Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

A one-dimensional polymeric nickel(II) complex of an imino nitroxide radical with thiocyanate bridges

Shu-Ping Wang, Dong-Zhao Gao, Dai-Zheng Liao and Zong-Hui Jiang*

Department of Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail: coord@nankai.edu.cn

Received 7 April 2004
Accepted 21 April 2004
Online 30 April 2004

Key indicators

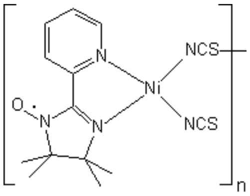
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.051
wR factor = 0.112
Data-to-parameter ratio = 14.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, *catena*-poly[[[2-(2-pyridinyl)-4,4,5,5-tetramethylimidazolin-1-oxyl]thiocyanatonickel(II)]-μ-thiocyanato], $[Ni(NCS)_2(C_{12}H_{16}N_3O)]_n$, was prepared from the imino nitroxide radical (IM2py), nickel(II) nitrate and potassium thiocyanate. The crystal structure consists of neutral chains of [Ni(IM2py)(SCN)] units linked by further thiocyanate as bridges. The Ni—NCS—Ni chains form a zigzag structure. Each Ni^{II} ion is located in a distorted square-pyramidal environment. The basal plane positions are occupied by two N atoms from the imino nitroxide radical and two thiocyanate N atoms, one from the terminal thiocyanate group and the other from the bridging thiocyanate group. The apical position is occupied by an S atom from the bridging thiocyanate group.

Comment

Nitroxide radicals are widely used as units in the synthesis of molecular-based magnetic materials. In particular, they can act as ligands in the formation of metal–radical complexes (Vostrikova *et al.*, 2000; Oshio *et al.*, 2002). On the other hand, pseudo-halide anions have attracted considerable attention in recent years because of their strong ability to coordinate transition metals and to act as magnetic couplers, giving rise to new functional materials with very rich structural topology (Manson *et al.*, 1999). By combining the two approaches, the title novel Ni^{II} complex, (I), with the imino nitroxide radical (IM2py) and thiocyanate bridges, has been prepared.



(I)

The structure of complex (I) is shown in Fig. 1. It consists of neutral chains of [Ni(IM2py)(SCN)] units linked by thiocyanate bridges. The Ni—NCS—Ni backbone forms a zigzag polymeric chain along the crystallographic a axis (Fig. 2), with an intrachain Ni···Ni distance of 5.943 (6) Å. Each Ni^{II} ion is located in a distorted square-pyramidal environment. The basal plane positions are occupied by two N atoms from the imino nitroxide radical [Ni—N = 2.018 (4) and 2.036 (4) Å] and two thiocyanate N atoms, one from the terminal thiocyanate [Ni—N = 1.942 (6) Å] and the other from the bridging thiocyanate group [Ni—N = 1.961 (6) Å; Table 1]. The Ni^{II} ion is displaced by 0.2289 (7) Å above the basal plane. The apical

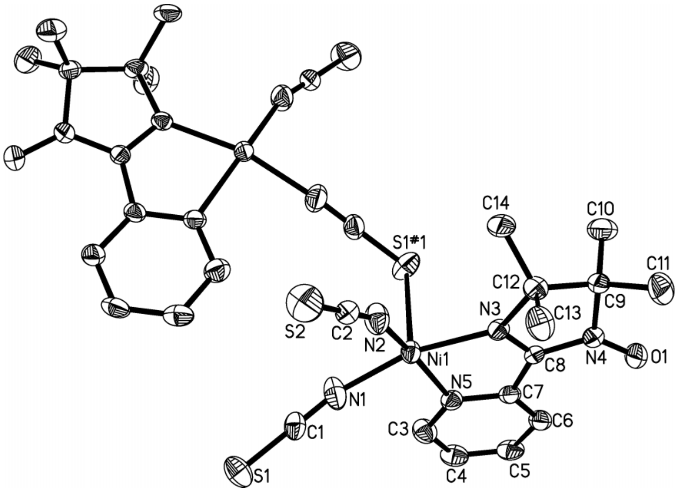


Figure 1
A view of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity. The atom labelled with the suffix #1 is at symmetry position ($x-\frac{1}{2}, \frac{5}{2}-y, -z$).

position is occupied by an S atom from the bridging thiocyanate group [Ni—S = 2.679 (3) Å]. The N3/C8/N4/O1 plane containing the unpaired electron is roughly coplanar with the basal plane [dihedral angle 7.5 (7)°].

Experimental

The title compound was prepared by adding an aqueous solution (10 ml) of KSCN (0.2 mmol) to a methanol solution (10 ml) of IM2py (0.1 mmol) and nickel(II) nitrate (0.1 mmol). The mixture was stirred for 1 h and then filtered. The filtrate was slowly evaporated at room temperature, and green crystals of (I) suitable for X-ray analysis were obtained. Analysis calculated for $C_{14}H_{16}N_5NiOS_2$: C 42.77, H 4.10, N 17.81%; found: C 42.76, H 4.06, N 17.77%.

