

Bis(propane-1,3-diamine)-disaccharinatonickel(II)

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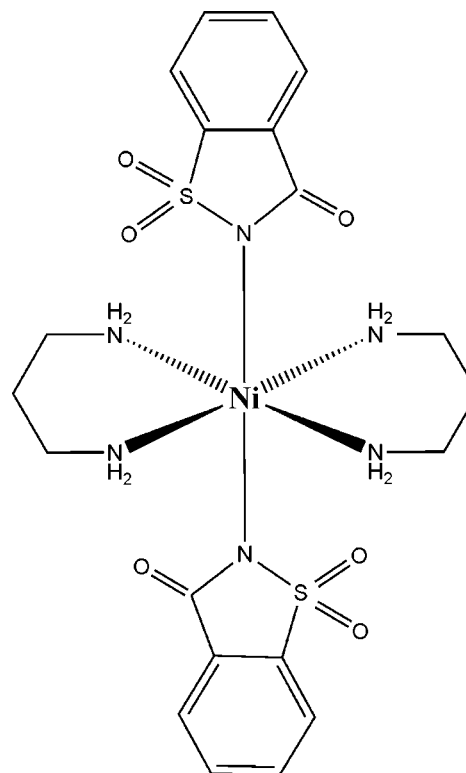
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å;
 R factor = 0.033; wR factor = 0.101; data-to-parameter ratio = 14.6.

In the title complex, $[\text{Ni}(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_3\text{H}_{10}\text{N}_2)_2]$ or $[\text{Ni}(\text{sac})_2(\text{pen})_2]$ (sac = saccharinate or 1,1,3-trioxo-2,3-dihydro-1 λ 6,2-benzothiazol-2-ide and pen = propane-1,3-diamine), the Ni^{II} ion sits on an inversion center, being coordinated by two N atoms of the sac ligands, which occupy *trans* positions, and four N atoms of the bidentate pen ligands to define a distorted octahedral geometry. The pen ligands chelate the metal ion, forming a six-membered ring which adopts a half-chair conformation, while the sac ligands adopt the most common coordination mode. The crystal packing is stabilized by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, which form a one-dimensional network along [010]. It is also supported by an $\text{N}-\text{H}\cdots\text{S}$ hydrogen bond between the amine group of the pen ligand and the sulfonyl group of the sac ligand.

Related literature

For background to saccharin and the use of the saccharinato anion (sac), as a polyfunctional ligand, see: Baran & Yilmaz (2006); Heren *et al.* (2008); Paşaoğlu *et al.* (2007). For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For a related structure, see: Bulut *et al.* (2007).



Experimental

Crystal data

$[\text{Ni}(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_3\text{H}_{10}\text{N}_2)_2]$
 $M_r = 571.31$
Monoclinic, $P2_1/n$
 $a = 11.447$ (5) Å
 $b = 7.209$ (6) Å
 $c = 15.236$ (5) Å
 $\beta = 109.313$ (5)°

$V = 1186.5$ (12) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 1.04$ mm⁻¹
 $T = 296$ K
 $0.52 \times 0.35 \times 0.15$ mm

Data collection

Stoe IPDS 2 diffractometer
Absorption correction: integration
(*X-RED*; Stoe & Cie, 2002)
 $T_{\text{min}} = 0.673$, $T_{\text{max}} = 0.873$

