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# Crystal structure of $[\text{NiHg}(\text{SCN})_4(\text{CH}_3\text{OH})_2]_n$

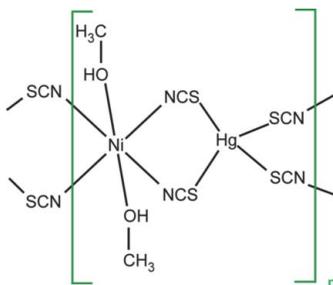
Matthias Weil\* and Thomas Häusler

 Institute for Chemical Technologies and Analytics, Division of Structural Chemistry, Vienna University of Technology,  
Getreidemarkt 9/164-SC, A-1060 Vienna, Austria. \*Correspondence e-mail: mweil@mail.zserv.tuwien.ac.at

The title compound, *catena*-poly[[bis(methanol- $\kappa$ O)nickel(II)]-di- $\mu$ -thiocyanato- $\kappa^4$ N:S-mercurate(II)-di- $\mu$ -thiocyanato- $\kappa^4$ N:S], was obtained from a gel-growth method using tetramethoxysilane as gelling agent. The crystal structure is composed of rather regular  $\text{HgS}_4$  tetrahedra (point group symmetry  $2_2$ ) and *trans*- $\text{NiN}_4\text{O}_2$  octahedra (point group symmetry  $2_2$ ) that are linked through thiocyanato bridges into a three-dimensional framework. The methanol molecules coordinate *via* the O atom to the  $\text{Ni}^{2+}$  cations and point into the voids of this arrangement while a weak O—H $\cdots$ S hydrogen bond to an adjacent S atom stabilizes it.

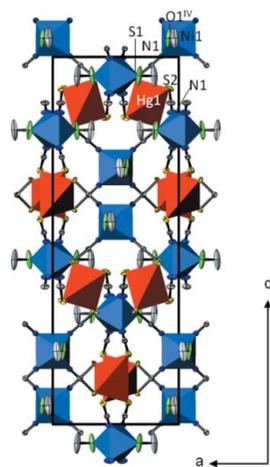
## 1. Chemical context

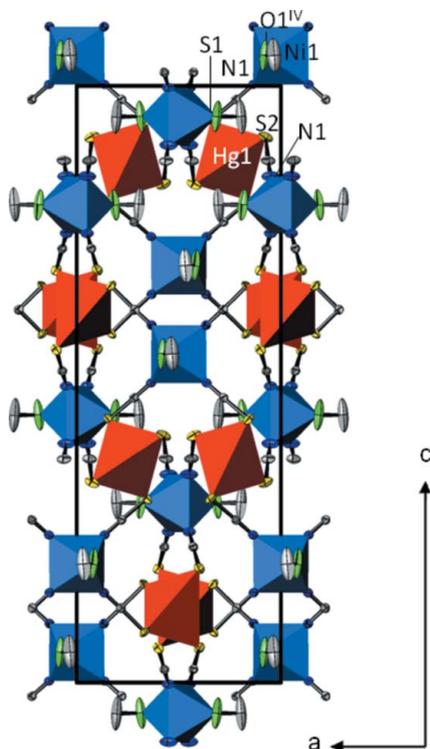
Compounds of the type  $M\text{Hg}(\text{SCN})_4$  ( $M$  is a divalent transition metal) exhibit interesting physical properties. For example,  $\text{CoHg}(\text{SCN})_4$  is a calibrant for magnetic susceptibility measurements using the Faraday method (Brown *et al.*, 1977), and representatives with  $M = \text{Fe}$ ,  $\text{Mn}$ ,  $\text{Zn}$  and  $\text{Cd}$  show second-order non-linear optical (NLO) properties (Bergman *et al.*, 1970; Yan *et al.*, 1999).



