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Bis(triethanolamine)nickel(II) sulfate

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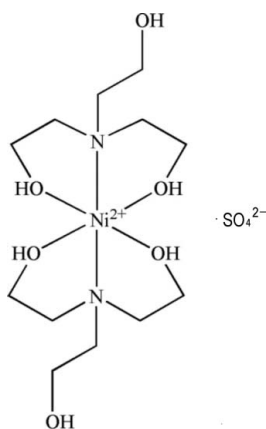
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.030; wR factor = 0.085; data-to-parameter ratio = 17.0.

The title compound, $[\text{Ni}(\text{C}_6\text{H}_{15}\text{NO}_3)_2]\text{SO}_4$, contains two triethanolamine (TEA) ligands bound to an Ni^{2+} metal centre, which lies on a crystallographic inversion centre, and one sulfate anion located on a twofold rotation axis such that the asymmetric unit contains one-half molecule of the cation and of the anion. The triethanolamine ligands coordinate *via* each axial N atom and two of the three O atoms, while the third arm of the ligand has the hydroxyl group pointing away from the metal centre. The sulfate anions are hydrogen bonded to the coordinated hydroxyl groups and also to the free arm, forming a two-dimensional supramolecular hydrogen-bonded network expanding parallel to (010).

Related literature

For background to metal-ion-containing supramolecular compounds, see: Venkataraman *et al.* (1995); Kepert & Rosseinsky (1999); Fujita *et al.* (1994). For magnetic materials, see: Kahn (1993). For other TEA compounds, see: Krabbes *et al.* (2000); Topcu *et al.* (2001); Ucar *et al.* (2004); Haukka *et al.* (2005). For similar structures, see: İçbudak *et al.* (1995); Yeşilil *et al.* (2004).



Experimental

Crystal data

$[\text{Ni}(\text{C}_6\text{H}_{15}\text{NO}_3)_2]\text{SO}_4$
 $M_r = 453.15$
 Monoclinic, $C2/c$
 $a = 10.316$ (2) Å
 $b = 11.234$ (2) Å
 $c = 15.498$ (3) Å
 $\beta = 90.04$ (3)°

$V = 1796.0$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.25$ mm⁻¹
 $T = 293$ K
 $0.41 \times 0.21 \times 0.11$ mm

Data collection

Siemens SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.741$, $T_{\max} = 0.879$

8633 measured reflections
 2042 independent reflections
 1905 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.029$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.085$
 $S = 1.02$
 2042 reflections
 120 parameters

3 restraints
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.66$ e Å⁻³
 $\Delta\rho_{\min} = -0.65$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{O1}-\text{H1C}\cdots\text{O5}^i$ | 0.82 | 2.19 | 2.663 (2) | 117 |
| $\text{O2}-\text{H2C}\cdots\text{O4}^{ii}$ | 0.82 | 2.28 | 2.6227 (19) | 106 |
| $\text{O3}-\text{H3C}\cdots\text{O5}$ | 0.82 | 2.00 | 2.818 (2) | 174 |
| $\text{C3}-\text{H3B}\cdots\text{O3}$ | 0.97 | 2.26 | 3.055 (3) | 139 |

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

Data collection: SMART (Siemens, 1994); cell refinement: SAINT (Siemens, 1994); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2167).

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supplementary materials

Acta Cryst. (2009). E65, m810-m811 [doi:10.1107/S1600536809023046]

Bis(triethanolamine)nickel(II) sulfate

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Comment

Many workers from a variety of scientific disciplines are interested in the crystal design and engineering of multidimensional arrays and networks containing metal ions as nodes. Metal-ion containing supramolecular structures can be used as zeolite-like materials (Venkataraman *et al.*, 1995; Kepert & Rosseinsky, 1999), catalysts (Fujita *et al.*, 1994) or magnetic materials (Kahn, 1993). Triethanolamine (TEA) is a good potential ligand to the incorporation of metals into metal-ion containing supramolecular framework, and many compounds constructed from TEA have been reported in the last decade (Krabbes *et al.*, 2000; Topcu *et al.*, 2001; Ucar *et al.*, 2004; Haukka *et al.*, 2005). In this work we employed TEA and NiSO₄ to produce a novel complex, [Ni(C₆H₁₅NO₃)₂]SO₄(I).