18160 measured reflections
2590 independent reflections
2334 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.101$
 $S = 0.83$
2590 reflections
177 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.74$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.37$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-----------|-----------|-----------|-------------|
| Ni1—N2 | 2.092 (2) | Ni1—N1 | 2.2604 (18) |
| Ni1—N3 | 2.118 (2) | | |
| N2—Ni1—N3 | 91.43 (9) | N3—Ni1—N1 | 88.87 (8) |
| N2—Ni1—N1 | 93.21 (8) | | |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|----------|-------------|-------------|---------------|
| N3—H3A \cdots O3 ⁱ | 0.78 (3) | 2.58 (3) | 3.194 (3) | 137 (3) |
| N3—H3B \cdots O2 ⁱⁱ | 0.85 (4) | 2.34 (4) | 3.143 (3) | 158 (3) |
| N3—H3B \cdots S1 ⁱⁱ | 0.85 (4) | 2.82 (4) | 3.431 (2) | 130 (3) |
| N2—H2A \cdots O1 ⁱⁱ | 0.90 (4) | 2.60 (3) | 3.227 (3) | 128 (3) |
| N2—H2B \cdots O3 ⁱⁱⁱ | 0.81 (4) | 2.39 (4) | 3.090 (3) | 146 (3) |
| N3—H3A \cdots O1 | 0.78 (3) | 2.36 (3) | 2.952 (3) | 134 (3) |
| N2—H2B \cdots O3 | 0.81 (4) | 2.56 (4) | 3.085 (3) | 124 (3) |

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DS2179).

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supplementary materials

Acta Cryst. (2012). E68, m425–m426 [doi:10.1107/S1600536812010525]

Bis(propane-1,3-diamine)disaccharinatonickel(II)**Gökhan Kaştaş and Can Kocabıyık****Comment**

Saccharin (3*H*-benzisothiazol-3-one 1,1-dioxide or *o*-benzosulfimide) is a well known artificial sweetener. It is readily deprotonated to form saccharinato anion (sac), which is a versatile polyfunctional ligand (Baran & Yilmaz, 2006). Because of the biological significance of saccharin, there has been increased interest in its metal complexes, especially with first-row transition metals (Paşaoğlu *et al.*, 2007; Heren *et al.*, 2008). Saccharin, or its anion, may bond to metals by means of their imino nitrogen, carbonyl oxygen, or sulfonyl oxygen atoms. In the last two decades, the metal saccharinates and metal complexes including saccharin and various N-donor ligands (mono- or bidentate) have been intensively studied by many investigators. In the present study, the mixed-ligand Ni^{II} complex of saccharinate with pen (Scheme) is investigated.

In the title compound, Ni^{II} ion sits on an inversion center, being coordinated by two N atoms of sac ligand and four N atoms of bidentate pen ligands in *trans* positions. The bond distances and angles (Table 1) show that the coordination polyhedron of the Ni^{II} ion is a distorted octahedron. The equatorial plane of the octahedron is defined by the N atoms of pen ligands, whereas the axial positions are occupied by the N atoms of the sac ligands (Fig. 1). It is seen that the pen ligands chelate the metal ion to form a six-membered ring adopting a half-chair conformation while sac ligands prefer the most common coordination mode. The intra-ligand bond lengths and angles of the sac ligand are similar to those observed in previous studies (Paşaoğlu *et al.*, 2007; Heren *et al.*, 2008). The sac ligand is planar, with an r.m.s deviation of 0.022 Å. The dihedral angle between the equatorial plane and the mean plane of sac was measured as 84.50 (7)°. It is seen in Table 1 that the Ni—N_{pen} bond distances span the range 2.094 (3)–2.119 (3) Å, being shorter than Ni—N_{sac} distance (2.262 (2) Å) which is comparable to that observed in [Ni(C₇H₄NO₃S)(C₅H₉N₃)₂] (2.2874 (19) Å) (Bulut *et al.*, 2007). The crystal packing of the complex is mainly stabilized by hydrogen bonds of N—H⁺⋯O type (Table 2). Multiple H-bond donor and acceptor behaviors of amine and sulfonyl groups enable a variety of chain and ring systems in the crystal packing. For example, the inter-molecular N2—H2B⁺⋯O3ⁱⁱⁱ (iii: -x, -1 - y, 1 - z) hydrogen bonds form centrosymmetric R²₂(12) (Bernstein *et al.*, 1995) rings located at (0, 1/2+n, 1/2: n=0 or integer) positions to form one-dimensional hydrogen bonding network along [010] (Fig. 2). On the other hand, the same ring motifs are also formed by N3—H3B⁺⋯O2ⁱ (i: -x, -y, 1 - z) hydrogen bonds (Fig. 3).

