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# Hexakis(1*H*-imidazole-$\kappa N^3$)nickel(II) bis(3-thienylacetate)

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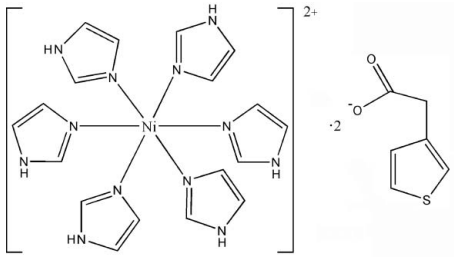
Key indicators: single-crystal X-ray study; $T$ = 296 K; mean $\sigma$(C–C) = 0.005 Å; disorder in solvent or counterion; $R$ factor = 0.040; $wR$ factor = 0.111; data-to-parameter ratio = 14.9.

In the title complex, $[Ni(C_3H_4N_2)_6](C_6H_5O_2S)_2$, the $Ni^{II}$ atom displays an octahedral coordination geometry, defined by six N atoms from the imidazole ligands. Intermolecular N—H···O hydrogen-bonding interactions between the cationic complex and 3-thienylacetate anions form a three-dimensional network architecture. The two 3-thienylacetate anions are disordered, with occupancy ratios of *circa* 0.774 (1):0.226 (1) and *ca* 0.753 (5):0.247 (5).

## Related literature

For related literature, see: Ng *et al.* (2001).



## Experimental

### Crystal data

$[Ni(C_3H_4N_2)_6](C_6H_5O_2S)_2$
$M_r$ = 749.52
Triclinic, $P\overline{1}$
$a$ = 9.2483 (3) Å
$b$ = 9.8529 (3) Å
$c$ = 19.6365 (6) Å
$\alpha$ = 84.696 (1)°
$\beta$ = 88.380 (2)°
$\gamma$ = 80.157 (2)°
$V$ = 1755.30 (9) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.72 mm$^{-1}$
$T$ = 296 (2) K
0.20 × 0.16 × 0.11 mm

### Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min}$ = 0.869, $T_{max}$ = 0.924
13333 measured reflections
7140 independent reflections
5337 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.025

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.111
$S$ = 1.06
7140 reflections
480 parameters
38 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.27 e Å$^{-3}$

**Table 1**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N12—H12···O4$^{i}$ | 0.86 | 2.56 | 3.124 (3) | 124 |
| N12—H12···O3$^{i}$ | 0.86 | 1.97 | 2.826 (3) | 170 |
| N10—H10$A$···O3$^{ii}$ | 0.86 | 1.88 | 2.718 (3) | 164 |
| N8—H8$A$···O2$^{iii}$ | 0.86 | 1.81 | 2.660 (3) | 170 |
| N4—H4$A$···O4$^{iv}$ | 0.86 | 1.89 | 2.688 (3) | 153 |
| N2—H2···O1$^{v}$ | 0.86 | 1.91 | 2.749 (3) | 166 |
| N6—H6···O1$^{ii}$ | 0.86 | 1.90 | 2.711 (3) | 156 |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; 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 acknowledge Guang Dong Ocean University for supporting this work.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2218).

## References

Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Ng, S. W., Chantrapromma, S., Razak, I. A. & Fun, H.-K. (2001). *Acta Cryst.* C**57**, 291–292.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2008). E**64**, m550 [doi:10.1107/S1600536808006363]

## Hexakis(1*H*-imidazole-$\kappa N^3$)nickel(II) bis(3-thienylacetate)

**Wen-Dong Song, Li-Li Ji and Hao Wang**

### S1. Comment

In the structural investigation of 3-thienylacetate complexes, it has been found that the 3-thienylacetate functions as a multidentate ligand [Ng *et al.* (2001)], with versatile binding and coordination modes. In this study, we expected to obtain a complex composed of nickel(II), 3-thienylacetate and imidazole by hydrothermal reaction. Unfortunately, the $Ni^{II}$ atom was not coordinated by 3-thienylacetate. We finally obtained the title structure, (I), composed of cations and anions.

As shown in Fig. 1, the crystal structure of the title complex consists of $[Ni(C_3H_4N_2)_6]^{2+}$ and two different 3-thienylacetate anions. The $Ni^{II}$ atom is coordinated by six different imidazole molecules in a slightly distorted octahedral geometry. The cationic complexes link the 3-thienylacetate anions by intermolecular N—H···O hydrogen bonding interactions (table 1) to form a three-dimensional network structure (Fig. 2).

### S2. Experimental

A mixture of nickel chloride (1 mmol), 3-thienylacetic acid (1 mmol), imidazole (1 mmol), NaOH (1.5 mmol) and $H_2O$ (12 ml) was placed in a 23 ml Teflon reactor, which was heated to 433 K for three days and then cooled to room temperature at a rate of 10 K $h^{-1}$. The crystals obtained were washed with water and dryed in air.

### S3. Refinement

Two independent 3-thienylacetate anions are disordered and they are split into two sets of positions, with occupancy ratios of 0.774 (1):0.226 (1) and 0.753 (5):0.247 (5), respectively. Due to the significant overlap of the disordered atoms the following restraints were applied: The two rings C1 C2 C3 C4 S1 (and ring C7 C8 C9 C10 S2) and their disordered counterparts were each restrained to be flat and their equivalent bond distances were restrained to be the same within a standard deviation of 0.01 Å. All H atoms were placed at calculated positions and were treated as riding on the parent C atoms with C—H = 0.93 Å (aromatic ring), and 0.97 Å (methylene); N—H = 0.86 Å, and with $U_{iso}$(H) = 1.2 $U_{eq}$(C, N).