A view of the title compound and its numbering scheme are shown in Fig. 1. The crystal structure consists of a complex cation and one sulfate anion. In the cation, the Ni^{II} ion lies on a centre of symmetry, sandwiched by two bulky TEA ligands. Each TEA acts as a tridentate ligand through two of the three hydroxyl O atoms and the amine N atom, resulting in a six-coordinate Ni^{II} ion similar to those observed for the Ni complex of TEA with chloride (İçbudak *et al.*, 1995), saccharine (Topcu *et al.*, 2001), acetate (Krabbes *et al.*, 2000) and squarate (Yeşilel *et al.*, 2004).

The coordination geometry around the Ni^{II} ion is that of a distorted octahedron. The hydroxyl O atoms of two TEA ligands form the equatorial plane of the octahedral geometry, while atoms N1 and N1ⁱ are placed in axial positions (symmetry code as in Fig. 1). In the complex, Ni—O distances are in the range 2.0762 (13)–2.0768 (12) Å and the Ni—N distance is 2.1072 (16) Å, while the bond angles at Ni range from 82.30 (5)° to 97.70 (6)°.

In the crystal structure, classical intermolecular O—H...O hydrogen bonds are observed (Table 1), which link the cations and one sulfate anion into a two-dimensional hydrogen-bonded network and stabilize the crystal packing (Fig. 2). Each cation is bonded to four SO₄²⁻ anions, which in turn link four cations through O—H...O hydrogen bonding interactions to form a 2-D hydrogen bonding network. In addition, there is extensive hydrogen bonding between the —CH₂ and the uncoordinated hydroxyl O atoms, with C...O interatomic distances of 3.055 (3) Å.

Experimental

NiSO₄·7H₂O (0.5257 g, 2 mmol) was dissolved in 10 ml water and the pH was adjusted to 7 with triethanolamine. Blue crystals were separated from the filtered solution at room temperature over several days.

Refinement

All H atoms bound to carbon were refined using a riding model with C—H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Three hydroxy H atoms were located in a difference map and included with O—H distance constraints of 0.82 Å and with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures



Fig. 1. View of the structure showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level; H-atoms are shown as small spheres of arbitrary radius. (Symmetry codes: i: $-x+1/2, -y+3/2, -z+2$; ii: $-x+2, y, -z+3/2$)

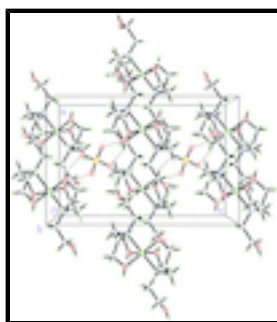


Fig. 2. View of the 2-D hydrogen-bonded network in the packing of the title compound. The packing is viewed along the b axis; O—H \cdots O and C—H \cdots O interactions are shown as dashed lines.

Bis(triethanolamine)nickel(II) sulfate

Crystal data

$[\text{Ni}(\text{C}_6\text{H}_{15}\text{NO}_3)_2]\text{SO}_4$

$M_r = 453.15$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$a = 10.316\ (2)\ \text{\AA}$

$b = 11.234\ (2)\ \text{\AA}$

$c = 15.498\ (3)\ \text{\AA}$

$\beta = 90.04\ (3)^\circ$

$V = 1796.0\ (6)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 960$

$D_x = 1.676\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 8633 reflections