Most of the  $M\text{Hg}(\text{SCN})_4$  compounds have been structurally characterized, including  $\text{MnHg}(\text{SCN})_4$ ,  $\text{FeHg}(\text{SCN})_4$  (Yan *et al.*, 1999),  $\text{CoHg}(\text{SCN})_4$  (Jeffery & Rose, 1968),  $\text{CuHg}(\text{SCN})_4$  (Porai Koshits, 1963; Khandar *et al.*, 2011),  $\text{ZnHg}(\text{SCN})_4$  (Xu *et al.*, 1999) and  $\text{CdHg}(\text{SCN})_4$  (Iizuka & Sudo, 1968). The crystal structure of  $\text{NiHg}(\text{SCN})_4$  has not been reported up to now, and only the structures of the related hydrous phase  $[\text{NiHg}(\text{SCN})_4(\text{H}_2\text{O})_2]_n$  (Porai Koshits, 1960) and of the mercury-richer phase  $\text{NiHg}_2(\text{SCN})_6$  (Iizuka, 1978) have been determined.

In an attempt to grow crystals of the desired compound  $\text{NiHg}(\text{SCN})_4$  using a gel-growth method (Henisch, 1996), starting from TMOS (tetramethoxysilane) as gelling agent, we obtained the title compound,  $[\text{NiHg}(\text{SCN})_4(\text{CH}_3\text{OH})_2]_n$  *viz.* a methanol-containing phase, instead. Methanol is generated during the gelling process of the silicate-based material according to the idealized reaction  $(\text{H}_3\text{CO})_4\text{Si} + 4 \text{H}_2\text{O} \rightarrow 4 \text{H}_4\text{SiO}_4 + 4 \text{H}_3\text{COH}$  and then becomes part of the crystal structure.

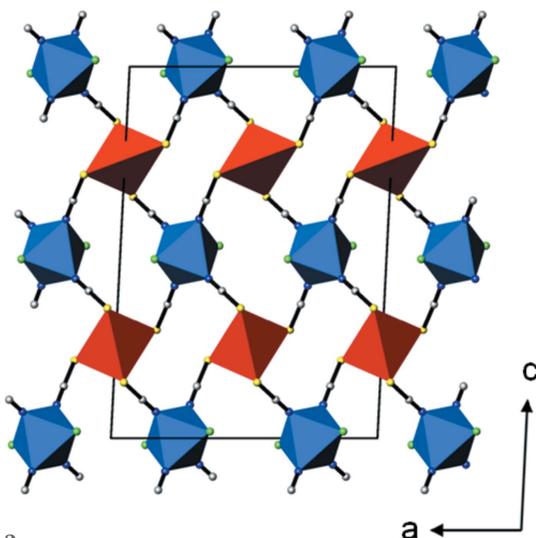




**Figure 1**  
The crystal structure of  $[\text{NiHg}(\text{SCN})_4(\text{CH}_3\text{OH})_2]$  in a projection along  $[010]$ . Displacement ellipsoids are drawn at the 90% probability level. H atoms are omitted for clarity. [Symmetry code: (iv)  $-x, -y + 1, z$ .]

## 2. Structural commentary

The basic structure units of  $[\text{NiHg}(\text{SCN})_4(\text{CH}_3\text{OH})_2]_n$  are  $\text{HgS}_4$  tetrahedra (point group symmetry  $T_d$ ) and  $\text{trans-NiN}_4\text{O}_2$  octahedra (point group symmetry  $C_{2v}$ ) that are linked through the bridging thiocyanate anions into a three-dimensional framework structure (Fig. 1). The Hg—S bond lengths [mean 2.552 (3) Å; Table 1] are in very good agreement compared with those of  $\text{HgS}_4$  tetrahedra in the above-mentioned



**Figure 2**  
The crystal structure of  $[\text{NiHg}(\text{SCN})_4(\text{H}_2\text{O})_2]$  (Porai Koshits, 1960) in a projection along  $[010]$ . Colour code as in Fig. 1.

