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Bis(5,8-diazoniadispiro[4.2.4.2]tetradecane) hexakis(thiocyanato-κN)-nickelate(II) dihydrate

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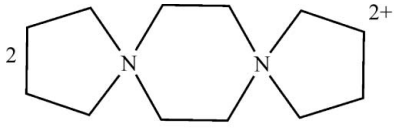
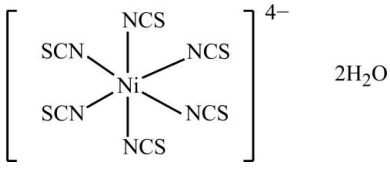
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.038; wR factor = 0.105; data-to-parameter ratio = 15.8.

The asymmetric unit of the title compound, $(C_{12}H_{24}N_2)_2[Ni(NCS)_6]\cdot 2H_2O$, comprises one cation, one half-anion and one water molecule. The anion lies on an inversion centre and shows the expected octahedral coordination with only minor deviations from the ideal geometry. Intermolecular O—H···S, O—H···N, C—H···S, C—H···O and C—H···N hydrogen-bonding interactions link all components into a three-dimensional extended network.

Related literature

For structures containing the hexakis(isothiocyanato)-nickelate(II) tetraanion see: Vicente *et al.* (1996); Kruger & McKee (1996); Hoffman & Wood (1982).





Experimental

Crystal data

$(C_{12}H_{24}N_2)_2[Ni(NCS)_6]\cdot 2H_2O$
M_r = 835.89
Monoclinic, $P2_1/n$
a = 9.959 (6) Å
b = 13.537 (8) Å
c = 14.718 (9) Å
β = 96.186 (10)°
V = 1973 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.85 mm^{-1}
T = 273 (2) K
0.30 × 0.15 × 0.15 mm

Data collection

Siemens SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.857, T_{max} = 0.884
10750 measured reflections
3660 independent reflections
3220 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.105
S = 1.06
3660 reflections
231 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C10—H10A···S3	0.97	2.88	3.767 (3)	153
C11—H11A···O1	0.97	2.59	3.269 (4)	127
C12—H12B···O1	0.97	2.60	3.463 (5)	149
O1—H1C···N2	1.05 (7)	2.40 (7)	3.273 (4)	139 (5)
C6—H6A···S3^{i}	0.97	2.77	3.587 (3)	142
C6—H6B···S2^{ii}	0.97	2.88	3.775 (4)	155
C15—H15A···N1^{iii}	0.97	2.60	3.507 (3)	156
O1—H1B···S1^{iii}	0.99 (8)	2.49 (8)	3.290 (3)	139 (6)
O1—H1C···N1^{iv}	1.05 (7)	2.52 (7)	3.435 (5)	145 (5)

Symmetry codes: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+1, -y+1, -z$; (iii) $x-1, y, z$; (iv) $-x+2, -y+1, -z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1994); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2160).

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supplementary materials

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Bis(5,8-diazoniadispiro[4.2.4.2]tetradecane) hexakis(thiocyanato-*κN*)nickelate(II) dihydrate

M.-P. Song, L.-K. Li, B.-L. Wu and Y.-Y. Niu

Comment

Organo-inorganic hybrid compounds containing the hexakis(isothiocyanato)nickel(II) tetraanion have been the subject of several investigations, but the structural examples are scarce (Vicente *et al.*, 1996; Kruger & McKee, 1996; Hoffman & Wood, 1982). In this work we present the crystal structure of a new hexakis(isothiocyanato)nickel(II) salt with the 5,8-diazoniadispiro[4.2.4.2]tetradecane dication.

The structure of the title compound (Fig. 1) comprises discrete $(C_{12}H_{24}N_2)^{2+}$ cations, $[Ni(NCS)_6]^{4-}$ anions and water molecules in the ratio 2:1:2. The anion, which lies on an inversion centre, displays the expected N^6-octahedral coordination with only minor deviations from the ideal geometry. In the cation, the six-membered ring displays a chair conformation, while the five-membered rings adopt a twist conformation. In the crystal structure, all components are linked into a three-dimensional extended network through intermolecular O—H···S, O—H···N, C—H···S, C—H···O and C—H···N hydrogen bonding interactions (Table 1, Fig. 2).

