

# Crystal structure of bis(1-hexyl-*N,N*-dimethylpyridinium) bis(maleonitriledithiolato)nickelate(II)

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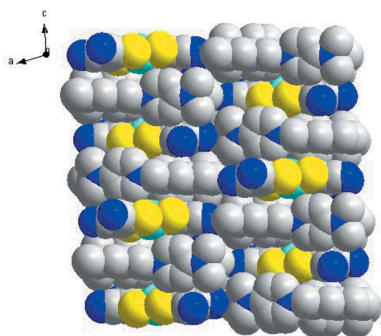
The asymmetric unit of the title compound,  $(C_{13}H_{23}N_2)_2[Ni(C_4N_2S_2)_2]$ , consists of a 1-hexyl-*N,N*-dimethylpyridinium cation and one half of a  $[Ni(mnt)_2]^{2-}$  dianion ( $mnt^{2-}$  = maleonitriledithiolate) in which the  $Ni^{2+}$  cation lies on a crystallographic inversion centre. The square-planar coordination about  $Ni^{2+}$  comprises four S atoms from two bidentate chelate  $mnt^{2-}$  ligands [ $Ni-S$  = 2.1791 (9) and 2.1810 (8) Å, and  $S-Ni-S$  bite angle = 91.93 (3)°]. The hydrocarbon chains of cations show *trans*-planar conformations and lie approximately parallel to the long molecular axis of the  $[Ni(mnt)_2]^{2-}$  anions, giving stacks along the *c* axis. The anions and cations form layers lying parallel to the *bc* plane. Only weak  $C-H \cdots Ni$  and  $C-H \cdots \pi$  associations are present in the crystal packing.

## 1. Chemical context

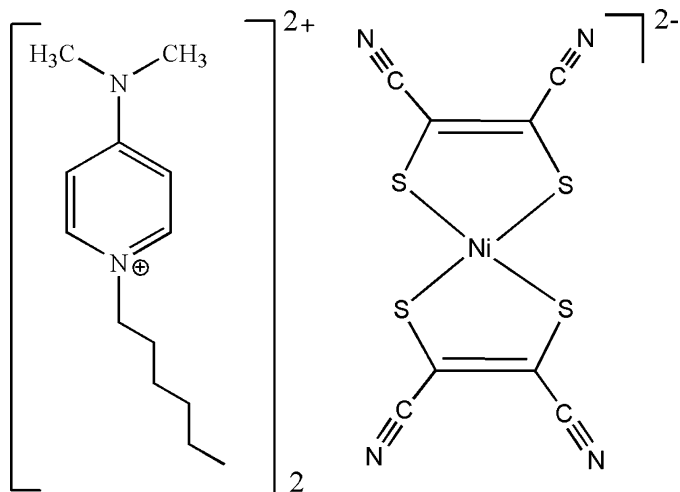
Molecular solids based on transition metal dithiolene complexes have attracted much interest in recent years, not only regarding fundamental research of magnetic interactions and magneto-structural correlations but also in the development of new functional-molecule-based materials (Robertson & Cronin, 2002). Much work has been performed in molecular solids based on  $M[dithiolene]_2$  complexes because of their application as building blocks in molecular-based materials showing magnetic, superconducting and optical properties (Nishijo *et al.*, 2000; Ni *et al.*, 2004; Ren *et al.*, 2004). In our previous studies, we have investigated the effect of the introduction of mobile organic cations into the rigid  $[Ni(mnt)_2]^{2-}$  spin system and created some multi-functional compounds (Yu *et al.*, 2012, 2013; Duan *et al.*, 2011). In order to further explore the correlation between the structural features of the counter-cations and the stacking patterns of the anions as well as their physical properties, we have designed and synthesized the soft 1-hexyl-*N,N*-dimethylpyridinium cation and combined it with the  $[Ni(mnt)_2]^{2-}$  dianion, giving the title compound,  $(C_{13}H_{23}N_2)_2[Ni(C_4N_2S_2)_2]$ , (I), and the crystal structure is reported herein.