**Table 1**  
Selected bond lengths (Å).

Hg1—S1 <sup>i</sup>	2.5499 (7)	Ni1—N2 <sup>iii</sup>	2.041 (2)
Hg1—S1	2.5499 (7)	Ni1—N1 <sup>iv</sup>	2.045 (2)
Hg1—S2	2.5546 (7)	Ni1—N1	2.045 (2)
Hg1—S2 <sup>i</sup>	2.5546 (7)	Ni1—O1	2.066 (2)
Ni1—N2 <sup>ii</sup>	2.041 (2)	Ni1—O1 <sup>iv</sup>	2.066 (2)

Symmetry codes: (i)  $-x + \frac{1}{2}, y, -z + \frac{7}{4}$ ; (ii)  $y - 1, x + \frac{1}{2}, z + \frac{1}{4}$ ; (iii)  $-y + 1, -x + \frac{1}{2}, z + \frac{1}{4}$ ; (iv)  $-x, -y + 1, z$ .

**Table 2**  
Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
O1—H1···S1 <sup>v</sup>	0.90 (1)	2.40 (2)	3.262 (2)	160 (4)

Symmetry code: (v)  $-y, x, -z + 2$ .

solvent-free  $\text{MHg}(\text{SCN})_4$  structures, which have a mean of 2.57 (5) Å. The *trans*- $\text{NiN}_4\text{O}_2$  octahedra are defined by four N atoms belonging to four bridging thiocyanate anions and by two O atoms of isolated methanol molecules. The displacement parameters of the methanol molecule are rather high. The methanol molecule has relatively much space for libration, because it is not part of the framework structure and points into the remaining free space. Thus the displacement ellipsoids of the methanol O and especially of the C atom are enlarged (Fig. 1). Moreover, there is only a weak hydrogen-bonding interaction to an adjacent S atom that stabilizes this arrangement (Table 2).

$[\text{NiHg}(\text{SCN})_4(\text{CH}_3\text{OH})_2]_n$  and  $[\text{NiHg}(\text{SCN})_4(\text{H}_2\text{O})_2]_n$  have a similar composition. Although the basic structure units ( $\text{HgS}_4$  tetrahedra and *trans*- $\text{NiN}_4\text{O}_2$  octahedra linked by thiocyanate bridges) are the same, the corresponding crystal structures are markedly different. The methanol-containing structure has tetragonal symmetry and is non-centrosymmetric, the water-containing structure has monoclinic symmetry and is centrosymmetric (space group  $C2/c$ ). Whereas in the water-containing structure the  $\text{HgS}_4$  and  $\text{NiN}_4\text{O}_2$  polyhedra are alternately arranged in layers parallel to (001) (Fig. 2), the arrangement in the methanol-containing compound is markedly different (Fig. 1).

The common structural motif in the above-mentioned  $\text{MHg}(\text{SCN})_4$  compounds is the linkage of  $\text{MN}_4$  units (planar configuration for Cu and tetrahedral for all other *M* members) and tetrahedral  $\text{HgS}_4$  units through thiocyanate bridges. It seems that a coordination number of four is not favoured for structures with  $M = \text{Ni}$ . In the structures of  $[\text{NiHg}(\text{SCN})_4(\text{CH}_3\text{OH})_2]_n$ ,  $[\text{NiHg}(\text{SCN})_4(\text{H}_2\text{O})_2]_n$  and  $\text{NiHg}_2(\text{SCN})_6$ , the  $\text{Ni}^{2+}$  ions all have coordination numbers of six, which is probably the reason why a compound with composition  $\text{NiHg}(\text{SCN})_4$  (most probably requiring a [4]-coordination for  $\text{Ni}^{2+}$ ) has not yet been isolated.

## 3. Synthesis and crystallization

$\text{Hg}(\text{SCN})_2$  was prepared by adding stoichiometric amounts of KSCN to a slightly acidified aqueous solution of  $\text{Hg}(\text{NO}_3)_2$ .

The colourless precipitate was filtered off, washed with water and dried.