Experimental

The title salt was synthesized from the reaction of 5,8-diazoniadispiro[4.2.4.2]tetradecane dibromide (0.034 g, 0.1 mmol) in methanol (5 ml) and a mixture of $NiCl_2$ (0.013 g, 0.1 mmol) and KNCS(0.074 g, 0.4 mmol) in DMF (10 ml). The resulting mixture was set aside for the formation of green crystals (yield 40%) after several days on slow evaporation of the solvent.

Refinement

The methylene H atoms were positioned geometrically with C—H = 0.97 Å and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. The H atoms of the water molecule were located from a difference Fourier map, and their positional and isotropic displacement parameters were refined.

Figures

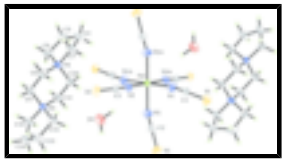

Fig. 1. Perspective view of the title compound with atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level. Unlabelled atoms and those with the suffix A are related to labelled atoms by the symmetry operation $(2 - x, 1 - y, -z)$.

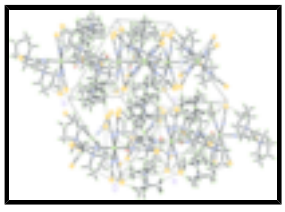

Fig. 2. Packing diagram of the title compound. Dashed lines indicate hydrogen bonding interactions.

Bis(5,8-diazoniadispiro[4.2.4.2]tetradecane) hexakis(thiocyanato-κ*N*)nickel(II) dihydrate

Crystal data

$(C_{12}H_{24}N_2)_2[Ni(NCS)_6]\cdot 2H_2O$	$F_{000} = 884$
$M_r = 835.89$	$D_x = 1.407$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2yn	Cell parameters from 5316 reflections
$a = 9.959$ (6) Å	$\theta = 2.6$–27.3º
$b = 13.537$ (8) Å	$\mu = 0.85$ mm^{-1}
$c = 14.718$ (9) Å	$T = 273$ (2) K
$\beta = 96.186$ (10)º	Block, green
$V = 1973$ (2) Å^3	0.30 × 0.15 × 0.15 mm
$Z = 2$	

Data collection

Siemen SMART CCD diffractometer	3660 independent reflections
Radiation source: fine-focus sealed tube	3220 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.024$
$T = 273$(2) K	$\theta_{max} = 25.5$º
ω scan	$\theta_{min} = 2.1$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12\rightarrow12$
$T_{min} = 0.857$, $T_{max} = 0.884$	$k = -16\rightarrow16$
10750 measured reflections	$l = -9\rightarrow17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.038$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.105$	$w = 1/[\sigma^2(F_o^2) + (0.0548P)^2 + 0.9458P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
3660 reflections	$\Delta\rho_{max} = 0.57$ e Å^{-3}
231 parameters	$\Delta\rho_{min} = -0.40$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
Ni1	1.0000	0.5000	0.0000	0.03897 (14)
S1	1.38818 (7)	0.68186 (6)	0.13836 (5)	0.0622 (2)
S2	0.81710 (8)	0.83015 (5)	−0.00780 (6)	0.0666 (2)
S3	0.88411 (6)	0.38952 (5)	0.29815 (4)	0.04961 (18)
O1	0.6492 (3)	0.5411 (2)	0.1087 (3)	0.1166 (12)
N1	1.1787 (2)	0.56492 (16)	0.05640 (15)	0.0542 (5)
N2	0.9026 (2)	0.63532 (16)	0.01364 (15)	0.0535 (5)
N3	0.9581 (2)	0.45753 (16)	0.13052 (14)	0.0502 (5)
N4	0.62283 (18)	0.20076 (14)	0.15891 (13)	0.0422 (4)
N5	0.42206 (17)	0.34300 (13)	0.21603 (12)	0.0364 (4)
C1	1.2661 (2)	0.61201 (17)	0.09023 (16)	0.0439 (5)
C2	0.8688 (2)	0.71628 (17)	0.00460 (14)	0.0394 (5)
C3	0.9277 (2)	0.42916 (16)	0.19955 (16)	0.0387 (5)
C4	0.7825 (3)	0.1334 (3)	0.0665 (2)	0.0785 (10)
H4A	0.8509	0.0824	0.0663	0.094*
H4B	0.8082	0.1886	0.0301	0.094*
C5	0.6463 (3)	0.0939 (2)	0.0289 (2)	0.0677 (8)
H5A	0.6489	0.0224	0.0242	0.081*
H5B	0.6194	0.1211	−0.0313	0.081*
C6	0.5482 (3)	0.12534 (19)	0.0955 (2)	0.0591 (7)
H6A	0.5214	0.0690	0.1300	0.071*
H6B	0.4680	0.1546	0.0630	0.071*
C7	0.7685 (3)	0.1662 (2)	0.1626 (2)	0.0613 (7)
H7A	0.8305	0.2197	0.1807	0.074*
H7B	0.7857	0.1119	0.2053	0.074*
C8	0.5808 (2)	0.20186 (18)	0.25351 (16)	0.0478 (6)
H8A	0.6426	0.2430	0.2923	0.057*
H8B	0.5859	0.1354	0.2782	0.057*
C9	0.4389 (2)	0.24057 (18)	0.25463 (17)	0.0481 (6)
H9A	0.3765	0.1965	0.2194	0.058*
H9B	0.4161	0.2408	0.3171	0.058*
C10	0.6074 (2)	0.30442 (17)	0.12066 (15)	0.0425 (5)
H10A	0.6695	0.3481	0.1565	0.051*