Experimental

An aqueous solution of pen (2.54 mmol, 0.19 g) was added dropwise with stirring to a solution of [Ni(sac)₂(H₂O)₄].2H₂O (1.27 mmol, 1.00 g). The mixture was heated to 343 K in a temperature-controlled bath and stirred for 3 h. The green reaction mixture was filtered and left for crystallization in room temperature. After two weeks, the violet crystals of the title complex were selected for X-ray experiment. IR (KBr, ν cm⁻¹), stretching vibrations: 3326–3290 (NH₂), 3153–3060 (CH)_{ring}, 2948–2881 (CH₂) 1658 (C=O), 1575 (C=C)_{sac} 1406 (C=N)_{pen}, 1325 (CNS)_{sym}, 1269 (SO₂)_{asym} 1141 (SO₂)_{sym}, 950 (CNS)_{asym}.

Refinement

All H atoms except those involved in H-bonds were positioned geometrically and treated using a riding model, with the distances of 0.970 and 0.930 Å for methylene and aromatic groups, respectively. The displacement parameters of the H atoms were constrained with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ for parent atoms.

Computing details

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2006) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

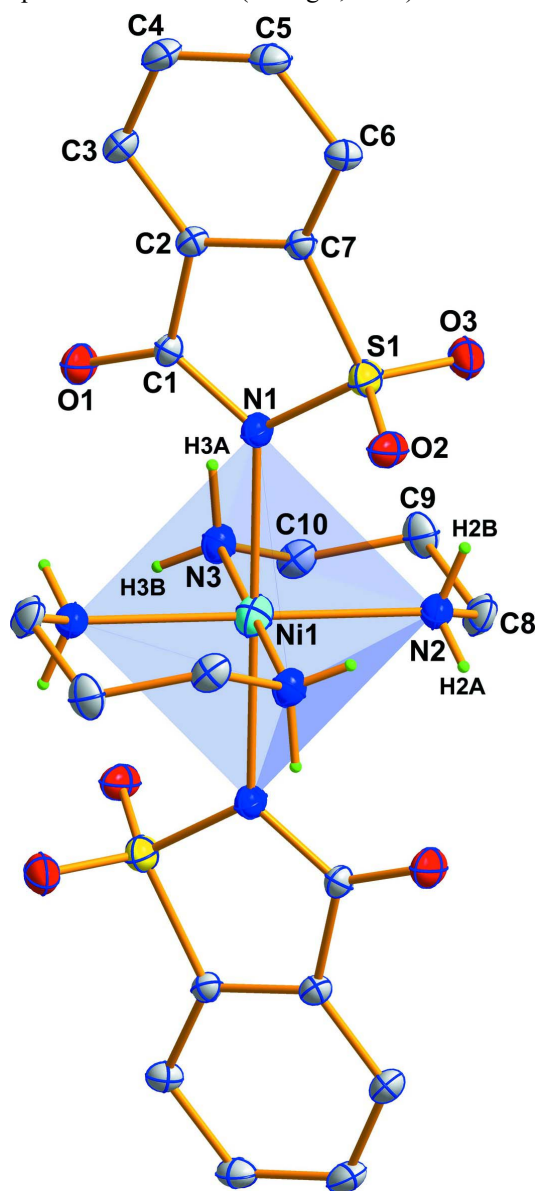
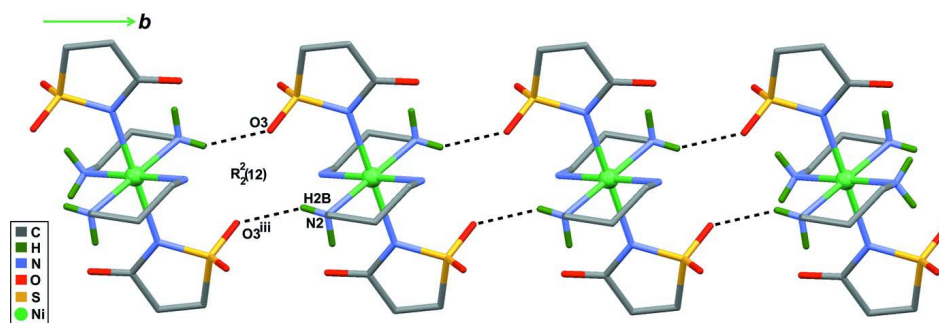
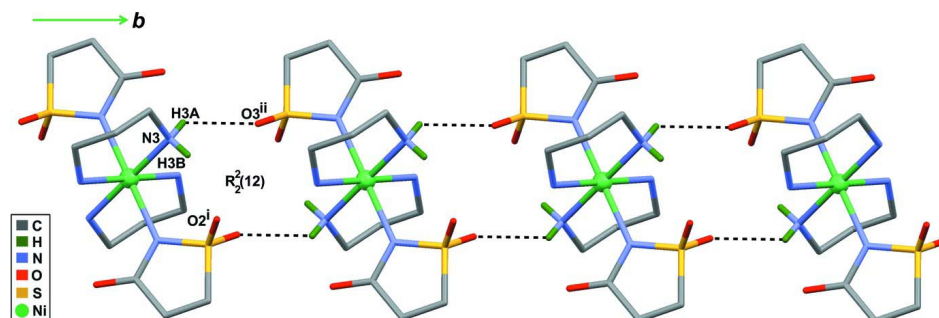