For the gel-growth experiment, 1.2 g Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and 1.2 g NH<sub>4</sub>SCN were dissolved in 20 ml water. To this solution, 0.5 g freshly prepared Hg(SCN)<sub>2</sub> was slowly added until complete dissolution. Then 2 ml TMOS was added dropwise under stirring. Gelling time was about 3 h. After one week, blue single crystals of the title compound up to 5 mm in length had formed in the gel matrix.

#### 4. Refinement

The H atom of the methanol hydroxy group was located from a difference map and was refined with a distance restraint of 0.90 (1) Å. The H atoms associated with the methyl group of the methanol molecule could not be located from difference Fourier maps. As a result of the high libration of this molecule, it seems probable that the methyl H atoms are disordered and were therefore refined with two positions with half-occupancy and rotated by 60 degrees.  $U_{\text{eq}}$  of these H atoms were set 1.5 $U_{\text{iso}}$  of the parent C atom. The remaining maximum and minimum electron densities are found 0.36 and 0.06 Å<sup>-3</sup>, respectively, from atom O1. Reflection (011) was affected by the beamstop and was discarded from the refinement. Experimental details are given in Table 3.

#### Acknowledgements

The X-ray centre of the Vienna University of Technology is acknowledged for providing access to the single-crystal diffractometer.

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**Table 3**  
Experimental details.

Crystal data	
Chemical formula	[NiHg(NCS) <sub>4</sub> (CH <sub>4</sub> O) <sub>2</sub> ]
$M_r$	555.70
Crystal system, space group	Tetragonal, $I\bar{4}2d$
Temperature (K)	100
$a, c$ (Å)	10.1746 (3), 29.5107 (11)
$V$ (Å <sup>3</sup> )	3055.02 (17)
$Z$	8
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	11.81
Crystal size (mm)	0.18 × 0.18 × 0.18
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2008)
$T_{\text{min}}, T_{\text{max}}$	0.567, 0.748
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	17699, 4684, 4214
$R_{\text{int}}$	0.028
( $\sin \theta/\lambda$ ) <sub>max</sub> (Å <sup>-1</sup> )	0.904
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.026, 0.051, 0.96
No. of reflections	4684
No. of parameters	87
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	1.63, -1.46
Absolute structure	Flack (1983), 2098 Friedel pairs
Absolute structure parameter	0.011 (4)

Computer programs: *APEX2* and *SAINT* (Bruker, 2008), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *ATOMS for Windows* (Dowty, 2006).

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## supporting information

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## Crystal structure of $[\text{NiHg}(\text{SCN})_4(\text{CH}_3\text{OH})_2]_n$

Matthias Weil and Thomas Häusler

### Computing details

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINTE* (Bruker, 2008); data reduction: *SAINTE* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ATOMS for Windows* (Dowty, 2006); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008).

### catena-Poly[[bis(methanol- $\kappa$ O)nickel(II)]-di- $\mu$ -thiocyanato- $\kappa^4$ N:S-mercurate(II)-di- $\mu$ -thiocyanato- $\kappa^4$ N:S]

#### Crystal data

$[\text{NiHg}(\text{NCS})_4(\text{CH}_4\text{O})_2]$

$M_r = 555.70$

Tetragonal,  $I4_2d$

Hall symbol: I -4 2bw

$a = 10.1746$  (3) Å

$c = 29.5107$  (11) Å

$V = 3055.02$  (17) Å<sup>3</sup>

$Z = 8$

$F(000) = 2080$

$D_x = 2.416$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å

Cell parameters from 9082 reflections

$\theta = 2.9\text{--}40.0^\circ$

$\mu = 11.81$  mm<sup>-1</sup>

$T = 100$  K

Spherical, blue

$0.18 \times 0.18 \times 0.18$  mm

#### Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  and  $\phi$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2008)

$T_{\min} = 0.567$ ,  $T_{\max} = 0.748$

17699 measured reflections

4684 independent reflections

4214 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.028$

$\theta_{\max} = 40.0^\circ$ ,  $\theta_{\min} = 2.8^\circ$

$h = -17 \rightarrow 17$

$k = -18 \rightarrow 12$

$l = -41 \rightarrow 53$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.026$