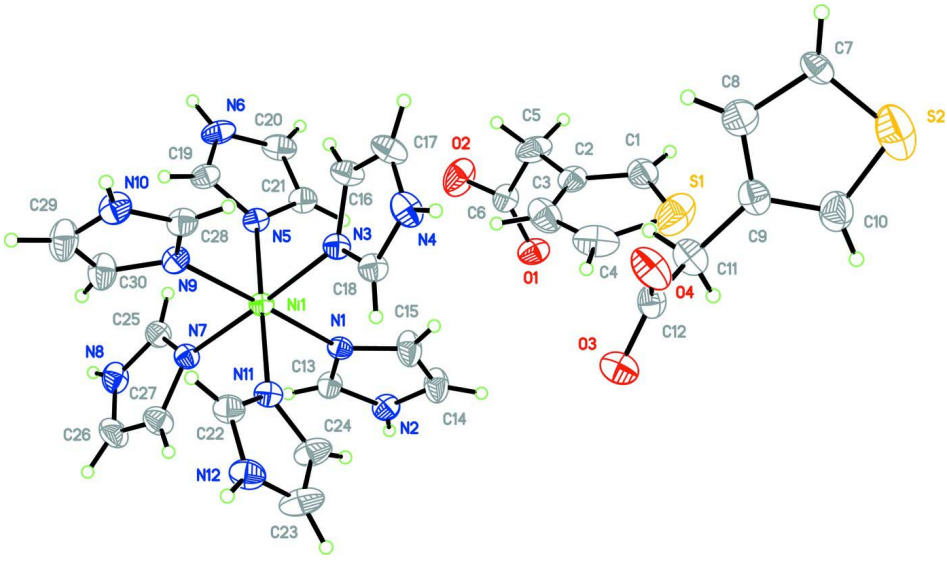



**Figure 1**

The structure of (I), showing the atomic numbering scheme. Non-H atoms are shown with 30% probability displacement ellipsoids.

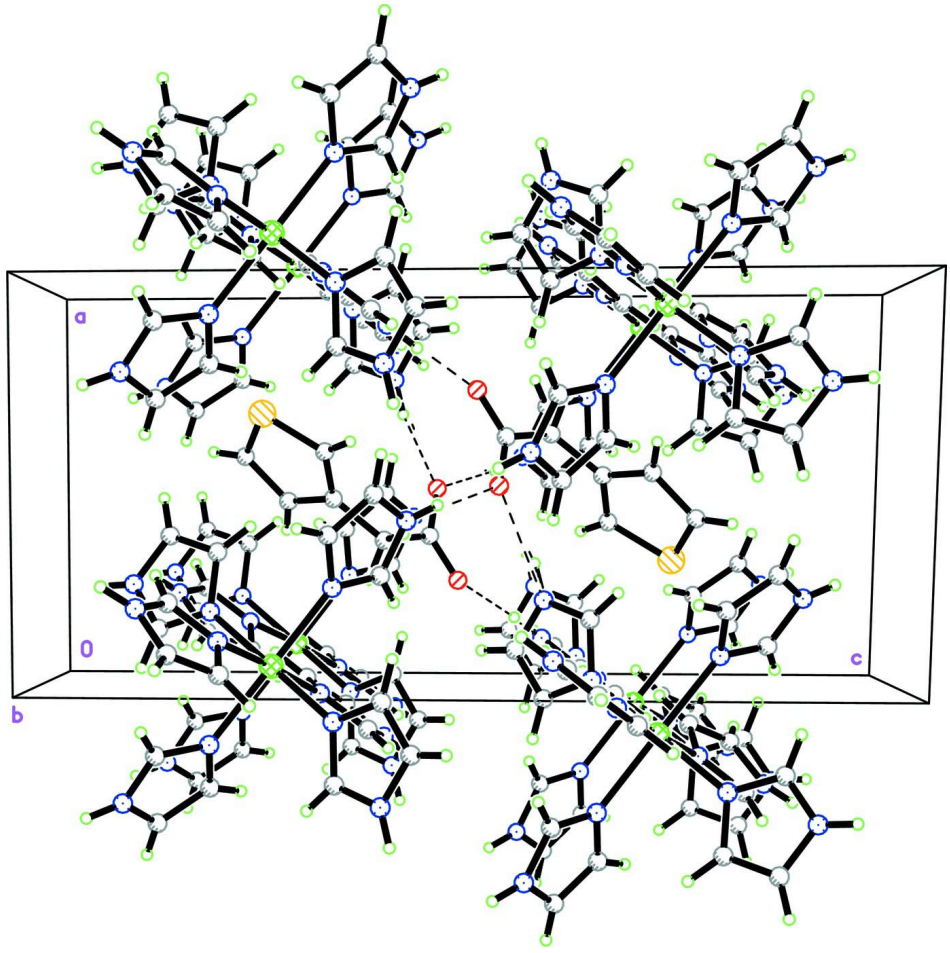



**Figure 2**

A packing view of the title compound. The intermolecluar hydrogen bonds are shown as dashed lines.

