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Bis(O-ethyl dithiocarbonato- κ^2S,S')bis-(pyridine-3-carbonitrile- κN^1)nickel(II)

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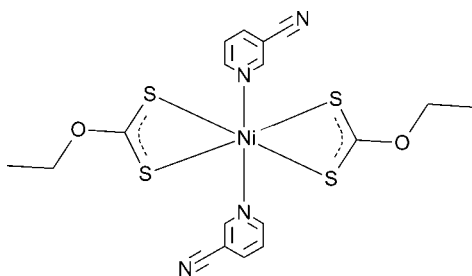
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(C-C) = 0.003$ Å; R factor = 0.025; wR factor = 0.060; data-to-parameter ratio = 14.4.

The Ni²⁺ ion in the title complex, [Ni(C₃H₅OS₂)₂(C₆H₄N₂)₂], is in a strongly distorted octahedral coordination environment formed by an N₂S₄ donor set, with the Ni²⁺ ion located on a centre of inversion. In the crystal, weak C—H...S and C—H...N interactions are observed.

Related literature

For related structures, see: Tiekink & Haiduc (2005); Dakternieks *et al.* (2006); Hill & Tiekink (2007); Hogarth *et al.* (2009)



Experimental

Crystal data

[Ni(C₃H₅OS₂)₂(C₆H₄N₂)₂] $M_r = 509.31$ Monoclinic, $P2_1/c$ $a = 6.7302$ (2) Å $b = 18.8959$ (5) Å $c = 8.7242$ (2) Å $\beta = 95.916$ (2)° $V = 1103.58$ (5) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 1.28$ mm⁻¹ $T = 293$ K $0.3 \times 0.3 \times 0.1$ mm

Data collection

Oxford Diffraction Xcalibur
Sapphire3 diffractometer
Absorption correction: multi-scan
(*CrysAlis PRO*; Oxford
Diffraction, 2007)
 $T_{\min} = 0.728$, $T_{\max} = 1.000$

18847 measured reflections
1930 independent reflections
1723 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.025$ $wR(F^2) = 0.060$ $S = 1.07$

1930 reflections

134 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.23$ e Å⁻³ $\Delta\rho_{\min} = -0.22$ e Å⁻³

Table 1

Selected bond lengths (Å).

Ni1—N1	2.1273 (17)	Ni1—S2	2.4450 (6)
Ni1—S1	2.4335 (5)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9A...S2 ⁱ	0.97	2.85	3.455 (2)	121
C10—H10C...N2 ⁱⁱ	0.96	2.65	3.595 (4)	169

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2007); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2009) and *PARST* (Nardelli, 1995).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GK2438).

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Bis(*O*-ethyl dithiocarbonato- κ^2S,S')bis(pyridine-3-carbonitrile- κN^1)nickel(II)

Sanjay Kapoor, Ramandeep Kour, Renu Sachar, Rajni Kant, Vivek K. Gupta and Kamini Kapoor

S1. Comment

Xanthates (*O*-alkyl/aryl dithiocarbonates) have been known for a long time and many adducts of metal xanthates with different ligands have been prepared and studied in the last several decades. Adducts of transition metal xanthates with N-donor ligands are well represented in the literature, the most extensively studied being those of nickel(II). Nitrogen containing adducts of nickel(II) xanthates are known to adopt a variety of supramolecular assemblies (Tiekink & Haiduc, 2005). The Ni atom in (I) is located on a center of inversion and exists within a *trans*-N₂S₄ donor set that defines an approximately octahedral coordination geometry. The chelating xanthate ligand forms essentially equivalent Ni—S bond distances; this equivalence is reflected in the parameters defining the xanthate ligand. The bond angles around the Ni atom are in the range of 73.83 (2) to 180.00 (3)°. The Ni—S bond lengths, Ni1—S1 = 2.4335 (5); Ni1—S2 = 2.4450 (6) Å, are in good agreement with those reported for other Ni-dithiocarbonato complexes (Tiekink & Haiduc, 2005; Dakternieks *et al.*, 2006; Hill & Tiekink, 2007; Hogarth *et al.*, 2009). Molecules in the unit cell are packed together to form well defined layers. While no classical hydrogen bonds are present, the C—H···S and C—H···N hydrogen bonds (Table 1) play important role in stabilizing the crystal structure.