## 2. Structural comment

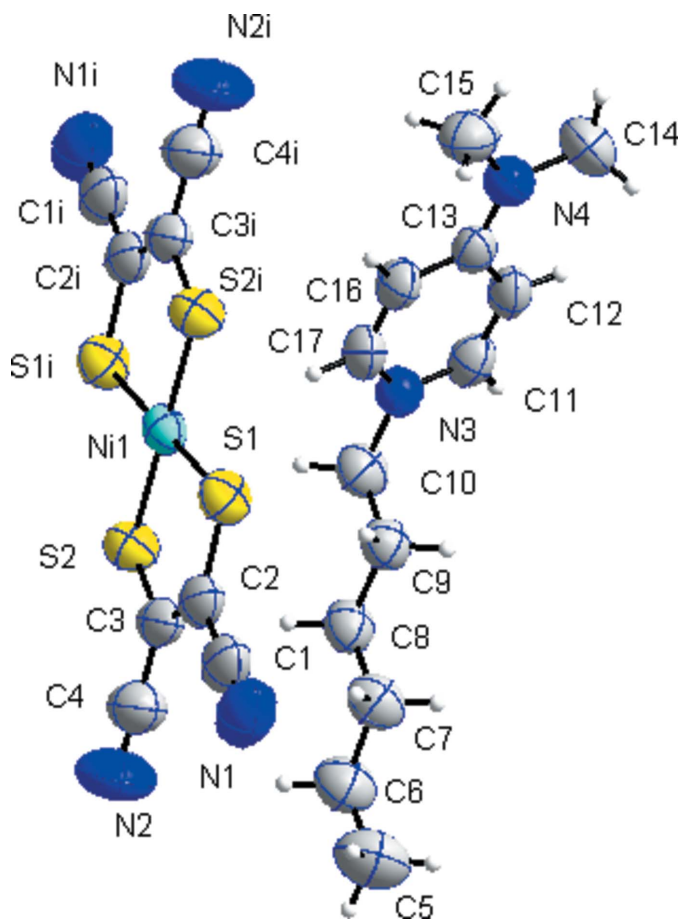
In the structure of (I) (Fig. 1), the asymmetric unit comprises a 1-hexyl-*N,N*-dimethylpyridinium cation and one half of an  $[Ni(mnt)_2]^{2-}$  dianion ( $mnt^{2-}$  = maleonitriledithiolate). The  $Ni^{2+}$  ion lies on a crystallographic inversion centre (Fig. 1). The complex dianion possesses an approximately planar geometry with  $Ni-S$  bond lengths of the bidentate ligands of 2.1791 (9) and 2.1810 (8) Å and an  $S-Ni-S_2$  bite angle of



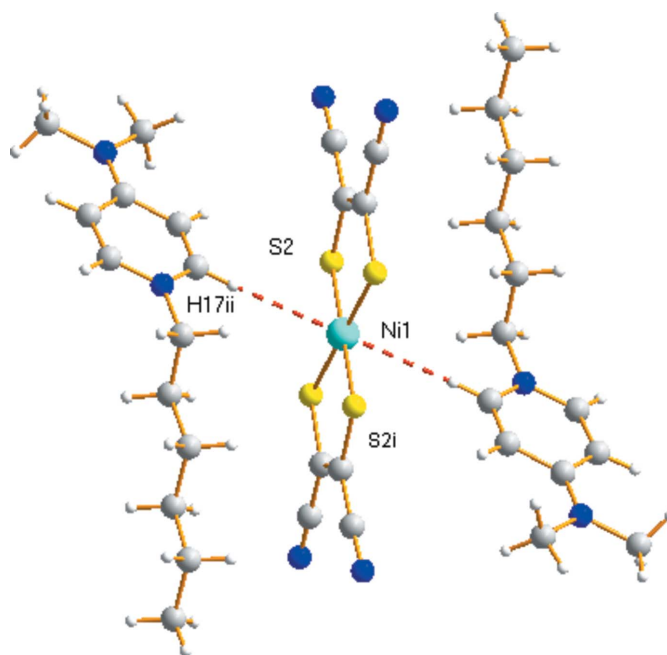
91.93 (3)°. These values are in good agreement with those found in various  $[\text{Ni}(\text{mnt})_2]^{2-}$  compounds (Duan *et al.*, 2014a).