**Hexakis(1*H*-imidazole-$\kappa N^3$)nickel(II) bis(3-thienylacetate)**

*Crystal data*

$[Ni(C_3H_4N_2)_6](C_6H_5O_2S)_2$
$M_r = 749.52$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.2483$ (3) Å
$b = 9.8529$ (3) Å
$c = 19.6365$ (6) Å
$\alpha = 84.696$ (1)°
$\beta = 88.380$ (2)°
$\gamma = 80.157$ (2)°
$V = 1755.30$ (9) Å$^3$
$Z = 2$
$F(000) = 780$
$D_x = 1.418$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5680 reflections
$\theta = 1.4$–$28.0$°
$\mu = 0.73$ mm$^{-1}$
$T = 296$ K
Block, blue
0.20 × 0.16 × 0.11 mm

*Data collection*

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min} = 0.869$, $T_{max} = 0.925$

13333 measured reflections
7140 independent reflections
5337 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 26.5°$, $\theta_{min} = 1.0°$
$h = -11 \rightarrow 11$
$k = -5 \rightarrow 12$
$l = -24 \rightarrow 24$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.111$
$S = 1.06$
7140 reflections
480 parameters
38 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
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2 + 0.8694P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.39$ e Å$^{-3}$
$\Delta\rho_{min} = -0.27$ e Å$^{-3}$

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| C1 | 0.4131 (4) | 0.4287 (3) | 0.66425 (18) | 0.0627 (9) | |
| H1 | 0.3888 | 0.3694 | 0.6339 | 0.075* | |
| C2 | 0.5286 (3) | 0.4973 (3) | 0.65584 (14) | 0.0484 (7) | |
| C3 | 0.5372 (4) | 0.5773 (4) | 0.71015 (18) | 0.0681 (9) | |
| H3 | 0.6112 | 0.6302 | 0.7111 | 0.082* | |
| C5 | 0.6323 (4) | 0.4848 (4) | 0.59608 (18) | 0.0710 (10) | |
| H5A | 0.6163 | 0.4062 | 0.5727 | 0.085* | |
| H5B | 0.7315 | 0.4641 | 0.6135 | 0.085* | |
| C6 | 0.6230 (3) | 0.6089 (3) | 0.54369 (13) | 0.0474 (7) | |
| C8 | 0.3563 (4) | 0.2371 (3) | 0.99004 (16) | 0.0598 (8) | |
| H8 | 0.4549 | 0.2448 | 0.9920 | 0.072* | |
| C9 | 0.2493 (3) | 0.3502 (3) | 0.96818 (14) | 0.0500 (7) | |
| C10 | 0.1119 (4) | 0.3137 (4) | 0.97049 (19) | 0.0683 (9) | |
| H10 | 0.0270 | 0.3748 | 0.9576 | 0.082* | |
| C11 | 0.2791 (4) | 0.4927 (3) | 0.94645 (14) | 0.0559 (8) | |
| H11A | 0.1976 | 0.5421 | 0.9192 | 0.067* | |
| H11B | 0.3661 | 0.4849 | 0.9174 | 0.067* | |
| C12 | 0.3015 (3) | 0.5780 (3) | 1.00487 (13) | 0.0427 (6) | |
| C13 | 0.7099 (3) | 1.1011 (3) | 0.59500 (13) | 0.0444 (6) | |
| H13 | 0.7758 | 1.1596 | 0.5804 | 0.053* | |
| C14 | 0.5169 (4) | 1.0069 (4) | 0.60100 (17) | 0.0663 (9) | |