$wR(F^2) = 0.051$

$S = 0.96$

4684 reflections

87 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

$w = 1/[\sigma^2(F_o^2) + (0.P)^2]$

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

$(\Delta/\sigma)_{\max} = 0.004$

$\Delta\rho_{\max} = 1.63$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -1.46$  e Å<sup>-3</sup>

Absolute structure: Flack (1983), 2098 Friedel  
pairs

Absolute structure parameter: 0.011 (4)

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 ( $\text{\AA}^2$ )

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Hg1	0.2500	0.529525 (12)	0.8750	0.01652 (3)	
Ni1	0.0000	0.5000	1.050596 (14)	0.01130 (8)	
S1	0.34284 (7)	0.39974 (6)	0.94163 (2)	0.01777 (12)	
S2	0.07460 (8)	0.66758 (7)	0.91464 (2)	0.02210 (14)	
C1	0.0628 (3)	0.7838 (2)	0.87530 (12)	0.0204 (4)	
C2	0.2196 (2)	0.4289 (2)	0.97739 (8)	0.0141 (4)	
N1	0.1356 (2)	0.4484 (2)	1.00263 (8)	0.0177 (4)	
N2	0.0514 (3)	0.8668 (2)	0.84917 (8)	0.0256 (5)	
O1	-0.0812 (2)	0.3139 (2)	1.05077 (16)	0.0593 (11)	
C3	-0.0338 (4)	0.1958 (4)	1.0511 (3)	0.075 (2)	
H2A	-0.1061	0.1322	1.0511	0.112*	0.50
H2B	0.0198	0.1834	1.0783	0.112*	0.50
H2C	0.0207	0.1825	1.0241	0.112*	0.50
H2D	0.0624	0.1999	1.0513	0.112*	0.50
H2E	-0.0635	0.1487	1.0240	0.112*	0.50
H2F	-0.0644	0.1495	1.0782	0.112*	0.50
H1	-0.164 (2)	0.331 (5)	1.0596 (16)	0.061 (14)*	

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Hg1	0.02513 (7)	0.01081 (5)	0.01362 (5)	0.000	0.00653 (5)	0.000
Ni1	0.00883 (17)	0.01668 (19)	0.00839 (15)	0.00111 (13)	0.000	0.000
S1	0.0170 (3)	0.0176 (3)	0.0187 (3)	0.0054 (2)	0.0082 (2)	0.0037 (2)
S2	0.0323 (4)	0.0168 (3)	0.0173 (3)	0.0118 (3)	0.0094 (3)	0.0071 (2)
C1	0.0318 (12)	0.0153 (10)	0.0141 (9)	0.0070 (9)	0.0014 (11)	-0.0010 (10)
C2	0.0144 (10)	0.0145 (9)	0.0133 (10)	0.0015 (8)	-0.0006 (8)	0.0005 (8)
N1	0.0153 (9)	0.0255 (11)	0.0121 (8)	0.0009 (8)	-0.0008 (7)	-0.0011 (8)
N2	0.0478 (16)	0.0150 (10)	0.0141 (9)	0.0107 (10)	-0.0019 (10)	-0.0006 (8)
O1	0.0126 (10)	0.0178 (11)	0.147 (4)	0.0008 (8)	0.0117 (16)	0.0146 (16)
C3	0.030 (2)	0.0187 (16)	0.176 (7)	0.0003 (15)	-0.011 (3)	-0.011 (3)

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Hg1—S1 <sup>i</sup>	2.5499 (7)	C1—N2	1.149 (4)
Hg1—S1	2.5499 (7)	C2—N1	1.151 (3)