The hydrocarbon chain of the cation is slightly disrupted close to the pyridyl ring in the completely *trans*-planar conformation, with a chain to pyridyl ring dihedral angle of



**Figure 1**  
The atom-numbering scheme in the molecular structure of (I), showing the cation and the centrosymmetric dianion, with displacement ellipsoids drawn at the 30% probability level. [Symmetry code: (i)  $-x + 1, -y, -z$ .]



**Figure 2**  
The Ni...H—C contact between the layered cation and anion species in (I). [Symmetry code: (ii)  $-x + 1, -y + 1, -z$ .]

83.03 (19)°. The direction of the hydrocarbon chains is approximately parallel to the long molecular axis of the anions, with a dihedral angle between the molecular plane of the hydrocarbon chain and that of the anion (defined by S1,S2,S2<sup>i</sup>,S1<sup>i</sup>) of 10.76 (18)° [symmetry code: (i)  $-x + 1, -y, -z$ ]. Between the cation and anion there is a novel Ni1...H—C17<sup>ii</sup> interaction (H...Ni = 2.72 Å) (Fig. 2) [symmetry code: (ii)  $-x + 1, -y + 1, -z$ ].

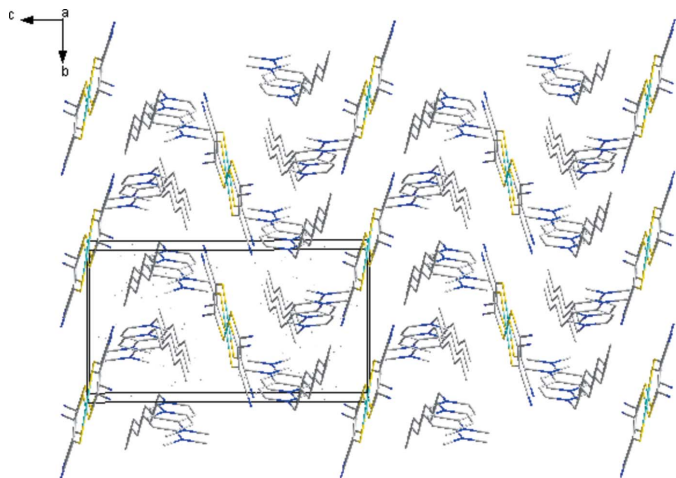
### 3. Supramolecular features

In the crystal of (I), both the anions and cations form layers lying parallel to the *bc* plane (Figs. 2 and 3). In the anion layer, two neighboring  $[\text{Ni}(\text{mnt})_2]^{2-}$  anions are associated *via* side-to-side stacking with typical interatomic separations of 8.713 (1) Å (Ni1...Ni1<sup>ii</sup>), and 6.218 (3) Å (S1...S2<sup>ii</sup>). The cations are arranged into bilayers, also lying parallel to the *ab* plane. In each layer, the cations exhibit an antiparallel arrangement. The cation and anion layers stack alternately, forming columns which extend along *c* (Fig. 4).

In the crystal there are no formal hydrogen-bonding interactions. However, there are two weak C17—H... $\pi$  associations to the chelate ring of the  $[\text{Ni}(\text{mnt})_2]^{2-}$  dianions (Cg1, defined by Ni1,S1,C2,C3,S2): to Cg1<sup>ii</sup> and Cg1<sup>iii</sup> (H...Cg = 2.77 Å) [symmetry code: (iii)  $x, y - 1, z$ ].

