Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[2,2′-(Ethylenedioxy)dibenzaldehyde bis(thiosemicarbazone)]nickel(II) diperchlorate methanol disolvate

Xiao-Hang Qiu* and Hong-Yan Wu

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

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

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.052
wR factor = 0.158
Data-to-parameter ratio = 15.7

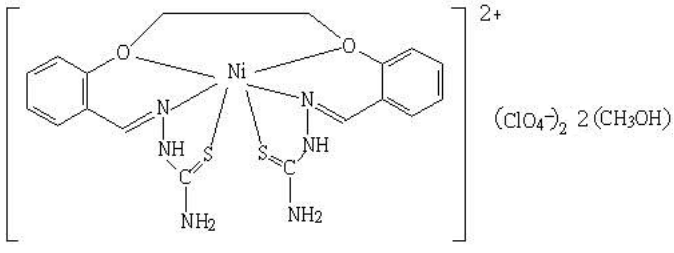
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 24 June 2004
Accepted 14 July 2004
Online 24 July 2004

The title compound, $[Ni(C_{18}H_{18}N_6O_2S_2)](ClO_4)_2\cdot 2CH_4O$, (I), has been prepared by the reaction of 2,2′-(ethylenedioxy)-dibenzaldehyde bis(thiosemicarbazone), *L*, with nickel(II) perchlorate. The chemical structural unit, which is twice the asymmetric unit of (I), consists of one Ni^{II} ion, one ligand *L*, two methanol solvent molecules and two uncoordinated perchlorate anions. The Ni atom lies on a crystallographic twofold axis of rotation. The ligand *L* coordinates to the central Ni atom through two O atoms, two S atoms and two N atoms, giving a distorted octahedral environment. The S and O atoms occupy equatorial positions, and are nearly coplanar with the nickel, while the two N atoms occupy axial positions, with the N—Ni—N angle being 177.06 (18)°.

Comment

Schiff bases and their metal complexes are of importance as a result of their antibacterial activity and antitumor properties (Yang *et al.*, 2000). Therefore, the synthesis of new Schiff bases and their metal complexes has become popular. In this study, we have prepared a new nickel(II) Schiff base complex, (I), from nickel(II) perchlorate and the hexadentate ligand 2,2′-(ethylenedioxy)dibenzaldehyde bis(thiosemicarbazone), and determined its crystal structure.



(I)

The molecular structure of (I) is shown in Fig. 1. The cation is disposed about a crystallographic twofold axis of symmetry. The structure adopts a distorted octahedral geometry about the Ni atom in which equatorial positions are occupied by two S atoms [S1 and S1^{i}; symmetry code: (i) $1-x, \frac{1}{2}-y, z$] and two O atoms (O1 and O1^{i}). The axial positions are occupied by two of the semicarbazone N atoms (N1 and N1^{i}). The Ni—N1, Ni—O1 and Ni—S1 bond distances of 2.034 (3), 2.156 (3) and 2.348 (2) Å, respectively, are similar to those reported for other Schiff base complexes (Dipesh *et al.*, 2003). The *trans* bond angles deviate slightly from the expected value of 180°. The Ni atom lies 3 Å out of the mean plane of the equatorial donors.

Experimental

The title compound was prepared by adding a methanol solution (5 ml) of nickel perchlorate (0.1 mmol) to an ethanol solution (10 ml) of *L* (Lu, 2003) (0.1 mmol) neutralized by triethylamine. The mixture was refluxed for about 2 h and then cooled to room temperature and filtered. The filtrate was slowly evaporated at room temperature to yield green block crystals of (I) suitable for X-ray analysis. Analysis calculated for $C_{20}H_{26}Cl_2N_6NiO_{12}S_2$: C 32.60, H 3.53, N 11.41%; found: C 32.43, H 3.46, N 11.32%.