S2. Experimental

The title complex was prepared by stirring the parent nickel(II) ethylxanthate (0.781g, 0.0026 mol.) with 3-cyanopyridine- (0.541g, 0.0052 mol.) in acetone(60 ml) for one hour. Green crystals of (I) were isolated by the slow evaporation of the resulting solution of the complex.

S3. Refinement

All H atoms were positioned geometrically and were treated as riding on their parent C atoms, with C—H distances of 0.93–0.97 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methylC})$.

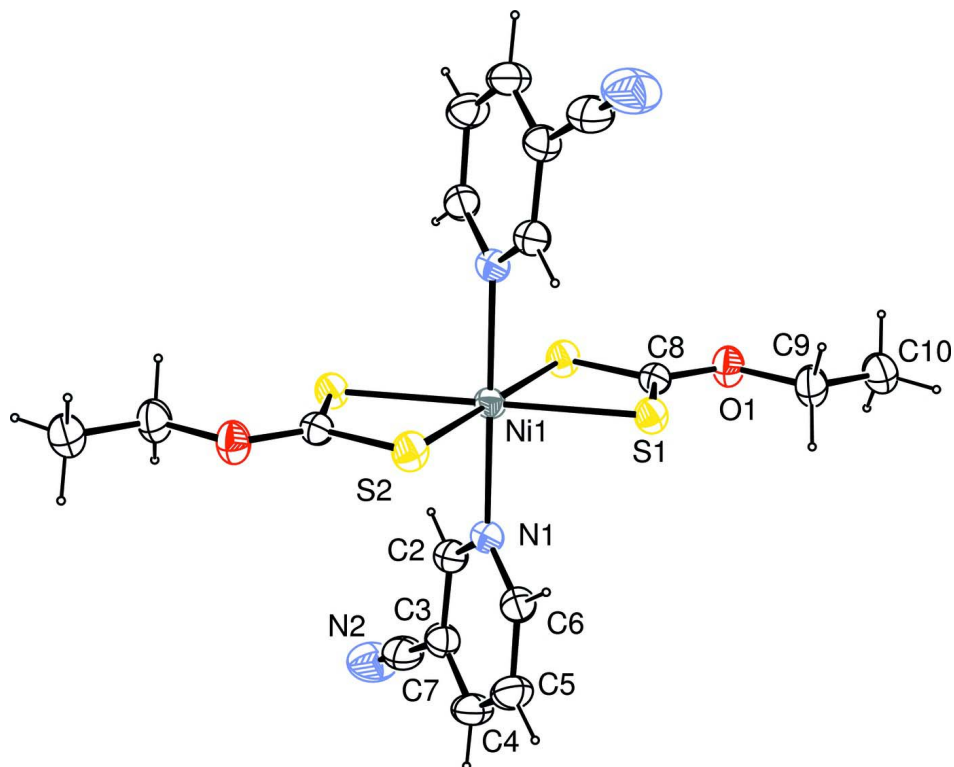


Figure 1

ORTEP view of the molecule with displacement ellipsoids drawn at the 30% probability level. Unlabeled atoms are generated by the symmetry operation $-x, 1-y, 1-z$.