Hg1—S2	2.5546 (7)	N2—Ni1 <sup>v</sup>	2.041 (2)
Hg1—S2 <sup>i</sup>	2.5546 (7)	O1—C3	1.295 (4)
Ni1—N2 <sup>ii</sup>	2.041 (2)	O1—H1	0.897 (10)
Ni1—N2 <sup>iii</sup>	2.041 (2)	C3—H2A	0.9800
Ni1—N1 <sup>iv</sup>	2.045 (2)	C3—H2B	0.9800
Ni1—N1	2.045 (2)	C3—H2C	0.9800
Ni1—O1	2.066 (2)	C3—H2D	0.9800
Ni1—O1 <sup>iv</sup>	2.066 (2)	C3—H2E	0.9800
S1—C2	1.666 (2)	C3—H2F	0.9800
S2—C1	1.661 (3)		
S1 <sup>i</sup> —Hg1—S1	117.62 (3)	C1—N2—Ni1 <sup>v</sup>	170.1 (3)
S1 <sup>i</sup> —Hg1—S2	112.27 (2)	C3—O1—Ni1	134.5 (2)
S1—Hg1—S2	100.98 (2)	C3—O1—H1	122 (3)
S1 <sup>i</sup> —Hg1—S2 <sup>i</sup>	100.98 (2)	Ni1—O1—H1	101 (3)
S1—Hg1—S2 <sup>i</sup>	112.27 (2)	O1—C3—H2A	109.5
S2—Hg1—S2 <sup>i</sup>	113.29 (3)	O1—C3—H2B	109.5
N2 <sup>ii</sup> —Ni1—N2 <sup>iii</sup>	90.77 (13)	H2A—C3—H2B	109.5
N2 <sup>ii</sup> —Ni1—N1 <sup>iv</sup>	88.42 (9)	O1—C3—H2C	109.5
N2 <sup>iii</sup> —Ni1—N1 <sup>iv</sup>	179.17 (9)	H2A—C3—H2C	109.5
N2 <sup>ii</sup> —Ni1—N1	179.17 (9)	H2B—C3—H2C	109.5
N2 <sup>iii</sup> —Ni1—N1	88.42 (9)	O1—C3—H2D	109.5
N1 <sup>iv</sup> —Ni1—N1	92.40 (12)	H2A—C3—H2D	141.1
N2 <sup>ii</sup> —Ni1—O1	88.12 (13)	H2B—C3—H2D	56.3
N2 <sup>iii</sup> —Ni1—O1	91.68 (14)	H2C—C3—H2D	56.3
N1 <sup>iv</sup> —Ni1—O1	88.11 (13)	O1—C3—H2E	109.5
N1—Ni1—O1	92.08 (13)	H2A—C3—H2E	56.3
N2 <sup>ii</sup> —Ni1—O1 <sup>iv</sup>	91.68 (14)	H2B—C3—H2E	141.1
N2 <sup>iii</sup> —Ni1—O1 <sup>iv</sup>	88.12 (13)	H2C—C3—H2E	56.3
N1 <sup>iv</sup> —Ni1—O1 <sup>iv</sup>	92.08 (13)	H2D—C3—H2E	109.5
N1—Ni1—O1 <sup>iv</sup>	88.11 (13)	O1—C3—H2F	109.5
O1—Ni1—O1 <sup>iv</sup>	179.7 (3)	H2A—C3—H2F	56.3
C2—S1—Hg1	96.75 (9)	H2B—C3—H2F	56.3
C1—S2—Hg1	97.01 (10)	H2C—C3—H2F	141.1
N2—C1—S2	177.4 (3)	H2D—C3—H2F	109.5
N1—C2—S1	179.0 (2)	H2E—C3—H2F	109.5
C2—N1—Ni1	173.3 (2)		

Symmetry codes: (i)  $-x+1/2, y, -z+7/4$ ; (ii)  $y-1, x+1/2, z+1/4$ ; (iii)  $-y+1, -x+1/2, z+1/4$ ; (iv)  $-x, -y+1, z$ ; (v)  $-y+1/2, -x+1, z-1/4$ .

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ S1 <sup>vi</sup>	0.90 (1)	2.40 (2)	3.262 (2)	160 (4)

Symmetry code: (vi)  $-y, x, -z+2$ .