Crystal data

$[Ni(NCS)_2(C_{12}H_{16}N_3O)]$
$M_r = 393.15$
Orthorhombic, $P2_12_12_1$
$a = 8.364$ (4) Å
$b = 9.463$ (5) Å
$c = 22.411$ (12) Å
$V = 1773.9$ (16) Å^3
$Z = 4$
$D_x = 1.472$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 695 reflections
$\theta = 3.0$–20.8°
$\mu = 1.34$ mm^{-1}
$T = 293$ (2) K
Block, green
0.40 × 0.36 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.645$, $T_{\max} = 0.818$
6085 measured reflections

3118 independent reflections
2353 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.044$
$\theta_{\max} = 25.0°$
$h = -9 \rightarrow 8$
$k = -11 \rightarrow 6$
$l = -20 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.112$
$S = 1.08$
3118 reflections
212 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0456P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.88$ e Å^{-3}
$\Delta\rho_{\min} = -0.40$ e Å^{-3}
Absolute structure: Flack (1983); 1305 Friedel pairs
Flack parameter = −0.03 (3)

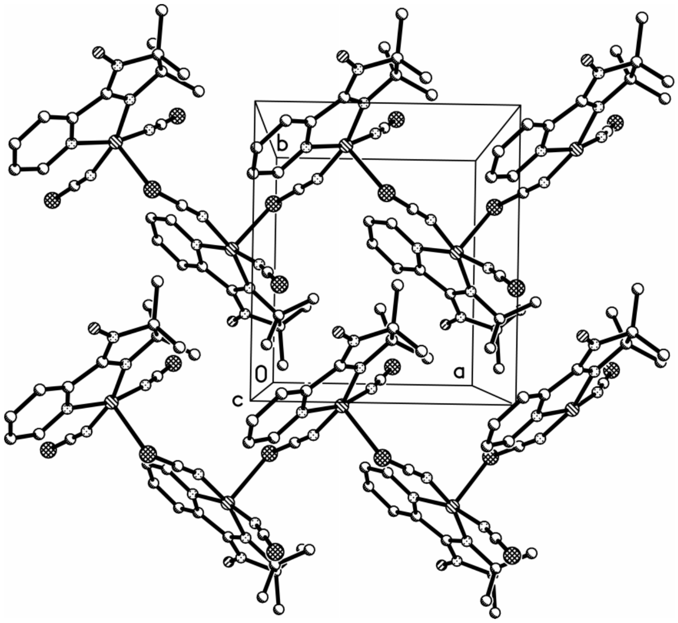


Figure 2
The packing of the polymeric molecules of (I), viewed down the *c* axis.

Table 1
Selected geometric parameters (Å, °).

Ni1—N2	1.942 (6)	N2—C2	1.156 (7)
Ni1—N1	1.961 (6)	C2—S2	1.601 (7)
Ni1—N3	2.018 (4)	N3—C8	1.279 (7)
Ni1—N5	2.036 (4)	N3—C12	1.514 (7)
Ni1—S1$^{\rm i}$	2.680 (2)	N4—C8	1.363 (7)
O1—N4	1.267 (5)	N4—C9	1.503 (7)
N1—C1	1.153 (8)	N5—C7	1.339 (7)
C1—S1	1.614 (7)	N5—C3	1.359 (7)

N2—Ni1—N1	89.7 (2)	C1—N1—Ni1	161.6 (6)
N2—Ni1—N3	96.2 (2)	C1—S1—Ni1$^{\rm ii}$	99.6 (2)
N1—Ni1—N3	165.8 (2)	C2—N2—Ni1	162.8 (5)
N2—Ni1—N5	165.4 (2)	C8—N3—Ni1	114.0 (4)
N1—Ni1—N5	91.4 (2)	C12—N3—Ni1	136.5 (4)
N3—Ni1—N5	79.69 (18)	O1—N4—C8	127.1 (5)
N2—Ni1—S1$^{\rm i}$	106.30 (19)	O1—N4—C9	123.8 (5)
N1—Ni1—S1$^{\rm i}$	102.2 (2)	C8—N4—C9	108.6 (4)
N3—Ni1—S1$^{\rm i}$	88.60 (14)	C7—N5—Ni1	115.9 (4)
N5—Ni1—S1$^{\rm i}$	87.70 (14)	C3—N5—Ni1	125.6 (4)

Symmetry codes: (i) $x-\frac{1}{2}, \frac{5}{2}-y, -z$; (ii) $\frac{1}{2}+x, \frac{5}{2}-y, -z$.

H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and were allowed to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(parent atom).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXL*97.

This work was supported by the National Natural Science Foundation of China (grant Nos. 20171025 and 20331010) and Tianjin Natural Science Foundation (grant No. 033602011).

Acta Cryst. (2004). E**60**, m682–m684 Shu-Ping Wang *et al.* • $[Ni(NCS)_2(C_{12}H_{16}N_3O)]$ **m683**

References

Bruker (1998). *SMART*, *SAINT* and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Manson, J. L., Arif, A. M. & Miller, J. S. (1999). *J. Chem. Soc. Chem. Commun.* pp. 1479–1480.

Oshio, H., Yamamoto, M. & Ito, T. (2002). *Inorg. Chem.* **41**, 5817–5820.

Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Vostrikova, K. E., Luneau, D., Wernsdorfer, W., Rey, P. & Verdaguer, M. (2000). *J. Am. Chem. Soc.* **122**, 718–719.

m684 Shu-Ping Wang *et al.* • [Ni(NCS)$_2$(C$_{12}$H$_{16}$N$_3$O)] *Acta Cryst.* (2004). E**60**, m682–m684