| | | | | |
|---|---|---|---|---|
| H14 | 0.4254 | 0.9851 | 0.5928 | 0.080* |
| C15 | 0.6119 (3) | 0.9449 (3) | 0.64942 (17) | 0.0637 (9) |
| H15 | 0.5958 | 0.8722 | 0.6808 | 0.076* |
| C16 | 0.9193 (3) | 0.6510 (3) | 0.75680 (15) | 0.0517 (7) |
| H16 | 0.9882 | 0.6223 | 0.7234 | 0.062* |
| C17 | 0.8696 (4) | 0.5679 (3) | 0.80681 (16) | 0.0601 (8) |
| H17 | 0.8984 | 0.4727 | 0.8145 | 0.072* |
| C18 | 0.7630 (3) | 0.7782 (3) | 0.81614 (13) | 0.0446 (6) |
| H18 | 0.7026 | 0.8539 | 0.8327 | 0.053* |
| C19 | 1.1837 (3) | 0.8033 (3) | 0.63842 (14) | 0.0460 (7) |
| H19 | 1.2480 | 0.8292 | 0.6684 | 0.055* |
| N6 | 1.2259 (3) | 0.7234 (3) | 0.58772 (11) | 0.0532 (6) |
| H6 | 1.3145 | 0.6887 | 0.5772 | 0.064* |
| C21 | 0.9893 (3) | 0.7778 (3) | 0.58834 (13) | 0.0478 (7) |
| H21 | 0.8912 | 0.7835 | 0.5772 | 0.057* |
| C22 | 0.8396 (3) | 1.1189 (3) | 0.84289 (14) | 0.0485 (7) |
| H22 | 0.9326 | 1.0865 | 0.8606 | 0.058* |
| C23 | 0.6170 (4) | 1.2253 (4) | 0.83415 (17) | 0.0744 (11) |
| H23 | 0.5270 | 1.2794 | 0.8429 | 0.089* |
| C24 | 0.6547 (3) | 1.1572 (3) | 0.77818 (16) | 0.0613 (9) |
| H24 | 0.5936 | 1.1560 | 0.7415 | 0.074* |
| C25 | 1.0829 (3) | 1.1481 (3) | 0.60810 (14) | 0.0462 (6) |
| H25 | 1.1209 | 1.0723 | 0.5843 | 0.055* |
| C26 | 1.0367 (4) | 1.3572 (3) | 0.63700 (17) | 0.0623 (8) |
| H26 | 1.0349 | 1.4516 | 0.6382 | 0.075* |
| C27 | 0.9656 (4) | 1.2747 (3) | 0.68025 (16) | 0.0554 (8) |
| H27 | 0.9057 | 1.3040 | 0.7168 | 0.066* |
| C28 | 1.1230 (3) | 0.8441 (3) | 0.83343 (13) | 0.0452 (6) |
| H28 | 1.0629 | 0.7789 | 0.8455 | 0.054* |
| C29 | 1.2965 (4) | 0.9652 (4) | 0.83702 (17) | 0.0705 (10) |
| H29 | 1.3772 | 1.0002 | 0.8507 | 0.085* |
| C30 | 1.2145 (3) | 1.0096 (4) | 0.78075 (16) | 0.0600 (8) |
| H30 | 1.2302 | 1.0813 | 0.7487 | 0.072* |
| N1 | 0.7348 (2) | 1.0039 (2) | 0.64586 (10) | 0.0367 (5) |
| N2 | 0.5794 (3) | 1.1062 (2) | 0.56680 (11) | 0.0480 (6) |
| H2 | 0.5425 | 1.1624 | 0.5332 | 0.058* |
| N3 | 0.8519 (2) | 0.7861 (2) | 0.76277 (10) | 0.0391 (5) |
| N4 | 0.7707 (3) | 0.6476 (3) | 0.84356 (12) | 0.0549 (6) |
| H4A | 0.7214 | 0.6201 | 0.8783 | 0.066* |
| N5 | 1.0399 (2) | 0.8411 (2) | 0.64094 (10) | 0.0390 (5) |
| C20 | 1.1030 (4) | 0.7072 (3) | 0.55600 (15) | 0.0567 (8) |
| H20 | 1.0984 | 0.6565 | 0.5187 | 0.068* |
| N7 | 0.9954 (2) | 1.1416 (2) | 0.66199 (10) | 0.0389 (5) |
| N8 | 1.1108 (3) | 1.2744 (3) | 0.59158 (13) | 0.0551 (6) |
| H8A | 1.1657 | 1.2993 | 0.5583 | 0.066* |
| N9 | 1.1042 (2) | 0.9323 (2) | 0.77832 (10) | 0.0419 (5) |
| N10 | 1.2373 (3) | 0.8589 (3) | 0.86970 (12) | 0.0527 (6) |
| H10A | 1.2682 | 0.8107 | 0.9068 | 0.063* |