$\theta = 3.6\text{--}27.5^\circ$

$\mu = 1.25\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, blue

$0.41 \times 0.21 \times 0.11\ \text{mm}$

Data collection

Siemens SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293\ \text{K}$

ω scans

Absorption correction: multi-scan

2042 independent reflections

1905 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$

$\theta_{\text{max}} = 27.5^\circ$

$\theta_{\text{min}} = 3.6^\circ$

$h = -13 \rightarrow 13$

(SADABS; Sheldrick, 1996)

$T_{\min} = 0.741$, $T_{\max} = 0.879$

8633 measured reflections

$k = -14 \rightarrow 14$

$l = -20 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.085$

$S = 1.02$

2042 reflections

120 parameters

3 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.053P)^2 + 2.3273P]$$

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

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

$\Delta\rho_{\max} = 0.66 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.65 \text{ e } \text{\AA}^{-3}$

Extinction correction: none

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 | 0.2500 | 0.7500 | 1.0000 | 0.01765 (12) |
| N1 | 0.40821 (14) | 0.71956 (14) | 0.91696 (9) | 0.0218 (3) |
| O1 | 0.15355 (12) | 0.64708 (12) | 0.90906 (8) | 0.0255 (3) |
| H1C | 0.0790 | 0.6254 | 0.8997 | 0.038* |
| O2 | 0.22752 (12) | 0.90445 (11) | 0.92788 (8) | 0.0242 (3) |
| H2C | 0.1762 | 0.9553 | 0.9113 | 0.036* |
| O3 | 0.70194 (16) | 0.79446 (17) | 0.87607 (13) | 0.0512 (5) |
| H3C | 0.7766 | 0.7847 | 0.8594 | 0.077* |
| O4 | 0.89097 (13) | 0.89514 (14) | 0.72175 (9) | 0.0351 (3) |
| O5 | 0.95708 (14) | 0.74424 (13) | 0.82252 (10) | 0.0337 (3) |
| C1 | 0.37671 (18) | 0.60337 (18) | 0.87593 (13) | 0.0303 (4) |
| H1A | 0.3897 | 0.5400 | 0.9176 | 0.036* |
| H1B | 0.4354 | 0.5898 | 0.8281 | 0.036* |
| C2 | 0.23847 (19) | 0.59917 (19) | 0.84339 (12) | 0.0317 (4) |

