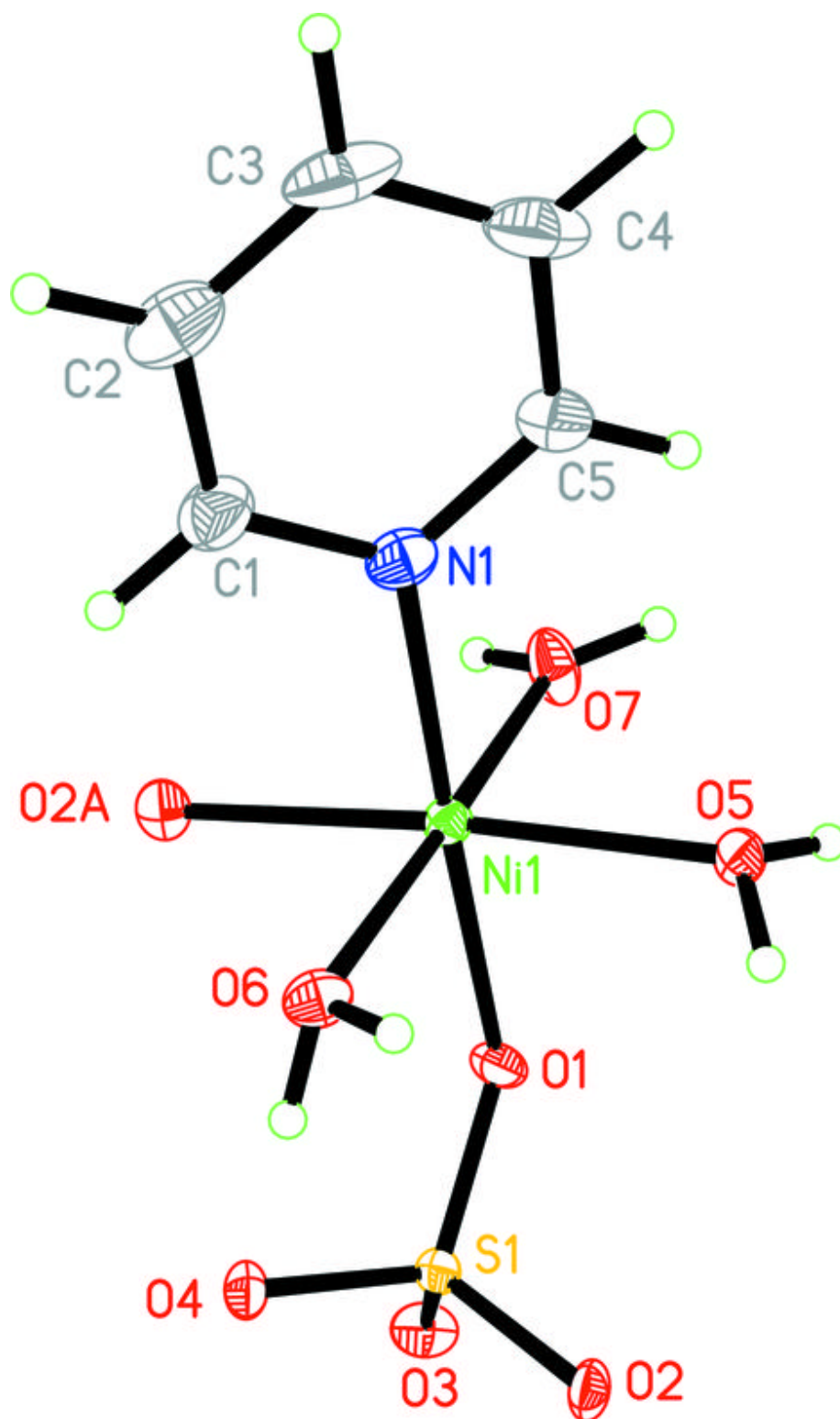


Fig. 2

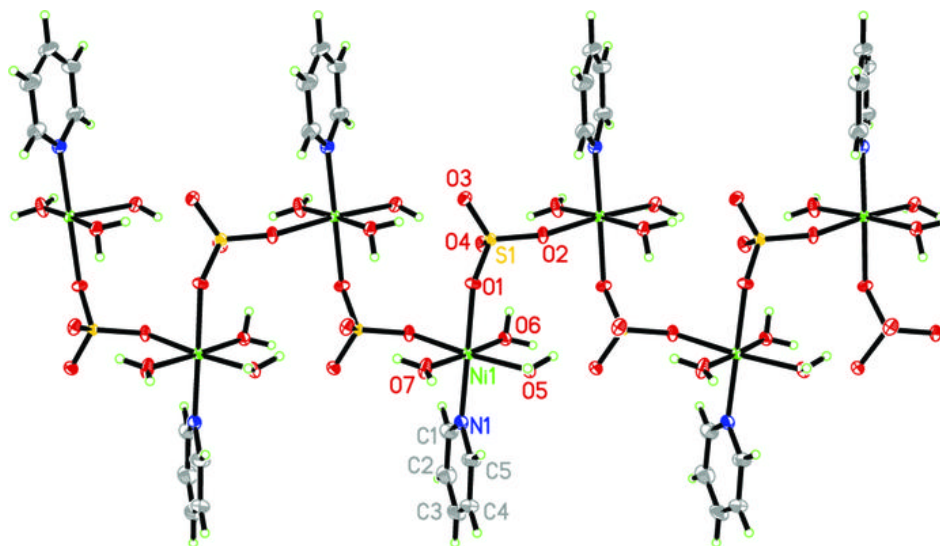


Fig. 3