Figure 1

A molecular view of the complex, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms not involved in hydrogen bonding were omitted for clarity.


Figure 2

The centrosymmetric $R_2^2(12)$ rings connecting the discrete molecules along [010]. Benzene rings and some H atoms were omitted for clarity.


Figure 3

The formation of centrosymmetric $R_2^2(12)$ rings. Benzene rings and some H atoms were omitted for clarity.

Bis(propane-1,3-diamine- κ^2N,N')bis(1,1,3-trioxo-2,3-dihydro-1 λ^6 -benzothiazol-2-ido- κN)nickel(II)

Crystal data

$[\text{Ni}(\text{C}_7\text{H}_4\text{NO}_3\text{S})_2(\text{C}_3\text{H}_{10}\text{N}_2)_2]$

$M_r = 571.31$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1n$

$a = 11.447\ (5)\ \text{\AA}$

$b = 7.209\ (6)\ \text{\AA}$

$c = 15.236\ (5)\ \text{\AA}$

$\beta = 109.313\ (5)^\circ$

$V = 1186.5\ (12)\ \text{\AA}^3$

$Z = 2$

$F(000) = 596$

$D_x = 1.599\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71069\ \text{\AA}$

Cell parameters from 27512 reflections

$\theta = 1.9\text{--}28.1^\circ$

$\mu = 1.04\ \text{mm}^{-1}$

$T = 296\ \text{K}$

Prism, violet

$0.52 \times 0.35 \times 0.15\ \text{mm}$

Data collection

Stoe IPDS 2

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

rotation method scans

Absorption correction: integration

(*X-RED*; Stoe & Cie, 2002)

$T_{\min} = 0.673$, $T_{\max} = 0.873$

18160 measured reflections

2590 independent reflections

2334 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 27.0^\circ$, $\theta_{\min} = 2.0^\circ$

$h = -14 \rightarrow 14$

$k = -9 \rightarrow 9$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.101$
 $S = 0.83$
 2590 reflections
 177 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods
 Secondary atom site location: difference Fourier
 map

Hydrogen site location: inferred from
 neighbouring sites
 H atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0792P)^2 + 0.9405P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.74 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.37 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001xF_c^2\lambda^3/\sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0040 (12)