supplementary materials

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|-----|--------------|--------------|--------------|--------------|
| H2A | 0.2307 | 0.6456 | 0.7909 | 0.038* |
| H2B | 0.2143 | 0.5176 | 0.8305 | 0.038* |
| C3 | 0.40912 (18) | 0.81992 (18) | 0.85289 (12) | 0.0293 (4) |
| H3A | 0.3573 | 0.7986 | 0.8030 | 0.035* |
| H3B | 0.4971 | 0.8347 | 0.8337 | 0.035* |
| C4 | 0.35485 (18) | 0.93041 (17) | 0.89416 (12) | 0.0288 (4) |
| H4A | 0.4114 | 0.9565 | 0.9405 | 0.035* |
| H4B | 0.3493 | 0.9938 | 0.8518 | 0.035* |
| C5 | 0.53313 (17) | 0.71242 (18) | 0.96482 (12) | 0.0265 (4) |
| H5A | 0.5251 | 0.6503 | 1.0080 | 0.032* |
| H5B | 0.5454 | 0.7870 | 0.9953 | 0.032* |
| C6 | 0.65530 (18) | 0.68791 (18) | 0.91236 (14) | 0.0321 (4) |
| H6A | 0.7212 | 0.6534 | 0.9493 | 0.039* |
| H6B | 0.6360 | 0.6314 | 0.8668 | 0.039* |
| S1 | 1.0000 | 0.82070 (6) | 0.7500 | 0.02289 (15) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| Ni1 | 0.01652 (17) | 0.02186 (18) | 0.01456 (17) | 0.00161 (10) | 0.00033 (11) | 0.00006 (10) |
| N1 | 0.0191 (7) | 0.0284 (7) | 0.0179 (7) | 0.0031 (6) | 0.0008 (5) | 0.0003 (6) |
| O1 | 0.0212 (6) | 0.0335 (7) | 0.0218 (6) | -0.0002 (5) | -0.0017 (5) | -0.0050 (5) |
| O2 | 0.0245 (6) | 0.0257 (6) | 0.0225 (6) | 0.0044 (5) | -0.0008 (5) | 0.0038 (5) |
| O3 | 0.0272 (8) | 0.0572 (10) | 0.0693 (12) | 0.0037 (7) | 0.0176 (8) | 0.0185 (9) |
| O4 | 0.0287 (7) | 0.0462 (8) | 0.0303 (7) | 0.0102 (6) | -0.0100 (6) | -0.0027 (6) |
| O5 | 0.0230 (7) | 0.0524 (9) | 0.0258 (7) | -0.0012 (5) | 0.0023 (6) | 0.0106 (6) |
| C1 | 0.0275 (9) | 0.0335 (10) | 0.0299 (9) | 0.0064 (7) | 0.0008 (7) | -0.0095 (8) |
| C2 | 0.0300 (10) | 0.0393 (10) | 0.0259 (9) | 0.0030 (8) | -0.0013 (7) | -0.0127 (8) |
| C3 | 0.0249 (9) | 0.0428 (11) | 0.0201 (8) | 0.0031 (7) | 0.0028 (7) | 0.0069 (7) |
| C4 | 0.0265 (9) | 0.0304 (9) | 0.0294 (9) | -0.0040 (7) | -0.0016 (7) | 0.0081 (7) |
| C5 | 0.0208 (8) | 0.0364 (9) | 0.0223 (8) | 0.0031 (7) | -0.0005 (7) | 0.0024 (7) |
| C6 | 0.0221 (9) | 0.0359 (10) | 0.0383 (10) | 0.0063 (7) | 0.0015 (7) | -0.0019 (8) |
| S1 | 0.0172 (3) | 0.0350 (3) | 0.0165 (3) | 0.000 | -0.0021 (2) | 0.000 |

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

| | | | |
|---------------------|-------------|--------|-----------|
| Ni1—O1 ⁱ | 2.0762 (13) | C1—C2 | 1.513 (3) |
| Ni1—O1 | 2.0762 (13) | C1—H1A | 0.9700 |
| Ni1—O2 ⁱ | 2.0768 (12) | C1—H1B | 0.9700 |
| Ni1—O2 | 2.0768 (12) | C2—H2A | 0.9700 |
| Ni1—N1 | 2.1072 (16) | C2—H2B | 0.9700 |
| Ni1—N1 ⁱ | 2.1072 (16) | C3—C4 | 1.505 (3) |
| N1—C1 | 1.488 (2) | C3—H3A | 0.9700 |
| N1—C5 | 1.489 (2) | C3—H3B | 0.9700 |
| N1—C3 | 1.502 (2) | C4—H4A | 0.9700 |
| O1—C2 | 1.447 (2) | C4—H4B | 0.9700 |
| O1—H1C | 0.8200 | C5—C6 | 1.525 (3) |
| O2—C4 | 1.444 (2) | C5—H5A | 0.9700 |