Crystal data

$[Ni(C_{18}H_{18}N_6O_2S_2)](ClO_4)_2\cdot 2CH_4O$
$M_r = 736.20$
Tetragonal, $I4_1/a$
$a = 13.586$ (8) Å
$c = 32.60$ (4) Å
$V = 6018$ (9) Å^3
$Z = 8$
$D_x = 1.625$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 934 reflections
$\theta = 2.5$–25.5°
$\mu = 1.03$ mm^{-1}
$T = 293$ (2) K
Block, green
0.20 × 0.18 × 0.14 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.651$, $T_{max} = 0.866$
17255 measured reflections
3097 independent reflections
2286 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$
$\theta_{max} = 26.5^\circ$
$h = -10 \rightarrow 16$
$k = -16 \rightarrow 17$
$l = -40 \rightarrow 35$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.158$
$S = 1.08$
3097 reflections
197 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0804P)^2 + 15.2797P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.002$
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.81$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ni1—N1	2.034 (3)	Ni1—S1	2.3476 (18)
Ni1—O1	2.156 (3)		
N1^i—Ni1—N1	177.06 (18)	S1^i—Ni1—S1	101.98 (10)
N1^i—Ni1—O1	94.02 (12)	C9—S1—Ni1	95.83 (14)
N1—Ni1—O1	83.64 (12)	C2—O1—Ni1	123.1 (3)
N1—Ni1—S1^i	97.62 (10)	C1—O1—Ni1	114.4 (2)
N1—Ni1—S1	84.24 (10)	C8—N1—Ni1	127.7 (3)
O1—Ni1—S1	162.01 (8)	N2—N1—Ni1	116.5 (2)
O1^i—Ni1—S1	92.77 (11)		

Symmetry code: (i) $1-x, \frac{1}{2}-y, z$.

H atoms were included in calculated positions and refined as riding on their parent atoms, with C—H distances in the range 0.93–0.98 Å and N—H distances in the range 0.86–0.90 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

Data collection: *SMART-NT* (Bruker, 1998); cell refinement: *SMART-NT*; data reduction: *SAINT-NT* (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-NT* (Bruker, 1998); software used to prepare material for publication: *SHELXTL-NT*.

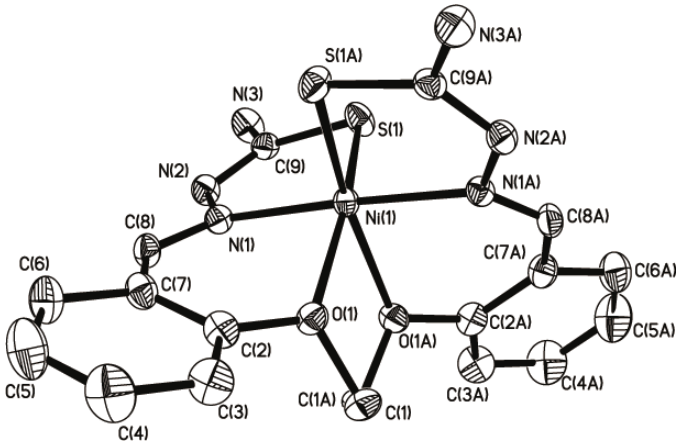


Figure 1
A view of the cation of the title compound, shown with 30% probability displacement ellipsoids. H atoms have been omitted. The suffix A corresponds to symmetry code (i) in Table 1.

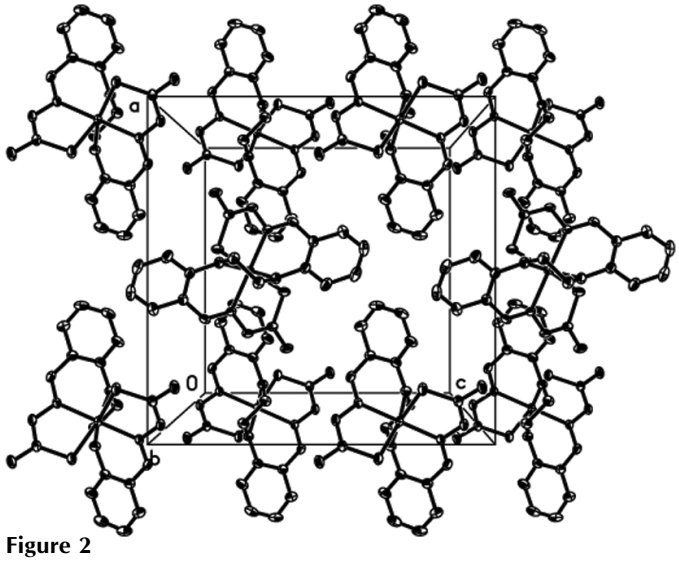


Figure 2
The packing of the title compound. H atoms, perchlorate ions and MeOH molecules have been omitted.

This work was supported by the Innovation Foundation of Nankai University.

References

Bruker (1998). *SMART-NT, SAINT-NT* and *SHELXTL-NT*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Dipesh, G., Suman, M., Satyabrata, S., Ki-Young, C., Akira, E. & Muktimoy, C. (2003). *Inorg. Chem.* **42**, 7189–7199.
Lu, S. R. (2003). *Chin. J. Synth. Chem.* **4**, 349–353.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Yang, Z. Y., Yang, R. D., Li, F. S. & Yu, K. B. (2000). *Polyhedron*, **19**, 2599–3604.