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 | 0.0000 | 0.0000 | 0.5000 | 0.03195 (14) |
| S1 | -0.09614 (5) | -0.33212 (7) | 0.62118 (4) | 0.03795 (16) |
| O3 | -0.00193 (18) | -0.4563 (2) | 0.61249 (13) | 0.0506 (4) |
| N1 | -0.05429 (17) | -0.1179 (2) | 0.61849 (12) | 0.0384 (4) |
| O2 | -0.21766 (18) | -0.3644 (3) | 0.55571 (12) | 0.0567 (5) |
| N3 | 0.1349 (2) | 0.1650 (3) | 0.59749 (14) | 0.0408 (4) |
| C2 | -0.0733 (2) | -0.1541 (3) | 0.76883 (15) | 0.0409 (5) |
| O1 | -0.02673 (19) | 0.1378 (2) | 0.71301 (14) | 0.0585 (5) |
| C7 | -0.10157 (19) | -0.3318 (3) | 0.73503 (15) | 0.0378 (4) |
| C1 | -0.0490 (2) | -0.0281 (3) | 0.69877 (17) | 0.0414 (5) |
| N2 | 0.12960 (19) | -0.2097 (3) | 0.50843 (14) | 0.0416 (4) |
| C6 | -0.1277 (2) | -0.4744 (4) | 0.78602 (17) | 0.0459 (5) |
| H6 | -0.1467 | -0.5931 | 0.7616 | 0.055* |
| C5 | -0.1241 (3) | -0.4313 (5) | 0.87586 (19) | 0.0578 (7) |
| H5 | -0.1404 | -0.5231 | 0.9131 | 0.069* |
| C3 | -0.0707 (3) | -0.1121 (4) | 0.85797 (18) | 0.0566 (6) |
| H3 | -0.0522 | 0.0071 | 0.8819 | 0.068* |
| C9 | 0.3068 (2) | -0.0680 (5) | 0.6283 (2) | 0.0633 (7) |
| H9A | 0.3305 | -0.0113 | 0.5790 | 0.076* |
| H9B | 0.3824 | -0.1027 | 0.6773 | 0.076* |
| C8 | 0.2373 (2) | -0.2388 (4) | 0.59136 (17) | 0.0527 (6) |
| H8A | 0.2924 | -0.3264 | 0.5767 | 0.063* |
| H8B | 0.2100 | -0.2939 | 0.6394 | 0.063* |
| C4 | -0.0965 (3) | -0.2530 (5) | 0.91060 (19) | 0.0641 (8) |