| | | | |
|--------------------------------------|--------------|---------------------------------------|--------------|
| O2—H2C | 0.8200 | C5—H5B | 0.9700 |
| O3—C6 | 1.407 (3) | C6—H6A | 0.9700 |
| O3—H3C | 0.8200 | C6—H6B | 0.9700 |
| O4—S1 | 1.4681 (14) | S1—O4 ⁱⁱ | 1.4681 (14) |
| O5—S1 | 1.4825 (14) | S1—O5 ⁱⁱ | 1.4825 (14) |
| O1 ⁱ —Ni1—O1 | 180.0 | O1—C2—H2A | 109.9 |
| O1 ⁱ —Ni1—O2 ⁱ | 92.68 (5) | C1—C2—H2A | 109.9 |
| O1—Ni1—O2 ⁱ | 87.32 (5) | O1—C2—H2B | 109.9 |
| O1 ⁱ —Ni1—O2 | 87.32 (5) | C1—C2—H2B | 109.9 |
| O1—Ni1—O2 | 92.68 (5) | H2A—C2—H2B | 108.3 |
| O2 ⁱ —Ni1—O2 | 180.0 | N1—C3—C4 | 109.61 (14) |
| O1 ⁱ —Ni1—N1 | 97.70 (6) | N1—C3—H3A | 109.7 |
| O1—Ni1—N1 | 82.30 (5) | C4—C3—H3A | 109.7 |
| O2 ⁱ —Ni1—N1 | 96.14 (5) | N1—C3—H3B | 109.7 |
| O2—Ni1—N1 | 83.86 (6) | C4—C3—H3B | 109.7 |
| O1 ⁱ —Ni1—N1 ⁱ | 82.30 (5) | H3A—C3—H3B | 108.2 |
| O1—Ni1—N1 ⁱ | 97.70 (6) | O2—C4—C3 | 109.04 (15) |
| O2 ⁱ —Ni1—N1 ⁱ | 83.86 (6) | O2—C4—H4A | 109.9 |
| O2—Ni1—N1 ⁱ | 96.14 (5) | C3—C4—H4A | 109.9 |
| N1—Ni1—N1 ⁱ | 180.0 | O2—C4—H4B | 109.9 |
| C1—N1—C5 | 110.76 (15) | C3—C4—H4B | 109.9 |
| C1—N1—C3 | 112.18 (14) | H4A—C4—H4B | 108.3 |
| C5—N1—C3 | 111.34 (15) | N1—C5—C6 | 117.35 (15) |
| C1—N1—Ni1 | 103.56 (11) | N1—C5—H5A | 108.0 |
| C5—N1—Ni1 | 112.02 (11) | C6—C5—H5A | 108.0 |
| C3—N1—Ni1 | 106.69 (11) | N1—C5—H5B | 108.0 |
| C2—O1—Ni1 | 113.23 (10) | C6—C5—H5B | 108.0 |
| C2—O1—H1C | 109.5 | H5A—C5—H5B | 107.2 |
| Ni1—O1—H1C | 137.3 | O3—C6—C5 | 110.01 (16) |
| C4—O2—Ni1 | 105.21 (10) | O3—C6—H6A | 109.7 |
| C4—O2—H2C | 109.5 | C5—C6—H6A | 109.7 |
| Ni1—O2—H2C | 145.3 | O3—C6—H6B | 109.7 |
| C6—O3—H3C | 109.5 | C5—C6—H6B | 109.7 |
| N1—C1—C2 | 112.04 (15) | H6A—C6—H6B | 108.2 |
| N1—C1—H1A | 109.2 | O4 ⁱⁱ —S1—O4 | 110.56 (13) |
| C2—C1—H1A | 109.2 | O4 ⁱⁱ —S1—O5 | 109.46 (8) |
| N1—C1—H1B | 109.2 | O4—S1—O5 | 109.09 (9) |
| C2—C1—H1B | 109.2 | O4 ⁱⁱ —S1—O5 ⁱⁱ | 109.09 (9) |
| H1A—C1—H1B | 107.9 | O4—S1—O5 ⁱⁱ | 109.46 (8) |
| O1—C2—C1 | 108.96 (15) | O5—S1—O5 ⁱⁱ | 109.18 (13) |
| O1 ⁱ —Ni1—N1—C1 | 152.13 (11) | O1 ⁱ —Ni1—O2—C4 | 72.48 (11) |
| O1—Ni1—N1—C1 | -27.87 (11) | O1—Ni1—O2—C4 | -107.52 (11) |
| O2 ⁱ —Ni1—N1—C1 | 58.58 (11) | O2 ⁱ —Ni1—O2—C4 | -86 (100) |
| O2—Ni1—N1—C1 | -121.42 (11) | N1—Ni1—O2—C4 | -25.56 (11) |