### 4. Database survey

In the structures of  $[\text{Ni}(\text{mnt})_2]^{2-}$  complex dianions, chair-shaped organic compounds have been chosen as counterions and a series of compounds with segregated anion and

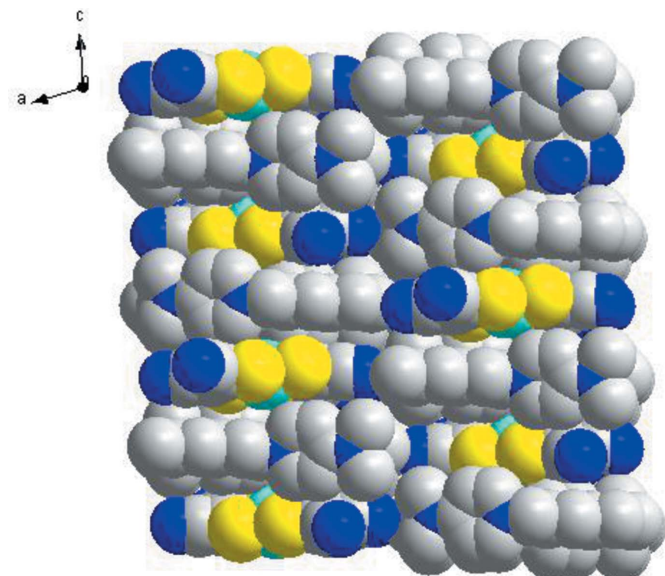


**Figure 3**  
Alternating anion and cation layers, which lie parallel to the crystallographic *ab* plane.

cation stacks have been obtained (Pei *et al.*, 2012; Tian *et al.*, 2009; Ren *et al.*, 2006). In addition, with  $[\text{Ni}(\text{mnt})_2]^{2-}$  anions, nine compounds with 1-alkyl-4-aminopyridinium analogs as counter-cations have been synthesized (Duan *et al.*, 2014*b*). In these, the hydrocarbon chains of the counter-ions adopt *trans*-planar conformations and mixed stacking structures of anions and cations are also observed

### 5. Synthesis and crystallization

Disodium maleonitriledithiolate (2.0 mmol) and nickel(II) chloride hexahydrate (1.0 mmol) were mixed with stirring in water (20 mL) at room temperature. Subsequently, a solution of 1-hexyl-*N,N*-dimethylpyridinium iodide (1.0 mmol) in methanol (10 mL) was added to the mixture and the red



**Figure 4**  
The packing diagram, viewed along *b*, of the crystals of (I).

**Table 1**  
Experimental details.

Crystal data	
Chemical formula	$(\text{C}_{13}\text{H}_{23}\text{N}_2)_2[\text{Ni}(\text{C}_4\text{N}_2\text{S}_2)_2]$
$M_r$	753.72
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
$a, b, c$ (Å)	14.241 (2), 8.7129 (14), 16.393 (3)
$\beta$ (°)	102.560 (2)
$V$ (Å <sup>3</sup> )	1985.4 (6)
$Z$	2
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	0.73
Crystal size (mm)	0.40 × 0.20 × 0.20
Data collection	
Diffractometer	Bruker SMART CCD area-detector
Absorption correction	Multi-scan ( <i>SADABS</i> ; Bruker, 2000)
$T_{\text{min}}, T_{\text{max}}$	0.811, 0.903
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	16992, 4553, 2772
$R_{\text{int}}$	0.065
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.651
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.047, 0.142, 1.00
No. of reflections	4553
No. of parameters	217
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	0.28, -0.47

Computer programs: *SMART* and *SAINT* (Bruker, 2000), *SHELXS97*, *SHELXL97* and *SHELXTL* (Sheldrick, 2008).

precipitate that was immediately formed was filtered off and washed with methanol. The crude product was recrystallized in acetone (20 mL) to give red block-shaped crystals which were used in the X-ray analysis.

### 6. Refinement

Crystal data, data collection and refinement details are summarized in Table 1. The H atoms were placed in geometrically idealized positions ( $\text{C}-\text{H} = 0.93\text{--}0.98$  Å) and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic or methylene})$  or  $1.5U_{\text{eq}}(\text{methyl})$ .