| | | | | |
|------|------------|------------|--------------|-------------|
| H4 | -0.0952 | -0.2274 | 0.9707 | 0.077* |
| C10 | 0.2423 (2) | 0.0754 (5) | 0.66625 (18) | 0.0614 (7) |
| H10A | 0.2148 | 0.0179 | 0.7136 | 0.074* |
| H10B | 0.3018 | 0.1708 | 0.6964 | 0.074* |
| H3A | 0.098 (3) | 0.220 (4) | 0.623 (2) | 0.045 (7)* |
| H3B | 0.168 (3) | 0.238 (5) | 0.569 (2) | 0.079 (11)* |
| H2A | 0.157 (3) | -0.179 (5) | 0.461 (2) | 0.068 (9)* |
| H2B | 0.091 (3) | -0.306 (5) | 0.498 (2) | 0.071 (10)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|--------------|---------------|
| Ni1 | 0.0364 (2) | 0.0281 (2) | 0.0298 (2) | -0.00006 (13) | 0.00887 (14) | -0.00239 (12) |
| S1 | 0.0495 (3) | 0.0318 (3) | 0.0353 (3) | -0.0060 (2) | 0.0177 (2) | -0.00277 (19) |
| O3 | 0.0724 (12) | 0.0318 (8) | 0.0610 (11) | -0.0004 (8) | 0.0399 (9) | -0.0036 (7) |
| N1 | 0.0512 (10) | 0.0299 (8) | 0.0373 (9) | -0.0036 (7) | 0.0189 (8) | -0.0019 (7) |
| O2 | 0.0613 (10) | 0.0609 (11) | 0.0423 (9) | -0.0209 (9) | 0.0098 (8) | -0.0012 (8) |
| N3 | 0.0475 (10) | 0.0392 (10) | 0.0351 (9) | -0.0075 (8) | 0.0125 (8) | -0.0044 (8) |
| C2 | 0.0426 (11) | 0.0438 (12) | 0.0393 (11) | -0.0010 (9) | 0.0176 (9) | -0.0056 (9) |
| O1 | 0.0853 (13) | 0.0364 (9) | 0.0662 (12) | -0.0125 (9) | 0.0419 (11) | -0.0154 (8) |
| C7 | 0.0375 (10) | 0.0408 (11) | 0.0371 (10) | 0.0010 (8) | 0.0152 (8) | -0.0012 (8) |
| C1 | 0.0471 (11) | 0.0362 (11) | 0.0448 (12) | -0.0018 (9) | 0.0204 (10) | -0.0074 (9) |
| N2 | 0.0458 (10) | 0.0352 (10) | 0.0430 (10) | 0.0030 (8) | 0.0138 (8) | -0.0004 (8) |
| C6 | 0.0488 (12) | 0.0479 (13) | 0.0453 (12) | 0.0005 (10) | 0.0214 (10) | 0.0061 (10) |
| C5 | 0.0595 (15) | 0.0734 (18) | 0.0474 (13) | -0.0001 (13) | 0.0270 (12) | 0.0104 (13) |
| C3 | 0.0673 (16) | 0.0627 (16) | 0.0461 (13) | -0.0074 (13) | 0.0275 (12) | -0.0155 (12) |
| C9 | 0.0435 (13) | 0.0687 (18) | 0.0675 (17) | 0.0012 (12) | 0.0045 (12) | -0.0007 (15) |
| C8 | 0.0532 (13) | 0.0592 (15) | 0.0434 (12) | 0.0150 (11) | 0.0128 (10) | 0.0037 (11) |
| C4 | 0.0736 (18) | 0.086 (2) | 0.0411 (13) | -0.0076 (16) | 0.0303 (13) | -0.0077 (13) |
| C10 | 0.0522 (14) | 0.081 (2) | 0.0412 (13) | -0.0014 (14) | 0.0020 (11) | -0.0180 (13) |

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

| | | | |
|---------------------|-------------|----------|-----------|
| Ni1—N2 | 2.092 (2) | N2—C8 | 1.459 (3) |
| Ni1—N2 ⁱ | 2.092 (2) | N2—H2A | 0.90 (4) |
| Ni1—N3 | 2.118 (2) | N2—H2B | 0.81 (4) |
| Ni1—N3 ⁱ | 2.118 (2) | C6—C5 | 1.391 (4) |
| Ni1—N1 ⁱ | 2.2604 (18) | C6—H6 | 0.9300 |
| Ni1—N1 | 2.2604 (18) | C5—C4 | 1.386 (5) |
| S1—O2 | 1.4375 (19) | C5—H5 | 0.9300 |
| S1—O3 | 1.4409 (18) | C3—C4 | 1.385 (4) |
| S1—N1 | 1.622 (2) | C3—H3 | 0.9300 |
| S1—C7 | 1.756 (2) | C9—C8 | 1.473 (4) |
| N1—C1 | 1.367 (3) | C9—C10 | 1.493 (4) |
| N3—C10 | 1.475 (3) | C9—H9A | 0.9700 |
| N3—H3A | 0.78 (3) | C9—H9B | 0.9700 |
| N3—H3B | 0.85 (4) | C8—H8A | 0.9700 |
| C2—C7 | 1.378 (3) | C8—H8B | 0.9700 |
| C2—C3 | 1.382 (3) | C4—H4 | 0.9300 |
| C2—C1 | 1.497 (3) | C10—H10A | 0.9700 |