| | | | | | |
|---|---|---|---|---|---|
| N11 | 0.7967 (2) | 1.0899 (2) | 0.78359 (10) | 0.0405 (5) | |
| N12 | 0.7343 (3) | 1.2000 (3) | 0.87473 (12) | 0.0579 (7) | |
| H12 | 0.7404 | 1.2305 | 0.9140 | 0.069* | |
| Ni1 | 0.91924 (3) | 0.96550 (3) | 0.711416 (15) | 0.03369 (11) | |
| O1 | 0.5032 (2) | 0.6882 (2) | 0.53592 (10) | 0.0561 (5) | |
| O2 | 0.7362 (3) | 0.6167 (3) | 0.50974 (13) | 0.0884 (8) | |
| O3 | 0.2792 (2) | 0.7082 (2) | 0.99273 (10) | 0.0554 (5) | |
| O4 | 0.3444 (3) | 0.5178 (2) | 1.06095 (10) | 0.0628 (6) | |
| C4 | 0.4398 (14) | 0.5770 (17) | 0.7597 (8) | 0.089 (3) | 0.774 (12) |
| H4 | 0.4352 | 0.6252 | 0.7985 | 0.107* | 0.774 (12) |
| S1 | 0.3174 (3) | 0.4661 (3) | 0.73853 (14) | 0.0805 (17) | 0.774 (12) |
| C7 | 0.3090 (15) | 0.1157 (12) | 1.0081 (8) | 0.067 (3) | 0.753 (5) |
| H7 | 0.3670 | 0.0318 | 1.0226 | 0.080* | 0.753 (5) |
| S2 | 0.1132 (3) | 0.1476 (2) | 0.99890 (12) | 0.0834 (8) | 0.753 (5) |
| S1′ | 0.3871 (14) | 0.5542 (15) | 0.7623 (6) | 0.124 (12) | 0.226 (12) |
| C4′ | 0.324 (3) | 0.429 (3) | 0.7164 (11) | 0.060 (8) | 0.226 (12) |
| H4′ | 0.2484 | 0.3778 | 0.7267 | 0.072* | 0.226 (12) |
| C7′ | 0.098 (3) | 0.1816 (18) | 0.9903 (14) | 0.16 (2) | 0.247 (5) |
| H7′ | 0.0132 | 0.1426 | 0.9924 | 0.193* | 0.247 (5) |
| S2′ | 0.2812 (15) | 0.0985 (12) | 1.0115 (8) | 0.091 (4) | 0.247 (5) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.064 (2) | 0.055 (2) | 0.069 (2) | −0.0105 (16) | −0.0164 (18) | 0.0026 (16) |
| C2 | 0.0470 (16) | 0.0488 (17) | 0.0432 (15) | 0.0005 (13) | −0.0061 (12) | 0.0146 (13) |
| C3 | 0.085 (3) | 0.063 (2) | 0.059 (2) | −0.0222 (18) | −0.0127 (19) | 0.0047 (17) |
| C5 | 0.065 (2) | 0.065 (2) | 0.068 (2) | 0.0153 (17) | 0.0114 (17) | 0.0182 (18) |
| C6 | 0.0476 (16) | 0.0587 (18) | 0.0361 (14) | −0.0126 (14) | 0.0016 (12) | 0.0004 (13) |
| C8 | 0.063 (2) | 0.059 (2) | 0.0593 (19) | −0.0130 (16) | 0.0103 (15) | −0.0139 (16) |
| C9 | 0.0613 (19) | 0.0543 (18) | 0.0395 (15) | −0.0195 (15) | 0.0042 (13) | −0.0138 (13) |
| C10 | 0.066 (2) | 0.071 (2) | 0.072 (2) | −0.0202 (19) | −0.0022 (18) | −0.0153 (19) |
| C11 | 0.077 (2) | 0.0567 (19) | 0.0371 (15) | −0.0195 (16) | −0.0023 (14) | −0.0053 (13) |
| C12 | 0.0457 (15) | 0.0502 (17) | 0.0345 (14) | −0.0154 (12) | 0.0054 (11) | −0.0035 (12) |
| C13 | 0.0444 (15) | 0.0462 (16) | 0.0408 (15) | −0.0085 (12) | −0.0051 (12) | 0.0078 (12) |
| C14 | 0.0580 (19) | 0.069 (2) | 0.074 (2) | −0.0256 (17) | −0.0246 (17) | 0.0182 (18) |
| C15 | 0.061 (2) | 0.062 (2) | 0.070 (2) | −0.0281 (16) | −0.0207 (16) | 0.0278 (17) |
| C16 | 0.0685 (19) | 0.0358 (15) | 0.0474 (16) | −0.0021 (13) | 0.0075 (14) | −0.0018 (13) |
| C17 | 0.090 (2) | 0.0334 (16) | 0.0547 (18) | −0.0088 (15) | 0.0014 (17) | 0.0036 (14) |
| C18 | 0.0506 (16) | 0.0429 (16) | 0.0381 (14) | −0.0062 (12) | 0.0021 (12) | 0.0033 (12) |
| C19 | 0.0461 (16) | 0.0480 (16) | 0.0391 (14) | 0.0020 (12) | 0.0026 (12) | 0.0020 (12) |
| N6 | 0.0540 (15) | 0.0539 (15) | 0.0418 (13) | 0.0140 (12) | 0.0104 (11) | 0.0011 (11) |
| C21 | 0.0533 (17) | 0.0492 (17) | 0.0400 (15) | −0.0046 (13) | −0.0008 (13) | −0.0071 (13) |
| C22 | 0.0545 (17) | 0.0491 (17) | 0.0415 (15) | −0.0043 (13) | 0.0022 (13) | −0.0113 (13) |
| C23 | 0.072 (2) | 0.081 (3) | 0.057 (2) | 0.0263 (19) | 0.0033 (18) | −0.0153 (18) |
| C24 | 0.0593 (19) | 0.069 (2) | 0.0482 (17) | 0.0155 (16) | −0.0044 (14) | −0.0168 (15) |
| C25 | 0.0505 (16) | 0.0413 (16) | 0.0462 (16) | −0.0102 (12) | 0.0024 (13) | 0.0024 (12) |
| C26 | 0.075 (2) | 0.0413 (18) | 0.074 (2) | −0.0226 (16) | 0.0059 (18) | −0.0003 (16) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C27 | 0.066 (2) | 0.0464 (18) | 0.0539 (18) | −0.0133 (15) | 0.0080 (15) | −0.0028 (14) |
| C28 | 0.0494 (16) | 0.0452 (16) | 0.0384 (14) | −0.0024 (12) | −0.0073 (12) | 0.0010 (12) |
| C29 | 0.063 (2) | 0.097 (3) | 0.056 (2) | −0.032 (2) | −0.0206 (16) | 0.0054 (19) |
| C30 | 0.0579 (19) | 0.075 (2) | 0.0492 (17) | −0.0245 (17) | −0.0101 (14) | 0.0100 (16) |
| N1 | 0.0421 (12) | 0.0319 (11) | 0.0344 (11) | −0.0026 (9) | −0.0028 (9) | 0.0007 (9) |
| N2 | 0.0514 (14) | 0.0482 (14) | 0.0413 (12) | −0.0034 (11) | −0.0130 (11) | 0.0067 (11) |
| N3 | 0.0439 (12) | 0.0386 (12) | 0.0331 (11) | −0.0054 (9) | −0.0008 (9) | 0.0021 (9) |
| N4 | 0.0746 (17) | 0.0543 (16) | 0.0373 (12) | −0.0229 (13) | 0.0035 (12) | 0.0090 (11) |
| N5 | 0.0402 (12) | 0.0418 (13) | 0.0332 (11) | −0.0036 (9) | 0.0021 (9) | −0.0008 (9) |
| C20 | 0.076 (2) | 0.0509 (18) | 0.0393 (15) | 0.0030 (15) | 0.0036 (15) | −0.0110 (13) |
| N7 | 0.0437 (12) | 0.0353 (12) | 0.0372 (11) | −0.0081 (9) | −0.0018 (9) | 0.0016 (9) |
| N8 | 0.0532 (15) | 0.0600 (17) | 0.0530 (15) | −0.0209 (12) | 0.0030 (12) | 0.0104 (13) |
| N9 | 0.0428 (12) | 0.0468 (13) | 0.0348 (11) | −0.0055 (10) | −0.0040 (9) | −0.0005 (10) |
| N10 | 0.0574 (15) | 0.0593 (16) | 0.0387 (13) | −0.0028 (12) | −0.0140 (11) | 0.0014 (11) |
| N11 | 0.0475 (13) | 0.0378 (12) | 0.0344 (11) | −0.0039 (10) | 0.0026 (9) | −0.0011 (9) |
| N12 | 0.0740 (18) | 0.0554 (16) | 0.0435 (14) | −0.0027 (13) | 0.0027 (13) | −0.0164 (12) |
| Ni1 | 0.03781 (18) | 0.03297 (18) | 0.02871 (17) | −0.00365 (13) | 0.00005 (12) | 0.00110 (12) |
| O1 | 0.0570 (13) | 0.0541 (13) | 0.0480 (11) | 0.0056 (10) | 0.0028 (9) | 0.0148 (10) |
| O2 | 0.0612 (15) | 0.118 (2) | 0.0812 (17) | −0.0209 (14) | 0.0233 (13) | 0.0226 (16) |
| O3 | 0.0763 (14) | 0.0448 (12) | 0.0438 (11) | −0.0058 (10) | −0.0097 (10) | −0.0023 (9) |
| O4 | 0.1051 (18) | 0.0539 (13) | 0.0348 (11) | −0.0320 (12) | −0.0040 (11) | 0.0037 (9) |
| C4 | 0.099 (5) | 0.095 (5) | 0.071 (5) | −0.018 (4) | −0.002 (4) | −0.001 (4) |
| S1 | 0.0613 (11) | 0.093 (3) | 0.077 (2) | −0.0054 (11) | 0.0176 (11) | 0.024 (2) |
| C7 | 0.069 (4) | 0.065 (6) | 0.068 (5) | −0.020 (4) | 0.014 (3) | −0.008 (4) |
| S2 | 0.0988 (18) | 0.0813 (11) | 0.0849 (12) | −0.0531 (10) | 0.0155 (10) | −0.0206 (9) |
| S1′ | 0.18 (2) | 0.109 (13) | 0.056 (4) | 0.037 (15) | 0.015 (7) | 0.012 (5) |
| C4′ | 0.069 (15) | 0.063 (16) | 0.054 (16) | −0.033 (13) | 0.005 (11) | 0.008 (10) |
| C7′ | 0.08 (2) | 0.25 (5) | 0.12 (2) | 0.03 (2) | −0.046 (17) | 0.05 (2) |
| S2′ | 0.141 (11) | 0.072 (4) | 0.073 (4) | −0.055 (5) | 0.026 (5) | −0.017 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| C1—C4′ | 1.299 (15) | N6—H6 | 0.8600 |
| C1—C2 | 1.357 (4) | C21—C20 | 1.336 (4) |
| C1—S1 | 1.721 (4) | C21—N5 | 1.386 (3) |
| C1—H1 | 0.9300 | C21—H21 | 0.9300 |
| C2—C3 | 1.395 (4) | C22—N11 | 1.312 (3) |
| C2—C5 | 1.495 (4) | C22—N12 | 1.330 (4) |
| C3—C4 | 1.307 (12) | C22—H22 | 0.9300 |
| C3—S1′ | 1.735 (9) | C23—N12 | 1.339 (4) |
| C3—H3 | 0.9300 | C23—C24 | 1.347 (4) |
| C5—C6 | 1.516 (4) | C23—H23 | 0.9300 |
| C5—H5A | 0.9700 | C24—N11 | 1.368 (4) |
| C5—H5B | 0.9700 | C24—H24 | 0.9300 |
| C6—O2 | 1.235 (3) | C25—N7 | 1.318 (3) |
| C6—O1 | 1.245 (3) | C25—N8 | 1.323 (4) |
| C8—C7 | 1.356 (12) | C25—H25 | 0.9300 |
| C8—C9 | 1.401 (4) | C26—C27 | 1.355 (4) |

