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Di-μ-thiocyanato-κ^2S:N;κ^2N:S-bis{[N,N-dimethyl-N'-(2-pyridylmethylene-κN)ethane-1,2-diamine-κ^2N,N']nickel(II)} bis(perchlorate)

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In the title centrosymmetric dinuclear complex, $[Ni_2(NCS)_2(C_{10}H_{15}N_3)_2](ClO_4)_2$, each Ni^{II} ion is pentacoordinated by the pyridine N, imine N, and amine N atoms of the Schiff base ligand, and by the terminal N and S atoms from two bridging thiocyanate ligands, forming a slightly distorted square-pyramidal coordination geometry.

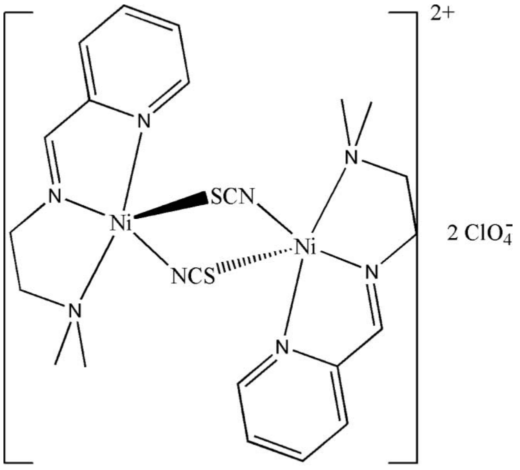
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.013 Å
Disorder in solvent or counterion
R factor = 0.078
wR factor = 0.215
Data-to-parameter ratio = 14.0

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

Comment

Nickel(II) complexes with Schiff base ligands have received much attention in recent years (Marganian *et al.*, 1995). Some of these complexes have pharmacological and antitumor properties (Harrop *et al.*, 2003; Brückner *et al.*, 2000; Ren *et al.*, 2002). In addition, nickel is present in the active sites of several important classes of metalloproteins, as either a homodinuclear or a heterodinuclear species. In a further development of the coordination chemistry of such nickel complexes, the crystal structure of the title nickel(II) complex, (I), is reported.



(I)

The asymmetic unit of (I) consists of one-half of a centrosymmetric dinuclear nickel(II) cation and a perchlorate anion (Fig. 1). Each Ni^{II} ion is five-coordinate, forming a slightly disorted square-pyramidal geometry, by a pyridine N, an imine N and an amine N atom from a Schiff base ligand and by a terminal N atom from a bridging thiocyanate ligand, forming the basal plane. A terminal S atom of a second bridging thiocyanate ligand completes the coordination in the apical position. The bond distances (Table 1) at the metal center are comparable with the values observed in other Schiff base–nickel(II) complexes (Arıcı *et al.*, 2005; Usman *et al.*, 2003; Van Hecke *et al.*, 2007; Li *et al.*, 2007).

Experimental

N,*N*-Dimethylethane-1,2-diamine (0.5 mmol, 44.0 mg), pyridine-2-carbaldehyde (0.5 mmol, 53.5 mg), ammonium thiocyanate (0.5 mmol, 38.2 mg) and $Ni(ClO_4)_2{\cdot}6H_2O$ (0.5 mmol, 183.0 mg) were dissolved in methanol (50 ml). The mixture was stirred at room temperature for 30 min, giving a green solution. After allowing the solution to stand in air for a week, green block-like crystals were formed.

Crystal data

$[Ni_2(NCS)_2(C_{10}H_{15}N_3)_2](ClO_4)_2$
$M_r = 786.96$
Monoclinic, $P2_1/c$
$a = 6.348$ (1) Å
$b = 24.834$ (3) Å
$c = 10.134$ (1) Å
$\beta = 98.442$ (2)°
$V = 1580.3$ (4) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 1.55$ mm^{-1}
$T = 298$ (2) K
0.15 × 0.15 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.801$, $T_{\max} = 0.836$
17206 measured reflections
3293 independent reflections
2366 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.059$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.078$
$wR(F^2) = 0.215$
$S = 1.04$
3293 reflections
236 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 2.00$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N4	1.923 (5)	Ni1—N3	2.047 (5)
Ni1—N2	1.933 (5)	Ni1—S1$^{\rm i}$	2.8238 (16)
Ni1—N1	2.025 (5)		
N4—Ni1—N2	168.6 (2)	N1—Ni1—N3	162.5 (2)
N4—Ni1—N1	97.3 (2)	N1—Ni1—S1$^{\rm i}$	88.1 (2)
N2—Ni1—N1	80.7 (2)	N2—Ni1—S1$^{\rm i}$	94.6 (2)
N4—Ni1—N3	98.0 (2)	N3—Ni1—S1$^{\rm i}$	98.6 (2)
N2—Ni1—N3	82.6 (2)	N4—Ni1—S1$^{\rm i}$	96.6 (2)