| | | | |
|--------------------------------------|--------------|---------------|--------------|
| O1—C1 | 1.227 (3) | C10—H10B | 0.9700 |
| C7—C6 | 1.379 (3) | | |
| N2—Ni1—N2 ⁱ | 180.00 (12) | N1—C1—C2 | 112.75 (19) |
| N2—Ni1—N3 | 91.43 (9) | C8—N2—Ni1 | 122.38 (16) |
| N2 ⁱ —Ni1—N3 | 88.57 (9) | C8—N2—H2A | 108 (2) |
| N2—Ni1—N3 ⁱ | 88.57 (9) | Ni1—N2—H2A | 101 (2) |
| N2 ⁱ —Ni1—N3 ⁱ | 91.43 (9) | C8—N2—H2B | 107 (3) |
| N3—Ni1—N3 ⁱ | 180.0 | Ni1—N2—H2B | 106 (2) |
| N2—Ni1—N1 ⁱ | 86.79 (8) | H2A—N2—H2B | 112 (3) |
| N2 ⁱ —Ni1—N1 ⁱ | 93.21 (8) | C7—C6—C5 | 116.5 (2) |
| N3—Ni1—N1 ⁱ | 91.13 (8) | C7—C6—H6 | 121.7 |
| N3 ⁱ —Ni1—N1 ⁱ | 88.87 (8) | C5—C6—H6 | 121.7 |
| N2—Ni1—N1 | 93.21 (8) | C4—C5—C6 | 120.8 (3) |
| N2 ⁱ —Ni1—N1 | 86.79 (8) | C4—C5—H5 | 119.6 |
| N3—Ni1—N1 | 88.87 (8) | C6—C5—H5 | 119.6 |
| N3 ⁱ —Ni1—N1 | 91.13 (8) | C2—C3—C4 | 117.9 (3) |
| N1 ⁱ —Ni1—N1 | 180.000 (1) | C2—C3—H3 | 121.0 |
| O2—S1—O3 | 114.68 (12) | C4—C3—H3 | 121.0 |
| O2—S1—N1 | 111.28 (11) | C8—C9—C10 | 116.9 (2) |
| O3—S1—N1 | 110.67 (11) | C8—C9—H9A | 108.1 |
| O2—S1—C7 | 110.08 (11) | C10—C9—H9A | 108.1 |
| O3—S1—C7 | 111.32 (11) | C8—C9—H9B | 108.1 |
| N1—S1—C7 | 97.53 (10) | C10—C9—H9B | 108.1 |
| C1—N1—S1 | 110.76 (15) | H9A—C9—H9B | 107.3 |
| C1—N1—Ni1 | 126.42 (15) | N2—C8—C9 | 113.9 (2) |
| S1—N1—Ni1 | 122.67 (9) | N2—C8—H8A | 108.8 |
| C10—N3—Ni1 | 119.70 (17) | C9—C8—H8A | 108.8 |
| C10—N3—H3A | 109 (2) | N2—C8—H8B | 108.8 |
| Ni1—N3—H3A | 105 (2) | C9—C8—H8B | 108.8 |
| C10—N3—H3B | 103 (2) | H8A—C8—H8B | 107.7 |
| Ni1—N3—H3B | 110 (2) | C3—C4—C5 | 121.6 (2) |
| H3A—N3—H3B | 110 (3) | C3—C4—H4 | 119.2 |
| C7—C2—C3 | 119.8 (2) | C5—C4—H4 | 119.2 |
| C7—C2—C1 | 111.95 (19) | N3—C10—C9 | 115.5 (2) |
| C3—C2—C1 | 128.2 (2) | N3—C10—H10A | 108.4 |
| C2—C7—C6 | 123.3 (2) | C9—C10—H10A | 108.4 |
| C2—C7—S1 | 106.84 (16) | N3—C10—H10B | 108.4 |
| C6—C7—S1 | 129.82 (18) | C9—C10—H10B | 108.4 |
| O1—C1—N1 | 124.4 (2) | H10A—C10—H10B | 107.5 |
| O1—C1—C2 | 122.9 (2) | | |
| O2—S1—N1—C1 | 111.22 (18) | O3—S1—C7—C6 | -62.0 (2) |
| O3—S1—N1—C1 | -120.04 (17) | N1—S1—C7—C6 | -177.7 (2) |
| C7—S1—N1—C1 | -3.81 (18) | S1—N1—C1—O1 | -175.6 (2) |
| O2—S1—N1—Ni1 | -72.90 (14) | Ni1—N1—C1—O1 | 8.7 (4) |
| O3—S1—N1—Ni1 | 55.84 (15) | S1—N1—C1—C2 | 4.4 (2) |
| C7—S1—N1—Ni1 | 172.06 (11) | Ni1—N1—C1—C2 | -171.32 (14) |
| N2—Ni1—N1—C1 | 122.6 (2) | C7—C2—C1—O1 | 177.2 (2) |