| | | | |
|---|---|---|---|
| C8—S2′ | 1.651 (10) | C26—N8 | 1.357 (4) |
| C8—H8 | 0.9300 | C26—H26 | 0.9300 |
| C9—C10 | 1.377 (5) | C27—N7 | 1.372 (4) |
| C9—C11 | 1.498 (4) | C27—H27 | 0.9300 |
| C10—C7′ | 1.350 (15) | C28—N9 | 1.320 (3) |
| C10—S2 | 1.677 (4) | C28—N10 | 1.327 (3) |
| C10—H10 | 0.9300 | C28—H28 | 0.9300 |
| C11—C12 | 1.522 (4) | C29—C30 | 1.353 (4) |
| C11—H11A | 0.9700 | C29—N10 | 1.364 (4) |
| C11—H11B | 0.9700 | C29—H29 | 0.9300 |
| C12—O4 | 1.243 (3) | C30—N9 | 1.377 (4) |
| C12—O3 | 1.265 (3) | C30—H30 | 0.9300 |
| C13—N1 | 1.315 (3) | N1—Ni1 | 2.127 (2) |
| C13—N2 | 1.334 (3) | N2—H2 | 0.8600 |
| C13—H13 | 0.9300 | N3—Ni1 | 2.130 (2) |
| C14—N2 | 1.338 (4) | N4—H4A | 0.8600 |
| C14—C15 | 1.344 (4) | N5—Ni1 | 2.103 (2) |
| C14—H14 | 0.9300 | C20—H20 | 0.9300 |
| C15—N1 | 1.360 (4) | N7—Ni1 | 2.125 (2) |
| C15—H15 | 0.9300 | N8—H8A | 0.8600 |
| C16—C17 | 1.342 (4) | N9—Ni1 | 2.149 (2) |
| C16—N3 | 1.384 (3) | N10—H10A | 0.8600 |
| C16—H16 | 0.9300 | N11—Ni1 | 2.133 (2) |
| C17—N4 | 1.340 (4) | N12—H12 | 0.8600 |
| C17—H17 | 0.9300 | C4—S1 | 1.783 (13) |
| C18—N3 | 1.318 (3) | C4—H4 | 0.9300 |
| C18—N4 | 1.339 (3) | C7—S2 | 1.796 (12) |
| C18—H18 | 0.9300 | C7—H7 | 0.9300 |
| C19—N5 | 1.319 (3) | S1′—C4′ | 1.782 (14) |
| C19—N6 | 1.335 (3) | C4′—H4′ | 0.9300 |
| C19—H19 | 0.9300 | C7′—S2′ | 1.792 (14) |
| N6—C20 | 1.351 (4) | C7′—H7′ | 0.9300 |