### Acknowledgements

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## supporting information

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## Crystal structure of bis(1-hexyl-*N,N*-dimethylpyridinium) bis(maleonitriledithiolato)nickelate(II)

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### Computing details

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART* (Bruker, 2000); data reduction: *SAINTE* (Bruker, 2000); 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* (Sheldrick, 2008).

(I)

### Crystal data

(C<sub>13</sub>H<sub>23</sub>N<sub>2</sub>)<sub>2</sub>[Ni(C<sub>4</sub>N<sub>2</sub>S<sub>2</sub>)<sub>2</sub>]

*M<sub>r</sub>* = 753.72

Monoclinic, *P*2<sub>1</sub>/*c*

Hall symbol: -*P* 2ybc

*a* = 14.241 (2) Å

*b* = 8.7129 (14) Å

*c* = 16.393 (3) Å

β = 102.560 (2)°

*V* = 1985.4 (6) Å<sup>3</sup>

*Z* = 2

*F*(000) = 796

*D<sub>x</sub>* = 1.261 Mg m<sup>-3</sup>

Mo *K*α radiation, λ = 0.71073 Å

Cell parameters from 773 reflections

θ = 2.6–21.2°

μ = 0.73 mm<sup>-1</sup>

*T* = 296 K

Prism, red

0.40 × 0.20 × 0.20 mm

### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan  
(SADABS; Bruker, 2000)

*T<sub>min</sub>* = 0.811, *T<sub>max</sub>* = 0.903

16992 measured reflections

4553 independent reflections

2772 reflections with *I* > 2σ(*I*)

*R<sub>int</sub>* = 0.065

θ<sub>max</sub> = 27.5°, θ<sub>min</sub> = 1.5°

*h* = -18→17

*k* = -11→11

*l* = -21→20

### Refinement

Refinement on *F*<sup>2</sup>

Least-squares matrix: full

*R*[*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.047

*wR*(*F*<sup>2</sup>) = 0.142

*S* = 1.00

4553 reflections

217 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

$$w = 1/[\sigma^2(F_o^2) + (0.0734P)^2]$$

where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$

$$\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.47 \text{ e } \text{\AA}^{-3}$$

### Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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  $F^2 > 2\text{sigma}(F^2)$  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 ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Ni1	0.5000	0.0000	0.0000	0.05898 (18)
S1	0.54081 (6)	0.24019 (9)	0.02097 (5)	0.0763 (2)
S2	0.64932 (5)	-0.07007 (9)	0.01137 (5)	0.0720 (2)
N1	0.7570 (3)	0.4849 (4)	0.0913 (2)	0.1208 (12)
N2	0.8973 (2)	0.0748 (5)	0.0871 (2)	0.1362 (13)
N3	0.55955 (17)	0.9719 (2)	0.27888 (15)	0.0658 (6)
N4	0.83752 (17)	0.8050 (3)	0.34593 (14)	0.0743 (6)
C1	0.7170 (3)	0.3720 (4)	0.07145 (18)	0.0856 (9)
C2	0.6657 (2)	0.2325 (3)	0.04658 (16)	0.0705 (7)
C3	0.7121 (2)	0.0976 (4)	0.04262 (16)	0.0689 (7)
C4	0.8156 (3)	0.0868 (4)	0.0665 (2)	0.0921 (10)
C5	0.0487 (3)	0.6987 (5)	0.1394 (3)	0.1196 (13)
H5A	0.0644	0.6439	0.0933	0.179*
H5B	-0.0169	0.7329	0.1244	0.179*
H5C	0.0568	0.6322	0.1871	0.179*
C6	0.1144 (2)	0.8359 (4)	0.1604 (2)	0.0938 (10)
H6A	0.1027	0.9054	0.1130	0.113*
H6B	0.0982	0.8898	0.2072	0.113*
C7	0.2197 (2)	0.7958 (4)	0.1820 (2)	0.0845 (9)
H7A	0.2355	0.7409	0.1353	0.101*
H7B	0.2312	0.7268	0.2296	0.101*
C8	0.2867 (2)	0.9327 (3)	0.20257 (19)	0.0748 (8)
H8A	0.2693	0.9910	0.2475	0.090*
H8B	0.2785	0.9990	0.1540	0.090*
C9	0.39199 (19)	0.8855 (3)	0.22865 (18)	0.0716 (7)
H9A	0.3997	0.8152	0.2756	0.086*
H9B	0.4103	0.8316	0.1827	0.086*
C10	0.4579 (2)	1.0208 (3)	0.2530 (2)	0.0779 (8)
H10A	0.4510	1.0907	0.2060	0.093*
H10B	0.4395	1.0754	0.2987	0.093*
C11	0.6025 (2)	0.9523 (3)	0.36010 (17)	0.0683 (7)
H11	0.5689	0.9780	0.4008	0.082*