H10B	0.6309	0.3047	0.0584	0.051*
C11	0.4657 (2)	0.34236 (16)	0.12166 (15)	0.0397 (5)
H11A	0.4603	0.4090	0.0972	0.048*
H11B	0.4044	0.3012	0.0824	0.048*
C12	0.4972 (2)	0.4200 (2)	0.27791 (17)	0.0500 (6)
H12A	0.5775	0.3916	0.3110	0.060*
H12B	0.5235	0.4757	0.2424	0.060*
C13	0.4000 (3)	0.4514 (3)	0.3420 (2)	0.0799 (10)
H13A	0.4099	0.4107	0.3966	0.096*
H13B	0.4155	0.5198	0.3598	0.096*
C14	0.2636 (3)	0.4392 (3)	0.2928 (2)	0.0667 (8)
H14A	0.2273	0.5032	0.2732	0.080*
H14B	0.2037	0.4097	0.3329	0.080*
C15	0.2732 (2)	0.37487 (19)	0.21222 (18)	0.0503 (6)
H15A	0.2462	0.4107	0.1562	0.060*
H15B	0.2152	0.3176	0.2147	0.060*
H1B	0.601 (8)	0.604 (6)	0.099 (5)	0.20 (3)*
H1C	0.720 (7)	0.539 (5)	0.061 (4)	0.20 (3)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0368 (2)	0.0394 (2)	0.0404 (2)	0.00054 (15)	0.00290 (17)	0.00225 (16)
S1	0.0511 (4)	0.0662 (4)	0.0686 (5)	−0.0110 (3)	0.0028 (3)	−0.0142 (3)
S2	0.0639 (4)	0.0423 (4)	0.0884 (6)	0.0048 (3)	−0.0152 (4)	−0.0038 (3)
S3	0.0476 (3)	0.0591 (4)	0.0417 (3)	−0.0066 (3)	0.0031 (3)	0.0020 (3)
O1	0.0871 (19)	0.0908 (19)	0.183 (3)	0.0212 (16)	0.065 (2)	0.043 (2)
N1	0.0489 (12)	0.0552 (12)	0.0569 (13)	−0.0076 (10)	−0.0013 (10)	0.0038 (10)
N2	0.0581 (13)	0.0509 (12)	0.0510 (12)	0.0082 (10)	0.0033 (10)	0.0004 (10)
N3	0.0477 (11)	0.0547 (12)	0.0483 (12)	0.0014 (9)	0.0059 (9)	0.0048 (10)
N4	0.0379 (10)	0.0412 (10)	0.0461 (11)	0.0066 (8)	−0.0014 (8)	−0.0067 (8)
N5	0.0308 (9)	0.0411 (9)	0.0368 (9)	0.0007 (7)	0.0021 (7)	−0.0006 (7)
C1	0.0434 (13)	0.0446 (12)	0.0445 (13)	0.0024 (10)	0.0080 (10)	0.0055 (10)
C2	0.0364 (11)	0.0467 (13)	0.0342 (11)	−0.0010 (9)	0.0002 (9)	−0.0031 (9)
C3	0.0318 (10)	0.0378 (11)	0.0456 (13)	0.0006 (8)	−0.0003 (9)	−0.0036 (10)
C4	0.0634 (18)	0.100 (2)	0.074 (2)	0.0218 (17)	0.0169 (16)	−0.0242 (19)
C5	0.089 (2)	0.0620 (17)	0.0509 (16)	0.0094 (15)	0.0024 (15)	−0.0116 (13)
C6	0.0533 (15)	0.0458 (13)	0.0744 (18)	0.0057 (11)	−0.0102 (13)	−0.0163 (12)
C7	0.0425 (14)	0.0744 (18)	0.0649 (17)	0.0172 (12)	−0.0035 (12)	−0.0142 (14)
C8	0.0520 (14)	0.0454 (13)	0.0451 (13)	0.0080 (10)	0.0016 (11)	0.0114 (10)
C9	0.0470 (13)	0.0511 (13)	0.0471 (13)	−0.0005 (10)	0.0092 (10)	0.0148 (11)
C10	0.0436 (12)	0.0501 (13)	0.0350 (11)	−0.0039 (10)	0.0089 (9)	−0.0003 (9)
C11	0.0445 (12)	0.0391 (11)	0.0349 (11)	0.0008 (9)	0.0011 (9)	0.0046 (9)
C12	0.0400 (12)	0.0589 (14)	0.0504 (14)	−0.0019 (11)	0.0016 (10)	−0.0172 (12)
C13	0.0538 (16)	0.114 (3)	0.071 (2)	0.0186 (17)	0.0001 (15)	−0.0402 (19)
C14	0.0437 (14)	0.090 (2)	0.0682 (18)	0.0034 (14)	0.0135 (13)	−0.0182 (16)
C15	0.0319 (11)	0.0584 (14)	0.0593 (15)	0.0062 (10)	−0.0004 (10)	−0.0016 (12)