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|---|---|---|---|
| C4′—C1—C2 | 126.7 (11) | C27—C26—N8 | 106.2 (3) |
| C2—C1—S1 | 111.0 (3) | C27—C26—H26 | 126.9 |
| C4′—C1—H1 | 108.8 | N8—C26—H26 | 126.9 |
| C2—C1—H1 | 124.5 | C26—C27—N7 | 109.7 (3) |
| S1—C1—H1 | 124.5 | C26—C27—H27 | 125.2 |
| C1—C2—C3 | 111.1 (3) | N7—C27—H27 | 125.2 |
| C1—C2—C5 | 123.2 (3) | N9—C28—N10 | 112.4 (3) |
| C3—C2—C5 | 125.7 (3) | N9—C28—H28 | 123.8 |
| C4—C3—C2 | 119.1 (7) | N10—C28—H28 | 123.8 |
| C2—C3—S1′ | 105.0 (6) | C30—C29—N10 | 106.4 (3) |
| C4—C3—H3 | 120.4 | C30—C29—H29 | 126.8 |
| C2—C3—H3 | 120.4 | N10—C29—H29 | 126.8 |
| S1′—C3—H3 | 134.6 | C29—C30—N9 | 109.6 (3) |
| C2—C5—C6 | 117.1 (3) | C29—C30—H30 | 125.2 |
| C2—C5—H5A | 108.0 | N9—C30—H30 | 125.2 |