C12	0.6928 (2)	0.8963 (3)	0.38405 (16)	0.0677 (7)
H12	0.7193	0.8825	0.4406	0.081*
C13	0.7480 (2)	0.8581 (3)	0.32450 (16)	0.0626 (7)
C14	0.8852 (2)	0.7825 (5)	0.4335 (2)	0.1024 (11)
H14A	0.8845	0.8770	0.4635	0.154*
H14B	0.9505	0.7509	0.4370	0.154*
H14C	0.8519	0.7048	0.4577	0.154*
C15	0.8934 (2)	0.7713 (4)	0.2824 (2)	0.0950 (10)
H15A	0.8617	0.6922	0.2457	0.143*
H15B	0.9567	0.7374	0.3094	0.143*
H15C	0.8983	0.8624	0.2506	0.143*
C16	0.7002 (2)	0.8802 (3)	0.23995 (16)	0.0669 (7)
H16	0.7318	0.8563	0.1975	0.080*
C17	0.6098 (2)	0.9351 (3)	0.22026 (17)	0.0706 (7)
H17	0.5806	0.9484	0.1642	0.085*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
Ni1	0.0627 (3)	0.0692 (3)	0.0450 (3)	0.0056 (2)	0.0115 (2)	-0.00043 (19)
S1	0.0775 (5)	0.0704 (4)	0.0804 (5)	0.0057 (4)	0.0161 (4)	-0.0003 (4)
S2	0.0666 (5)	0.0811 (5)	0.0697 (4)	0.0073 (4)	0.0176 (3)	-0.0089 (4)
N1	0.154 (3)	0.104 (2)	0.096 (2)	-0.047 (2)	0.011 (2)	-0.0003 (17)
N2	0.068 (2)	0.163 (3)	0.170 (4)	0.001 (2)	0.008 (2)	-0.009 (3)
N3	0.0689 (15)	0.0638 (13)	0.0647 (14)	0.0041 (11)	0.0145 (11)	0.0064 (10)
N4	0.0661 (15)	0.0834 (16)	0.0719 (15)	-0.0009 (12)	0.0114 (12)	0.0088 (12)
C1	0.100 (2)	0.095 (2)	0.0589 (17)	-0.018 (2)	0.0110 (16)	0.0051 (16)
C2	0.0776 (19)	0.0844 (19)	0.0497 (15)	-0.0099 (16)	0.0145 (13)	0.0027 (13)
C3	0.0636 (17)	0.094 (2)	0.0504 (15)	-0.0048 (15)	0.0156 (13)	0.0020 (13)
C4	0.075 (2)	0.107 (3)	0.093 (2)	-0.006 (2)	0.0149 (18)	-0.0019 (19)
C5	0.092 (3)	0.121 (3)	0.138 (3)	-0.001 (2)	0.008 (2)	-0.024 (3)
C6	0.080 (2)	0.093 (2)	0.105 (2)	0.0158 (18)	0.0139 (19)	-0.0008 (19)
C7	0.078 (2)	0.083 (2)	0.090 (2)	0.0143 (17)	0.0131 (16)	-0.0048 (17)
C8	0.0730 (19)	0.0747 (17)	0.0780 (19)	0.0143 (15)	0.0193 (15)	0.0096 (15)
C9	0.0698 (18)	0.0703 (17)	0.0746 (18)	0.0099 (14)	0.0154 (14)	0.0063 (14)
C10	0.073 (2)	0.0718 (18)	0.088 (2)	0.0142 (15)	0.0168 (17)	0.0060 (15)
C11	0.081 (2)	0.0683 (16)	0.0592 (17)	-0.0066 (14)	0.0231 (15)	-0.0001 (13)
C12	0.080 (2)	0.0693 (16)	0.0517 (15)	-0.0069 (14)	0.0110 (13)	0.0076 (12)
C13	0.0690 (18)	0.0560 (14)	0.0618 (16)	-0.0084 (13)	0.0117 (13)	0.0048 (12)
C14	0.083 (2)	0.122 (3)	0.090 (2)	0.008 (2)	-0.0071 (18)	0.019 (2)
C15	0.074 (2)	0.100 (2)	0.113 (3)	0.0033 (18)	0.0271 (19)	0.007 (2)
C16	0.0720 (18)	0.0781 (17)	0.0531 (15)	0.0012 (14)	0.0196 (13)	0.0047 (12)
C17	0.078 (2)	0.0781 (17)	0.0551 (15)	0.0001 (15)	0.0140 (14)	0.0096 (13)