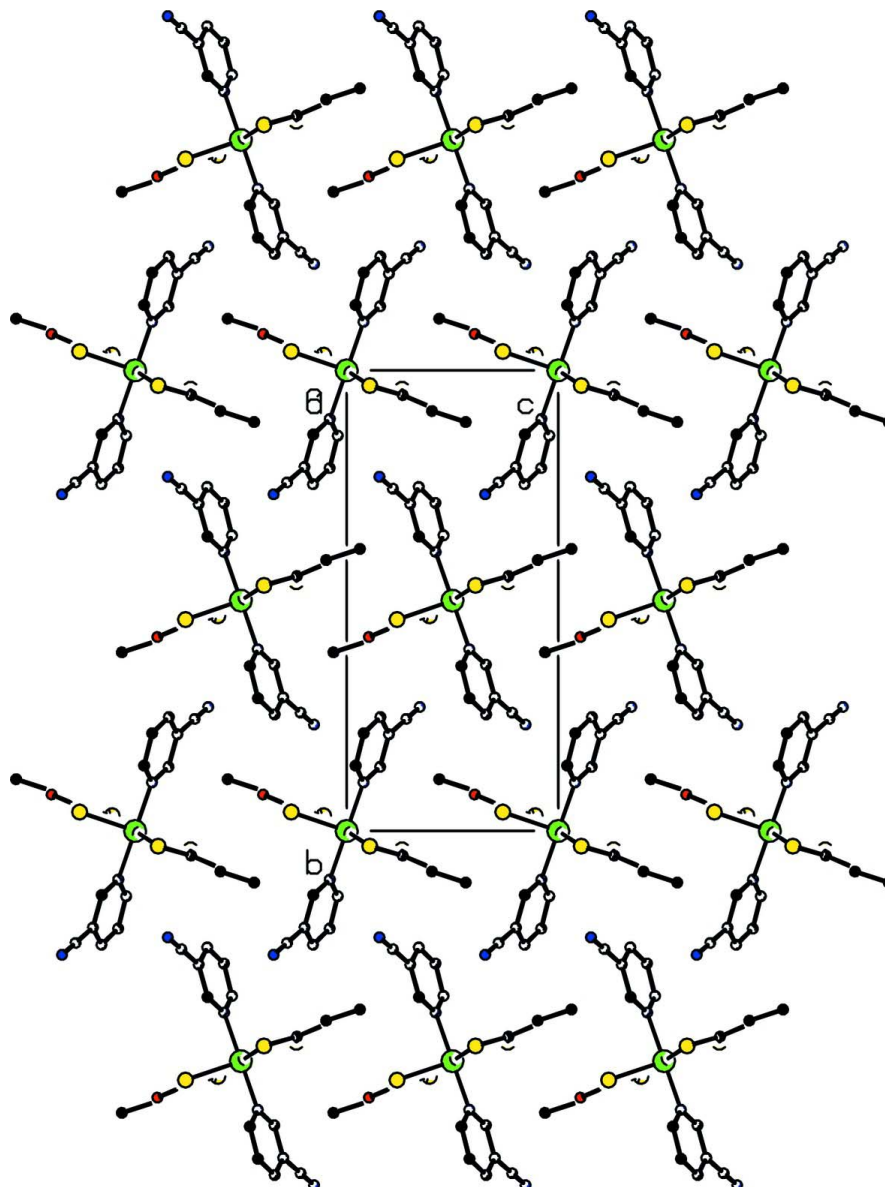


Figure 2

The packing arrangement of molecules viewed down the *a* axis.

Bis(O-ethyl dithiocarbonato- κ^2S,S')bis(pyridine-3-carbonitrile- κN^1)nickel(II)

Crystal data

$[\text{Ni}(\text{C}_3\text{H}_5\text{OS}_2)_2(\text{C}_6\text{H}_4\text{N}_2)_2]$

$M_r = 509.31$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 6.7302(2)\ \text{\AA}$

$b = 18.8959(5)\ \text{\AA}$

$c = 8.7242(2)\ \text{\AA}$

$\beta = 95.916(2)^\circ$

$V = 1103.58(5)\ \text{\AA}^3$

$Z = 2$

$F(000) = 524$

$D_x = 1.533\ \text{Mg m}^{-3}$

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

Cell parameters from 10361 reflections

$\theta = 3.6\text{--}29.0^\circ$

$\mu = 1.28\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Hexagonal plate, green

$0.3 \times 0.3 \times 0.1\ \text{mm}$

Data collection

Oxford Diffraction Xcalibur Sapphire3
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.1049 pixels mm⁻¹
 ω scans
Absorption correction: multi-scan
(*CrysAlis PRO*; Oxford Diffraction, 2007)
 $T_{\min} = 0.728$, $T_{\max} = 1.000$

18847 measured reflections
1930 independent reflections
1723 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.036$
 $\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 3.7^\circ$
 $h = -8 \rightarrow 8$
 $k = -22 \rightarrow 22$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.025$
 $wR(F^2) = 0.060$
 $S = 1.07$
1930 reflections
134 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-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0208P)^2 + 0.7659P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$

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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.0000	0.5000	0.5000	0.02943 (12)
S1	0.33453 (8)	0.46692 (3)	0.60894 (6)	0.03356 (14)
S2	0.03241 (8)	0.54100 (3)	0.23794 (7)	0.03737 (15)
O1	0.3099 (2)	0.42097 (8)	0.89474 (17)	0.0404 (4)
N1	0.0499 (3)	0.60555 (9)	0.5796 (2)	0.0340 (4)
N2	-0.3300 (4)	0.76898 (12)	0.8455 (3)	0.0691 (7)
C2	-0.0808 (3)	0.63914 (11)	0.6579 (3)	0.0368 (5)
H2	-0.1910	0.6143	0.6856	0.044*
C3	-0.0591 (3)	0.71000 (12)	0.6999 (3)	0.0401 (5)
C4	0.1050 (4)	0.74716 (13)	0.6592 (3)	0.0484 (6)
H4	0.1222	0.7947	0.6846	0.058*
C5	0.2413 (4)	0.71212 (13)	0.5805 (3)	0.0469 (6)
H5	0.3542	0.7354	0.5532	0.056*
C6	0.2086 (3)	0.64182 (12)	0.5425 (3)	0.0383 (5)
H6	0.3014	0.6186	0.4885	0.046*
C7	-0.2097 (4)	0.74323 (13)	0.7818 (3)	0.0497 (6)