Geometric parameters (Å, °)

Ni1—N1	2.076 (2)	C5—H5A	0.9700
Ni1—N1^{i}	2.076 (2)	C5—H5B	0.9700
Ni1—N3	2.090 (2)	C6—H6A	0.9700
Ni1—N3^{i}	2.090 (2)	C6—H6B	0.9700
Ni1—N2	2.093 (2)	C7—H7A	0.9700
Ni1—N2^{i}	2.093 (2)	C7—H7B	0.9700
S1—C1	1.640 (3)	C8—C9	1.508 (3)
S2—C2	1.629 (3)	C8—H8A	0.9700
S3—C3	1.649 (3)	C8—H8B	0.9700
O1—H1B	0.99 (8)	C9—H9A	0.9700
O1—H1C	1.05 (7)	C9—H9B	0.9700
N1—C1	1.148 (3)	C10—C11	1.503 (3)
N2—C2	1.150 (3)	C10—H10A	0.9700
N3—C3	1.156 (3)	C10—H10B	0.9700
N4—C8	1.497 (3)	C11—H11A	0.9700
N4—C10	1.514 (3)	C11—H11B	0.9700
N4—C7	1.520 (3)	C12—C13	1.485 (4)
N4—C6	1.522 (3)	C12—H12A	0.9700
N5—C11	1.499 (3)	C12—H12B	0.9700
N5—C9	1.501 (3)	C13—C14	1.478 (4)
N5—C12	1.526 (3)	C13—H13A	0.9700
N5—C15	1.539 (3)	C13—H13B	0.9700
C4—C7	1.503 (4)	C14—C15	1.483 (4)
C4—C5	1.507 (5)	C14—H14A	0.9700
C4—H4A	0.9700	C14—H14B	0.9700
C4—H4B	0.9700	C15—H15A	0.9700
C5—C6	1.517 (4)	C15—H15B	0.9700
N1—Ni1—N1^{i}	180.0	C4—C7—H7A	111.0
N1—Ni1—N3	89.79 (9)	N4—C7—H7A	111.0
N1^{i}—Ni1—N3	90.21 (9)	C4—C7—H7B	111.0
N1—Ni1—N3^{i}	90.21 (9)	N4—C7—H7B	111.0
N1^{i}—Ni1—N3^{i}	89.79 (9)	H7A—C7—H7B	109.0
N3—Ni1—N3^{i}	180.00 (11)	N4—C8—C9	111.71 (19)
N1—Ni1—N2	88.70 (10)	N4—C8—H8A	109.3
N1^{i}—Ni1—N2	91.30 (10)	C9—C8—H8A	109.3
N3—Ni1—N2	90.68 (9)	N4—C8—H8B	109.3
N3^{i}—Ni1—N2	89.32 (9)	C9—C8—H8B	109.3
N1—Ni1—N2^{i}	91.30 (10)	H8A—C8—H8B	107.9
N1^{i}—Ni1—N2^{i}	88.70 (10)	N5—C9—C8	112.55 (19)
N3—Ni1—N2^{i}	89.32 (9)	N5—C9—H9A	109.1
N3^{i}—Ni1—N2^{i}	90.68 (9)	C8—C9—H9A	109.1
N2—Ni1—N2^{i}	180.0	N5—C9—H9B	109.1
H1B—O1—H1C	107 (5)	C8—C9—H9B	109.1