*Geometric parameters (Å, °)*

Ni1—S1	2.1791 (9)	C16—C17	1.345 (4)
Ni1—S2	2.1810 (8)	C5—H5A	0.9600

Ni1—S1 <sup>i</sup>	2.1791 (9)	C5—H5B	0.9600
Ni1—S2 <sup>i</sup>	2.1810 (8)	C5—H5C	0.9600
S1—C2	1.738 (3)	C6—H6A	0.9700
S2—C3	1.731 (3)	C6—H6B	0.9700
N1—C1	1.148 (5)	C7—H7A	0.9700
N2—C4	1.144 (5)	C7—H7B	0.9700
N3—C10	1.480 (4)	C8—H8A	0.9700
N3—C11	1.350 (4)	C8—H8B	0.9700
N3—C17	1.356 (4)	C9—H9A	0.9700
N4—C14	1.462 (4)	C9—H9B	0.9700
N4—C15	1.471 (4)	C10—H10A	0.9700
N4—C13	1.330 (4)	C10—H10B	0.9700
C1—C2	1.431 (5)	C11—H11	0.9300
C2—C3	1.357 (4)	C12—H12	0.9300
C3—C4	1.444 (5)	C14—H14A	0.9600
C5—C6	1.511 (6)	C14—H14B	0.9600
C6—C7	1.505 (4)	C14—H14C	0.9600
C7—C8	1.519 (4)	C15—H15A	0.9600
C8—C9	1.524 (4)	C15—H15B	0.9600
C9—C10	1.506 (4)	C15—H15C	0.9600
C11—C12	1.351 (4)	C16—H16	0.9300
C12—C13	1.420 (4)	C17—H17	0.9300
C13—C16	1.418 (4)		
Ni1—S1—C2	103.00 (9)	C7—C6—H6B	109.00
Ni1—S2—C3	102.72 (11)	H6A—C6—H6B	108.00
S1—Ni1—S2	91.93 (3)	C6—C7—H7A	109.00
S1—Ni1—S1 <sup>i</sup>	180.00	C6—C7—H7B	109.00
S1—Ni1—S2 <sup>i</sup>	88.07 (3)	C8—C7—H7A	109.00
S1 <sup>i</sup> —Ni1—S2	88.07 (3)	C8—C7—H7B	109.00
S2—Ni1—S2 <sup>i</sup>	180.00	H7A—C7—H7B	108.00
S1 <sup>i</sup> —Ni1—S2 <sup>i</sup>	91.93 (3)	C7—C8—H8A	109.00
C11—N3—C17	118.3 (2)	C7—C8—H8B	109.00
C10—N3—C11	121.6 (2)	C9—C8—H8A	109.00
C10—N3—C17	120.0 (2)	C9—C8—H8B	109.00
C14—N4—C15	117.5 (2)	H8A—C8—H8B	108.00
C13—N4—C14	121.3 (2)	C8—C9—H9A	109.00
C13—N4—C15	121.2 (2)	C8—C9—H9B	109.00
N1—C1—C2	179.1 (4)	C10—C9—H9A	109.00
C1—C2—C3	121.7 (3)	C10—C9—H9B	109.00
S1—C2—C1	117.9 (2)	H9A—C9—H9B	108.00
S1—C2—C3	120.4 (2)	N3—C10—H10A	109.00
C2—C3—C4	121.5 (3)	N3—C10—H10B	109.00
S2—C3—C2	121.3 (2)	C9—C10—H10A	109.00
S2—C3—C4	117.2 (3)	C9—C10—H10B	109.00
N2—C4—C3	177.9 (4)	H10A—C10—H10B	108.00
C5—C6—C7	114.0 (3)	N3—C11—H11	119.00
C6—C7—C8	114.6 (3)	C12—C11—H11	119.00



