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trans-Diazidobis{2-[(3-cyclohexylaminopropylimino)methyl]phenolato}nickel(II)

Zhong-Lu You,[a,b] Zhong-Duo Xiong,[a] Wei-Sheng Liu,[b] Min-Yu Tan[b] and Hai-Liang Zhu[a,b]*

[a]Department of Chemistry, Wuhan University of Science and Engineering, Wuhan 430073, People's Republic of China, and [b]Department of Chemistry, Lanzhou University, Lanzhou 730000, People's Republic of China

Correspondence e-mail: hlzhu@wist.edu.cn

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Key indicators

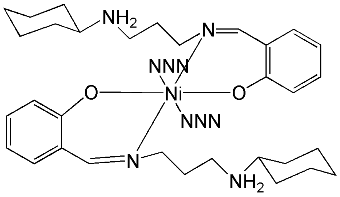
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.043
wR factor = 0.103
Data-to-parameter ratio = 13.8

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

The title compound, [Ni($C_{16}H_{24}N_{2}O$)$_2$(N_3)$_2$], crystallizes with two half-molecules per asymmetric unit; each mononuclear molecule is centrosymmetric. The Ni^{II} atoms are coordinated by six atoms from two Schiff bases and two azide anions. In the crystal structure, the molecules are held together by intermolecular N—H···N hydrogen bonds, forming one-dimensional chains extending in the **a** direction.

Comment

Transition metal compounds containing Schiff base ligands have been of great interest for many years (Yamada, 1999). These compounds play an important role in the coordination chemistry related to catalysis and enzymatic reactions, magnetism and molecular architectures. Since nickel compounds are present in the active sites of urease, and are used extensively in the design and construction of new magnetic materials, the study of nickel compounds is of great interest in various aspects of chemistry (Karplus & Pearson, 1997; Zhu *et al.*, 1999, 2001).



(I)

The title compound, (I), is a discrete neutral centrosymmetric compound (Fig. 1), and there are two independent half-molecules per asymmetric unit. The Ni^{II} atoms, lying on inversion centers, have an octahedral geometry and coord-

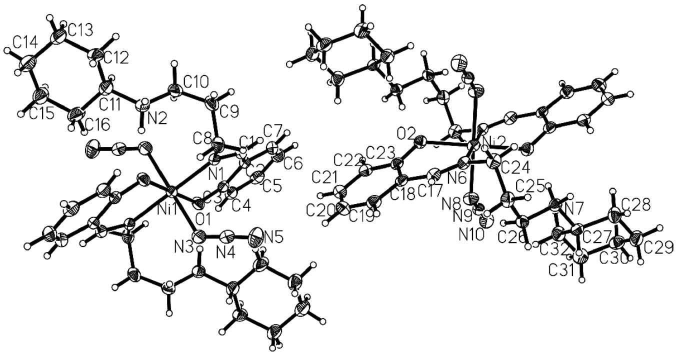


Figure 1
The structure of the title compound, (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme of the asymmetric unit.

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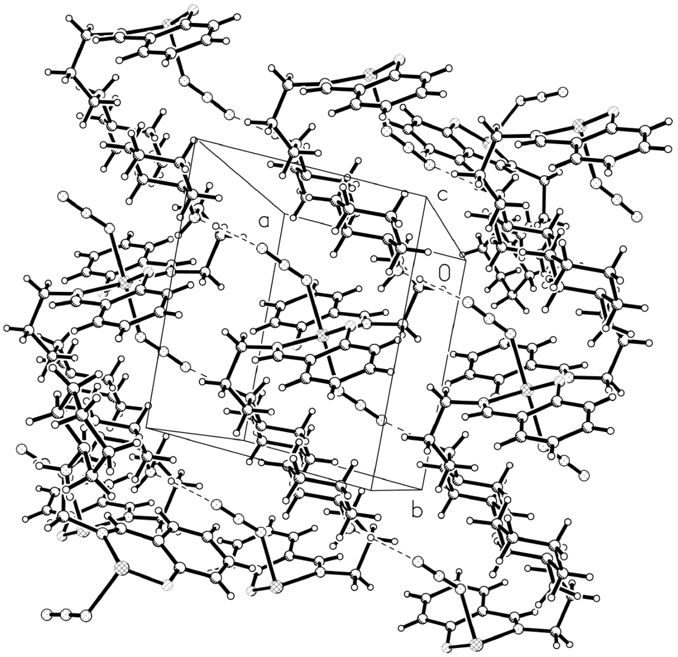

Figure 2
The crystal packing of (I), showing the N—H···N hydrogen-bonding interactions as dashed lines.