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| C6—C5—H5A | 108.0 | C13—N1—C15 | 104.1 (2) |
| C2—C5—H5B | 108.0 | C13—N1—Ni1 | 126.26 (18) |
| C6—C5—H5B | 108.0 | C15—N1—Ni1 | 129.40 (18) |
| H5A—C5—H5B | 107.3 | C13—N2—C14 | 106.7 (2) |
| O2—C6—O1 | 126.1 (3) | C13—N2—H2 | 126.7 |
| O2—C6—C5 | 115.1 (3) | C14—N2—H2 | 126.7 |
| O1—C6—C5 | 118.7 (3) | C18—N3—C16 | 104.6 (2) |
| C7—C8—C9 | 116.7 (6) | C18—N3—Ni1 | 128.34 (18) |
| C9—C8—S2′ | 110.8 (6) | C16—N3—Ni1 | 125.55 (18) |
| C7—C8—H8 | 121.6 | C18—N4—C17 | 107.4 (2) |
| C9—C8—H8 | 121.6 | C18—N4—H4A | 126.3 |
| S2′—C8—H8 | 127.5 | C17—N4—H4A | 126.3 |
| C10—C9—C8 | 110.9 (3) | C19—N5—C21 | 104.2 (2) |
| C10—C9—C11 | 124.2 (3) | C19—N5—Ni1 | 126.72 (18) |
| C8—C9—C11 | 124.9 (3) | C21—N5—Ni1 | 129.02 (18) |
| C7′—C10—C9 | 119.1 (12) | C21—C20—N6 | 107.1 (3) |
| C9—C10—S2 | 113.1 (3) | C21—C20—H20 | 126.5 |
| C7′—C10—H10 | 117.4 | N6—C20—H20 | 126.5 |
| C9—C10—H10 | 123.4 | C25—N7—C27 | 104.4 (2) |
| S2—C10—H10 | 123.4 | C25—N7—Ni1 | 128.32 (19) |
| C9—C11—C12 | 114.9 (2) | C27—N7—Ni1 | 127.25 (19) |
| C9—C11—H11A | 108.6 | C25—N8—C26 | 107.2 (2) |
| C12—C11—H11A | 108.6 | C25—N8—H8A | 126.4 |
| C9—C11—H11B | 108.6 | C26—N8—H8A | 126.4 |
| C12—C11—H11B | 108.6 | C28—N9—C30 | 104.6 (2) |
| H11A—C11—H11B | 107.5 | C28—N9—Ni1 | 125.81 (19) |
| O4—C12—O3 | 123.2 (3) | C30—N9—Ni1 | 128.84 (19) |
| O4—C12—C11 | 119.3 (3) | C28—N10—C29 | 107.0 (2) |
| O3—C12—C11 | 117.4 (2) | C28—N10—H10A | 126.5 |
| N1—C13—N2 | 112.3 (2) | C29—N10—H10A | 126.5 |
| N1—C13—H13 | 123.9 | C22—N11—C24 | 104.6 (2) |
| N2—C13—H13 | 123.9 | C22—N11—Ni1 | 128.08 (19) |
| N2—C14—C15 | 106.8 (3) | C24—N11—Ni1 | 127.36 (18) |
| N2—C14—H14 | 126.6 | C22—N12—C23 | 107.2 (3) |
| C15—C14—H14 | 126.6 | C22—N12—H12 | 126.4 |
| C14—C15—N1 | 110.2 (3) | C23—N12—H12 | 126.4 |
| C14—C15—H15 | 124.9 | N5—Ni1—N7 | 89.81 (8) |
| N1—C15—H15 | 124.9 | N5—Ni1—N1 | 90.40 (8) |
| C17—C16—N3 | 109.3 (3) | N7—Ni1—N1 | 89.69 (8) |
| C17—C16—H16 | 125.4 | N5—Ni1—N3 | 89.52 (8) |
| N3—C16—H16 | 125.4 | N7—Ni1—N3 | 177.57 (8) |
| N4—C17—C16 | 107.3 (3) | N1—Ni1—N3 | 92.65 (8) |
| N4—C17—H17 | 126.4 | N5—Ni1—N11 | 179.43 (8) |
| C16—C17—H17 | 126.4 | N7—Ni1—N11 | 90.73 (8) |
| N3—C18—N4 | 111.5 (3) | N1—Ni1—N11 | 89.78 (8) |
| N3—C18—H18 | 124.3 | N3—Ni1—N11 | 89.93 (8) |
| N4—C18—H18 | 124.3 | N5—Ni1—N9 | 90.79 (8) |
| N5—C19—N6 | 111.9 (3) | N7—Ni1—N9 | 89.38 (8) |

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|---|---|---|---|
| N5—C19—H19 | 124.0 | N1—Ni1—N9 | 178.48 (8) |
| N6—C19—H19 | 124.0 | N3—Ni1—N9 | 88.30 (8) |
| C19—N6—C20 | 107.1 (2) | N11—Ni1—N9 | 89.04 (8) |
| C19—N6—H6 | 126.5 | C3—C4—S1 | 106.7 (10) |
| C20—N6—H6 | 126.5 | C3—C4—H4 | 126.7 |
| C20—C21—N5 | 109.6 (3) | S1—C4—H4 | 126.7 |
| C20—C21—H21 | 125.2 | C1—S1—C4 | 92.1 (5) |
| N5—C21—H21 | 125.2 | C8—C7—S2 | 107.0 (8) |
| N11—C22—N12 | 112.0 (3) | C8—C7—H7 | 126.5 |
| N11—C22—H22 | 124.0 | S2—C7—H7 | 126.5 |
| N12—C22—H22 | 124.0 | C10—S2—C7 | 92.2 (4) |
| N12—C23—C24 | 106.7 (3) | C3—S1′—C4′ | 97.7 (11) |
| N12—C23—H23 | 126.6 | C1—C4′—S1′ | 99.2 (15) |
| C24—C23—H23 | 126.6 | C1—C4′—H4′ | 130.4 |
| C23—C24—N11 | 109.5 (3) | S1′—C4′—H4′ | 130.4 |
| C23—C24—H24 | 125.2 | C10—C7′—S2′ | 104.2 (16) |
| N11—C24—H24 | 125.2 | C10—C7′—H7′ | 127.9 |
| N7—C25—N8 | 112.5 (3) | S2′—C7′—H7′ | 127.9 |
| N7—C25—H25 | 123.7 | C8—S2′—C7′ | 94.9 (11) |
| N8—C25—H25 | 123.7 | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N12—H12···O4$^{i}$ | 0.86 | 2.56 | 3.124 (3) | 124 |
| N12—H12···O3$^{i}$ | 0.86 | 1.97 | 2.826 (3) | 170 |
| N10—H10*A*···O3$^{ii}$ | 0.86 | 1.88 | 2.718 (3) | 164 |
| N8—H8*A*···O2$^{iii}$ | 0.86 | 1.81 | 2.660 (3) | 170 |
| N4—H4*A*···O4$^{iv}$ | 0.86 | 1.89 | 2.688 (3) | 153 |
| N2—H2···O1$^{v}$ | 0.86 | 1.91 | 2.749 (3) | 166 |
| N6—H6···O1$^{ii}$ | 0.86 | 1.90 | 2.711 (3) | 156 |

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+2; (ii) *x*+1, *y*, *z*; (iii) −*x*+2, −*y*+2, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+2; (v) −*x*+1, −*y*+2, −*z*+1.