supplementary materials

| | | | |
|----------------------------|--------------|----------------------------|-------------|
| N1 ⁱ —Ni1—N1—C1 | -20 (100) | N1 ⁱ —Ni1—O2—C4 | 154.44 (11) |
| O1 ⁱ —Ni1—N1—C5 | 32.74 (13) | C5—N1—C1—C2 | 167.69 (16) |
| O1—Ni1—N1—C5 | -147.26 (13) | C3—N1—C1—C2 | -67.2 (2) |
| O2 ⁱ —Ni1—N1—C5 | -60.81 (13) | Ni1—N1—C1—C2 | 47.43 (17) |
| O2—Ni1—N1—C5 | 119.19 (13) | Ni1—O1—C2—C1 | 18.77 (19) |
| N1 ⁱ —Ni1—N1—C5 | -139 (100) | N1—C1—C2—O1 | -45.3 (2) |
| O1 ⁱ —Ni1—N1—C3 | -89.34 (12) | C1—N1—C3—C4 | 143.10 (16) |
| O1—Ni1—N1—C3 | 90.66 (12) | C5—N1—C3—C4 | -92.14 (18) |
| O2 ⁱ —Ni1—N1—C3 | 177.11 (11) | Ni1—N1—C3—C4 | 30.37 (17) |
| O2—Ni1—N1—C3 | -2.89 (11) | Ni1—O2—C4—C3 | 49.91 (15) |
| N1 ⁱ —Ni1—N1—C3 | 98 (100) | N1—C3—C4—O2 | -55.33 (19) |
| O1 ⁱ —Ni1—O1—C2 | 29 (100) | C1—N1—C5—C6 | 63.6 (2) |
| O2 ⁱ —Ni1—O1—C2 | -91.17 (12) | C3—N1—C5—C6 | -61.9 (2) |
| O2—Ni1—O1—C2 | 88.83 (12) | Ni1—N1—C5—C6 | 178.71 (13) |
| N1—Ni1—O1—C2 | 5.39 (12) | N1—C5—C6—O3 | 82.7 (2) |
| N1 ⁱ —Ni1—O1—C2 | -174.61 (12) | | |

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

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|-------|-------------|-------------|---------------|
| O1—H1C \cdots O5 ⁱⁱⁱ | 0.82 | 2.19 | 2.663 (2) | 117 |
| O2—H2C \cdots O4 ^{iv} | 0.82 | 2.28 | 2.6227 (19) | 106 |
| O3—H3C \cdots O5 | 0.82 | 2.00 | 2.818 (2) | 174 |
| C3—H3B \cdots O3 | 0.97 | 2.26 | 3.055 (3) | 139 |

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

Fig. 1

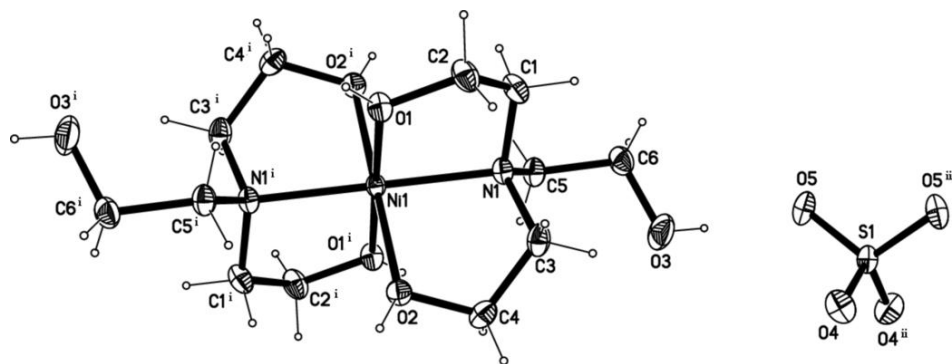


Fig. 2