C1—N1—Ni1	170.2 (2)	H9A—C9—H9B	107.8
C2—N2—Ni1	162.9 (2)	C11—C10—N4	111.77 (18)
C3—N3—Ni1	174.8 (2)	C11—C10—H10A	109.3
C8—N4—C10	108.03 (17)	N4—C10—H10A	109.3
C8—N4—C7	109.36 (19)	C11—C10—H10B	109.3
C10—N4—C7	110.9 (2)	N4—C10—H10B	109.3
C8—N4—C6	114.2 (2)	H10A—C10—H10B	107.9
C10—N4—C6	111.73 (19)	N5—C11—C10	112.15 (17)
C7—N4—C6	102.51 (18)	N5—C11—H11A	109.2
C11—N5—C9	108.28 (17)	C10—C11—H11A	109.2
C11—N5—C12	112.61 (17)	N5—C11—H11B	109.2
C9—N5—C12	112.18 (19)	C10—C11—H11B	109.2
C11—N5—C15	110.00 (17)	H11A—C11—H11B	107.9
C9—N5—C15	109.93 (18)	C13—C12—N5	105.3 (2)
C12—N5—C15	103.79 (17)	C13—C12—H12A	110.7
N1—C1—S1	178.5 (2)	N5—C12—H12A	110.7
N2—C2—S2	178.5 (2)	C13—C12—H12B	110.7
N3—C3—S3	179.6 (2)	N5—C12—H12B	110.7
C7—C4—C5	106.2 (3)	H12A—C12—H12B	108.8
C7—C4—H4A	110.5	C14—C13—C12	106.5 (2)
C5—C4—H4A	110.5	C14—C13—H13A	110.4
C7—C4—H4B	110.5	C12—C13—H13A	110.4
C5—C4—H4B	110.5	C14—C13—H13B	110.4
H4A—C4—H4B	108.7	C12—C13—H13B	110.4
C4—C5—C6	106.2 (2)	H13A—C13—H13B	108.6
C4—C5—H5A	110.5	C13—C14—C15	108.8 (2)
C6—C5—H5A	110.5	C13—C14—H14A	109.9
C4—C5—H5B	110.5	C15—C14—H14A	109.9
C6—C5—H5B	110.5	C13—C14—H14B	109.9
H5A—C5—H5B	108.7	C15—C14—H14B	109.9
C5—C6—N4	106.2 (2)	H14A—C14—H14B	108.3
C5—C6—H6A	110.5	C14—C15—N5	106.28 (19)
N4—C6—H6A	110.5	C14—C15—H15A	110.5
C5—C6—H6B	110.5	N5—C15—H15A	110.5
N4—C6—H6B	110.5	C14—C15—H15B	110.5
H6A—C6—H6B	108.7	N5—C15—H15B	110.5
C4—C7—N4	104.0 (2)	H15A—C15—H15B	108.7
N1—Ni1—N2—C2	68.3 (7)	C15—N5—C9—C8	175.6 (2)
$N1^{i}$—Ni1—N2—C2	−111.7 (7)	N4—C8—C9—N5	−58.1 (3)
N3—Ni1—N2—C2	158.1 (7)	C8—N4—C10—C11	−56.7 (2)
$N3^{i}$—Ni1—N2—C2	−21.9 (7)	C7—N4—C10—C11	−176.57 (19)
C7—C4—C5—C6	12.4 (4)	C6—N4—C10—C11	69.7 (2)
C4—C5—C6—N4	11.9 (3)	C9—N5—C11—C10	−55.6 (2)
C8—N4—C6—C5	−149.4 (2)	C12—N5—C11—C10	69.0 (2)
C10—N4—C6—C5	87.6 (2)	C15—N5—C11—C10	−175.73 (18)
C7—N4—C6—C5	−31.2 (3)	N4—C10—C11—N5	58.4 (2)
C5—C4—C7—N4	−32.0 (3)	C11—N5—C12—C13	149.0 (2)
C8—N4—C7—C4	160.3 (2)	C9—N5—C12—C13	−88.6 (3)