C7—C8—C9	112.5 (2)	C11—C12—H12	119.00
C8—C9—C10	112.4 (2)	C13—C12—H12	119.00
N3—C10—C9	111.4 (2)	N4—C14—H14A	109.00
N3—C11—C12	122.1 (3)	N4—C14—H14B	110.00
C11—C12—C13	121.3 (2)	N4—C14—H14C	110.00
N4—C13—C12	122.8 (2)	H14A—C14—H14B	109.00
N4—C13—C16	122.3 (2)	H14A—C14—H14C	109.00
C12—C13—C16	114.9 (3)	H14B—C14—H14C	109.00
C13—C16—C17	120.9 (3)	N4—C15—H15A	109.00
N3—C17—C16	122.6 (3)	N4—C15—H15B	109.00
C6—C5—H5A	110.00	N4—C15—H15C	109.00
C6—C5—H5B	109.00	H15A—C15—H15B	110.00
C6—C5—H5C	109.00	H15A—C15—H15C	109.00
H5A—C5—H5B	109.00	H15B—C15—H15C	109.00
H5A—C5—H5C	109.00	C13—C16—H16	120.00
H5B—C5—H5C	110.00	C17—C16—H16	120.00
C5—C6—H6A	109.00	N3—C17—H17	119.00
C5—C6—H6B	109.00	C16—C17—H17	119.00
C7—C6—H6A	109.00		
S2—Ni1—S1—C2	-6.92 (10)	C14—N4—C13—C16	-179.7 (3)
S2 <sup>i</sup> —Ni1—S1—C2	173.09 (10)	C15—N4—C13—C12	-178.1 (3)
S1—Ni1—S2—C3	7.10 (10)	S1—C2—C3—S2	0.5 (3)
S1 <sup>i</sup> —Ni1—S2—C3	-172.90 (10)	S1—C2—C3—C4	-177.7 (2)
Ni1—S1—C2—C1	-174.9 (2)	C1—C2—C3—S2	-179.3 (2)
Ni1—S1—C2—C3	5.3 (2)	C1—C2—C3—C4	2.5 (4)
Ni1—S2—C3—C2	-6.1 (2)	C5—C6—C7—C8	179.5 (3)
Ni1—S2—C3—C4	172.2 (2)	C6—C7—C8—C9	176.8 (3)
C17—N3—C11—C12	-0.6 (4)	C7—C8—C9—C10	-177.2 (3)
C10—N3—C17—C16	-175.5 (2)	C8—C9—C10—N3	179.4 (2)
C11—N3—C17—C16	0.0 (4)	N3—C11—C12—C13	1.3 (4)
C10—N3—C11—C12	174.8 (2)	C11—C12—C13—N4	178.9 (3)
C11—N3—C10—C9	-96.8 (3)	C11—C12—C13—C16	-1.3 (4)
C17—N3—C10—C9	78.5 (3)	N4—C13—C16—C17	-179.6 (3)
C14—N4—C13—C12	0.0 (4)	C12—C13—C16—C17	0.7 (4)
C15—N4—C13—C16	2.2 (4)	C13—C16—C17—N3	-0.1 (4)

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