C8	0.2162 (3)	0.44638 (11)	0.7649 (2)	0.0319 (5)
C9	0.5252 (3)	0.41173 (13)	0.9032 (3)	0.0446 (6)
H9A	0.5889	0.4561	0.8807	0.054*
H9B	0.5583	0.3767	0.8285	0.054*
C10	0.5962 (4)	0.38776 (14)	1.0625 (3)	0.0520 (6)
H10A	0.5554	0.4213	1.1357	0.078*
H10B	0.7392	0.3842	1.0732	0.078*
H10C	0.5395	0.3423	1.0810	0.078*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0264 (2)	0.0265 (2)	0.0358 (2)	0.00092 (15)	0.00515 (15)	0.00119 (16)
S1	0.0274 (3)	0.0369 (3)	0.0373 (3)	0.0017 (2)	0.0078 (2)	0.0051 (2)
S2	0.0275 (3)	0.0441 (3)	0.0418 (3)	0.0007 (2)	0.0097 (2)	0.0045 (2)
O1	0.0312 (8)	0.0509 (10)	0.0392 (9)	0.0036 (7)	0.0040 (7)	0.0093 (7)
N1	0.0334 (10)	0.0296 (9)	0.0387 (10)	0.0001 (8)	0.0028 (8)	0.0021 (8)
N2	0.0700 (17)	0.0489 (14)	0.0917 (19)	0.0032 (12)	0.0249 (14)	-0.0198 (13)
C2	0.0360 (12)	0.0332 (12)	0.0412 (13)	-0.0003 (9)	0.0035 (10)	-0.0004 (10)
C3	0.0443 (13)	0.0338 (12)	0.0412 (13)	0.0033 (10)	0.0001 (10)	-0.0040 (10)
C4	0.0590 (16)	0.0314 (12)	0.0535 (15)	-0.0084 (11)	-0.0004 (12)	-0.0019 (11)
C5	0.0466 (14)	0.0411 (13)	0.0531 (15)	-0.0124 (11)	0.0061 (11)	0.0036 (12)
C6	0.0351 (12)	0.0371 (12)	0.0424 (13)	-0.0012 (9)	0.0033 (10)	0.0038 (10)
C7	0.0561 (16)	0.0361 (13)	0.0565 (16)	-0.0024 (12)	0.0042 (13)	-0.0096 (12)
C8	0.0306 (11)	0.0294 (11)	0.0357 (11)	-0.0003 (9)	0.0028 (9)	0.0021 (9)
C9	0.0341 (12)	0.0503 (14)	0.0488 (14)	0.0072 (10)	0.0013 (10)	0.0061 (11)
C10	0.0525 (15)	0.0469 (15)	0.0536 (15)	0.0075 (12)	-0.0082 (12)	0.0037 (12)

Geometric parameters (Å, °)

Ni1—N1	2.1273 (17)	C3—C7	1.442 (3)
Ni1—S1	2.4335 (5)	C4—C5	1.372 (3)
Ni1—S2	2.4450 (6)	C4—H4	0.9300
S1—C8	1.691 (2)	C5—C6	1.381 (3)
S2—C8 ⁱ	1.688 (2)	C5—H5	0.9300
O1—C8	1.328 (2)	C6—H6	0.9300
O1—C9	1.454 (3)	C9—C10	1.493 (3)
N1—C2	1.329 (3)	C9—H9A	0.9700
N1—C6	1.336 (3)	C9—H9B	0.9700
N2—C7	1.138 (3)	C10—H10A	0.9600
C2—C3	1.392 (3)	C10—H10B	0.9600
C2—H2	0.9300	C10—H10C	0.9600
C3—C4	1.385 (3)		
N1 ⁱ —Ni1—N1	180.00 (3)	C2—C3—C7	119.3 (2)
N1 ⁱ —Ni1—S1	89.73 (5)	C5—C4—C3	118.4 (2)
N1—Ni1—S1	90.27 (5)	C5—C4—H4	120.8
N1 ⁱ —Ni1—S1 ⁱ	90.27 (5)	C3—C4—H4	120.8