| | | | |
|-----------------------------|--------------|----------------------------|-------------|
| N2 ⁱ —Ni1—N1—C1 | -57.4 (2) | C3—C2—C1—O1 | -2.7 (4) |
| N3—Ni1—N1—C1 | 31.3 (2) | C7—C2—C1—N1 | -2.8 (3) |
| N3 ⁱ —Ni1—N1—C1 | -148.7 (2) | C3—C2—C1—N1 | 177.3 (2) |
| N2—Ni1—N1—S1 | -52.59 (13) | N3—Ni1—N2—C8 | 27.2 (2) |
| N2 ⁱ —Ni1—N1—S1 | 127.41 (13) | N3 ⁱ —Ni1—N2—C8 | -152.8 (2) |
| N3—Ni1—N1—S1 | -143.95 (13) | N1 ⁱ —Ni1—N2—C8 | 118.2 (2) |
| N3 ⁱ —Ni1—N1—S1 | 36.05 (13) | N1—Ni1—N2—C8 | -61.8 (2) |
| N2—Ni1—N3—C10 | -26.0 (2) | C2—C7—C6—C5 | -0.2 (4) |
| N2 ⁱ —Ni1—N3—C10 | 154.0 (2) | S1—C7—C6—C5 | 179.64 (19) |
| N1 ⁱ —Ni1—N3—C10 | -112.9 (2) | C7—C6—C5—C4 | 0.5 (4) |
| N1—Ni1—N3—C10 | 67.1 (2) | C7—C2—C3—C4 | 0.3 (4) |
| C3—C2—C7—C6 | -0.2 (4) | C1—C2—C3—C4 | -179.9 (3) |
| C1—C2—C7—C6 | 179.9 (2) | Ni1—N2—C8—C9 | -48.1 (3) |
| C3—C2—C7—S1 | 179.9 (2) | C10—C9—C8—N2 | 66.0 (3) |
| C1—C2—C7—S1 | 0.1 (2) | C2—C3—C4—C5 | 0.0 (5) |
| O2—S1—C7—C2 | -113.86 (17) | C6—C5—C4—C3 | -0.4 (5) |
| O3—S1—C7—C2 | 117.82 (17) | Ni1—N3—C10—C9 | 47.0 (3) |
| N1—S1—C7—C2 | 2.12 (17) | C8—C9—C10—N3 | -66.7 (4) |
| O2—S1—C7—C6 | 66.3 (2) | | |

Symmetry code: (i) $-x, -y, -z+1$.

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|----------|-------------|-------------|---------------|
| N3—H3A \cdots O3 ⁱⁱ | 0.78 (3) | 2.58 (3) | 3.194 (3) | 137 (3) |
| N3—H3B \cdots O2 ⁱ | 0.85 (4) | 2.34 (4) | 3.143 (3) | 158 (3) |
| N3—H3B \cdots S1 ⁱ | 0.85 (4) | 2.82 (4) | 3.431 (2) | 130 (3) |
| N2—H2A \cdots O1 ⁱ | 0.90 (4) | 2.60 (3) | 3.227 (3) | 128 (3) |
| N2—H2B \cdots O3 ⁱⁱⁱ | 0.81 (4) | 2.39 (4) | 3.090 (3) | 146 (3) |
| N3—H3A \cdots O1 | 0.78 (3) | 2.36 (3) | 2.952 (3) | 134 (3) |
| N2—H2B \cdots O3 | 0.81 (4) | 2.56 (4) | 3.085 (3) | 124 (3) |

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