C10—N4—C7—C4	−80.7 (3)	C15—N5—C12—C13	30.0 (3)
C6—N4—C7—C4	38.7 (3)	N5—C12—C13—C14	−29.0 (4)
C10—N4—C8—C9	56.3 (2)	C12—C13—C14—C15	16.5 (4)
C7—N4—C8—C9	177.1 (2)	C13—C14—C15—N5	2.7 (4)
C6—N4—C8—C9	−68.7 (3)	C11—N5—C15—C14	−140.7 (2)
C11—N5—C9—C8	55.4 (2)	C9—N5—C15—C14	100.1 (2)
C12—N5—C9—C8	−69.4 (3)	C12—N5—C15—C14	−20.0 (3)

Symmetry codes: (i) −*x*+2, −*y*+1, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C10—H10A···S3	0.97	2.88	3.767 (3)	153
C11—H11A···O1	0.97	2.59	3.269 (4)	127
C12—H12B···O1	0.97	2.60	3.463 (5)	149
O1—H1C···N2	1.05 (7)	2.40 (7)	3.273 (4)	139 (5)
C6—H6A···S3^{ii}	0.97	2.77	3.587 (3)	142
C6—H6B···S2^{iii}	0.97	2.88	3.775 (4)	155
C15—H15A···N1^{iv}	0.97	2.60	3.507 (3)	156
O1—H1B···S1^{iv}	0.99 (8)	2.49 (8)	3.290 (3)	139 (6)
O1—H1C···N1^{i}	1.05 (7)	2.52 (7)	3.435 (5)	145 (5)

Symmetry codes: (ii) −*x*+3/2, *y*−1/2, −*z*+1/2; (iii) −*x*+1, −*y*+1, −*z*; (iv) *x*−1, *y*, *z*; (i) −*x*+2, −*y*+1, −*z*.

Fig. 1

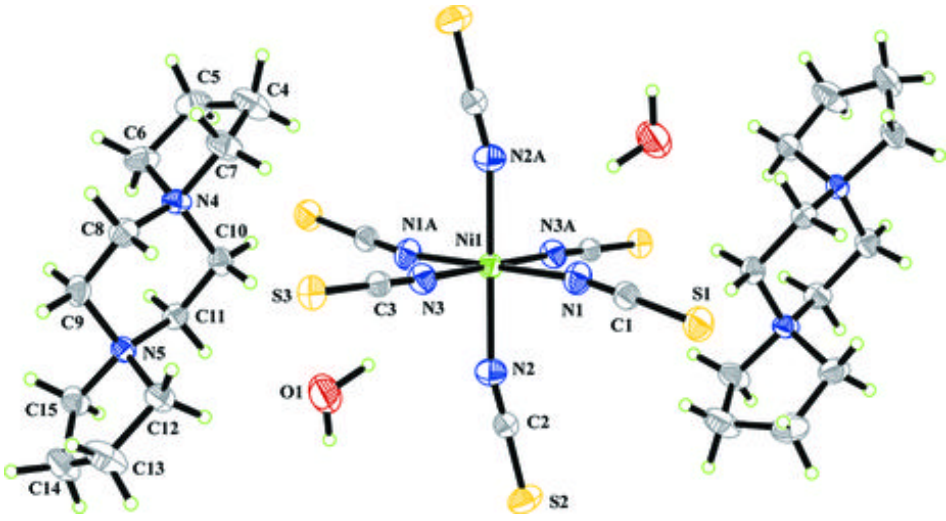
C5
C4
C6
C7
N4
C8
C10
C9
C11
N5
C15
C12
C13
C14
N2A
N1A
Ni1
N3A
S3
C3
N3
N1
C1
S1
O1
N2
C2
S2

Fig. 2