Symmetry code: (i) $-x+1, -y+1, -z+2$.

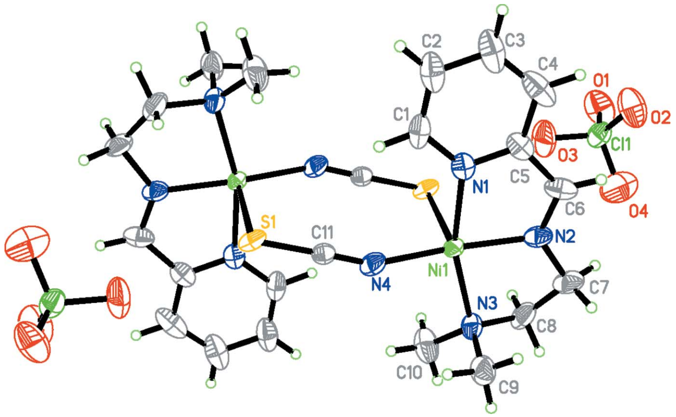


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. Unlabeled atoms are related to labeled atoms by the symmetry operation ($1-x$, $1-y$, $2-z$). Only the major component of the disordered perchlorate anions is shown.

Analysis of the data using *PLATON* (Spek, 2003) revealed that the crystal was a non-merohedral twin and a twin rotation matrix (0.997 0 −0.013, 0−1 0, −0.499 0 −0.997) was applied. In subsequent refinements the value of the weighted *R*-factor (for all data) improved from 0.376 to 0.214. The twin fraction refined to 0.974 (2):0.026 (2). All H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å, and with $U_{\rm iso}$(H) = 1.2 or $1.5U_{\rm eq}$(C). The O atoms of the unique perchlorate anion are disordered over two distinct sites. The occupancies were intially refined and then fixed at 0.58 and 0.42. The maximum residual peak in the final difference Fourier map is 1.04 Å from S1, although no disorder related to this atom is indicated. Only 95% of the data, to a maximum of 25° in θ, were available and this may lower the precision of the structure.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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References

Arıcı, C., Yüzer, D., Atakol, O., Fuess, H. & Svoboda, I. (2005). *Acta Cryst.* E**61**, m919–m921.
Brückner, C., Rettig, S. J. & Dolphin, D. (2000). *Inorg. Chem.* **39**, 6100–6106.
Bruker (2000). *SMART* (Version 5.625), *SAINT* (Version 6.01). *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Harrop, T. C., Olmstead, M. M. & Mascharak, P. K. (2003). *Chem. Commun.* pp. 410–411.
Li, J.-M., Jiang, Y.-M., Li, C.-Z. & Zhang, S.-H. (2007). *Acta Cryst.* E**63**, m447–m449.
Marganian, C. A., Vazir, H., Baidya, N., Olmstead, M. M. & Mascharak, P. K. (1995). *J. Am. Chem. Soc.* **117**, 1584–1594.
Ren, S., Wang, R., Komatsu, K., Bonaz-Krause, P., Zyrianov, Y., McKenna, C. E., Csipke, C., Tokes, Z. A. & Lien, E. J. (2002). *J. Med. Chem.* **45**, 410–419.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Usman, A., Fun, H.-K., Karmakar, T. K., Ghosh, B. K. & Chandra, S. K. (2003). *Acta Cryst.* E**59**, m387–m389.
Van Hecke, K., Nockemann, P., Binnemans, K. & Van Meervelt, L. (2007). *Acta Cryst.* E**63**, m569–m571.