N1—Ni1—S1 ⁱ	89.73 (5)	C4—C5—C6	119.1 (2)
S1—Ni1—S1 ⁱ	180.0	C4—C5—H5	120.5
N1 ⁱ —Ni1—S2 ⁱ	88.96 (5)	C6—C5—H5	120.5
N1—Ni1—S2 ⁱ	91.04 (5)	N1—C6—C5	123.1 (2)
S1—Ni1—S2 ⁱ	73.831 (18)	N1—C6—H6	118.4
S1 ⁱ —Ni1—S2 ⁱ	106.169 (18)	C5—C6—H6	118.4
N1 ⁱ —Ni1—S2	91.04 (5)	N2—C7—C3	179.3 (3)
N1—Ni1—S2	88.96 (5)	O1—C8—S2 ⁱ	116.53 (15)
S1—Ni1—S2	106.169 (18)	O1—C8—S1	123.21 (15)
S1 ⁱ —Ni1—S2	73.831 (18)	S2 ⁱ —C8—S1	120.26 (12)
S2 ⁱ —Ni1—S2	180.0	O1—C9—C10	107.76 (19)
C8—S1—Ni1	83.10 (7)	O1—C9—H9A	110.2
C8 ⁱ —S2—Ni1	82.80 (7)	C10—C9—H9A	110.2
C8—O1—C9	118.02 (17)	O1—C9—H9B	110.2
C2—N1—C6	117.86 (19)	C10—C9—H9B	110.2
C2—N1—Ni1	121.65 (14)	H9A—C9—H9B	108.5
C6—N1—Ni1	120.33 (15)	C9—C10—H10A	109.5
N1—C2—C3	122.5 (2)	C9—C10—H10B	109.5
N1—C2—H2	118.7	H10A—C10—H10B	109.5
C3—C2—H2	118.7	C9—C10—H10C	109.5
C4—C3—C2	119.0 (2)	H10A—C10—H10C	109.5
C4—C3—C7	121.7 (2)	H10B—C10—H10C	109.5
N1 ⁱ —Ni1—S1—C8	-88.58 (9)	C6—N1—C2—C3	0.8 (3)
N1—Ni1—S1—C8	91.42 (9)	Ni1—N1—C2—C3	-174.57 (16)
S2 ⁱ —Ni1—S1—C8	0.42 (7)	N1—C2—C3—C4	-0.1 (3)
S2—Ni1—S1—C8	-179.58 (7)	N1—C2—C3—C7	178.6 (2)
N1 ⁱ —Ni1—S2—C8 ⁱ	90.39 (9)	C2—C3—C4—C5	-0.9 (4)
N1—Ni1—S2—C8 ⁱ	-89.61 (9)	C7—C3—C4—C5	-179.6 (2)
S1—Ni1—S2—C8 ⁱ	-179.58 (7)	C3—C4—C5—C6	1.2 (4)
S1 ⁱ —Ni1—S2—C8 ⁱ	0.42 (7)	C2—N1—C6—C5	-0.5 (3)
S1—Ni1—N1—C2	-130.63 (16)	Ni1—N1—C6—C5	174.93 (18)
S1 ⁱ —Ni1—N1—C2	49.37 (16)	C4—C5—C6—N1	-0.5 (4)
S2 ⁱ —Ni1—N1—C2	-56.80 (16)	C9—O1—C8—S2 ⁱ	-177.93 (16)
S2—Ni1—N1—C2	123.20 (16)	C9—O1—C8—S1	1.2 (3)
S1—Ni1—N1—C6	54.10 (16)	Ni1—S1—C8—O1	-179.72 (18)
S1 ⁱ —Ni1—N1—C6	-125.90 (16)	Ni1—S1—C8—S2 ⁱ	-0.67 (12)
S2 ⁱ —Ni1—N1—C6	127.93 (16)	C8—O1—C9—C10	176.01 (19)
S2—Ni1—N1—C6	-52.07 (16)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C9—H9A \cdots S2 ⁱⁱ	0.97	2.85	3.455 (2)	121
C10—H10C \cdots N2 ⁱⁱⁱ	0.96	2.65	3.595 (4)	169

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