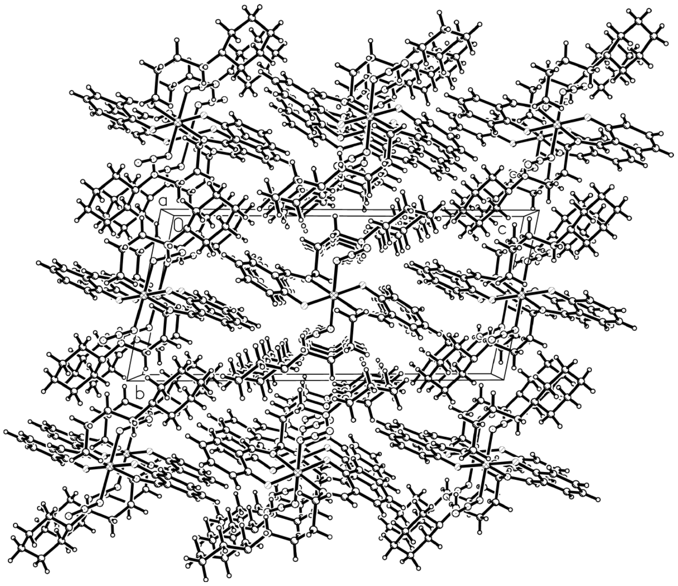

Figure 3
Crystal packing of (I), viewed along the *a* axis.

inated by two Schiff base ligands and two azide anions. The Schiff bases act as bidentate ligands and coordinate to the Ni^{II} atom through the phenolate O atoms and the imine N atoms. The azide anion is a monodentate ligand and coordinates to the metal *via* a terminal N atom. The three *trans* angles at Ni are, by symmetry, exactly 180°; the other angles are close to 90°, varying from 88.71 (9) to 91.88 (10)° for Ni1, and from 88.04 (9) to 91.96 (9)° for Ni2, which indicates a slightly distorted octahedral geometry of the Ni^{II} atoms. The Ni—O(phenolate) bond lengths [2.038 (2) Å for Ni1 and 2.049 (2) Å for Ni2] are comparable to the value of 2.030 (3) Å observed in a similar nickel(II) compound (Zhu *et al.*, 2003). The Ni—N(imine) bond distances [2.068 (3) Å for Ni1 and 2.068 (2) Å for Ni2] are equal to the value of 2.068 (3) Å observed in the compound $[Ni_2(tp)(pren)_4(Him)_2](ClO_4)_2$ (where tp is terephthalate, pren is 1,3-diaminopropane and Him is imidazole) (Zhu *et al.*, 2001). The azide N—N—N angle of 178.0 (4)° in the Ni1 complex and 178.1 (3)° in the Ni2 complex are comparable to the values of 178.4 (5) (Li *et al.*, 2003) and 178.8 (4)° (Choi *et al.*, 2002) observed previously. As expected, the cyclohexyl groups in the complex adopt chair conformations to minimize steric effects.

In the crystal structure, the molecules are connected by intermolecular N2—H2···N1 hydrogen bonds (see Fig. 2 and Table 1), forming one-dimensional chains extending in the *a*-axis direction (Fig. 3).

Experimental

N-Cyclohexyl-1,3-diaminopropane and salicylaldehyde were available commercially and were used without further purification. *N*-Cyclohexyl-1,3-diaminopropane (2.0 mmol, 312 mg) and salicylaldehyde (2.0 mmol, 244 mg) were dissolved in methanol (10 ml). The mixture was stirred for 1 h to obtain a clear orange solution of *L* (2.0 mmol), where *L* is 2-[(3-cyclohexylaminopropylimino)methyl]-phenol. To the solution of *L* was added a solution of $Ni(ClO_4)_2 \cdot 7H_2O$ (1.0 mmol, 383 mg) in methanol (10 ml), and NaN_3 (2.0 mmol, 13 mg) in distilled water (1 ml), with stirring. After allowing the resulting solution to stand in air for 7 d, green prismatic crystals were formed on slow evaporation of the solvent. The crystals were isolated, washed with methanol and dried in a vacuum desiccator using P_4O_{10} (yield 72.8%). Analysis found: C 58.08, H 7.08, N 21.12%; calculated for $C_{32}H_{48}N_{10}NiO_2$: C 57.93, H 7.29, N 21.11%.

Crystal data

$[Ni(C_{16}H_{24}N_2O)_2(N_3)_2]$
$M_r = 663.50$
Triclinic, $P\overline{1}$
$a = 7.911$ (4) Å
$b = 9.840$ (5) Å
$c = 21.292$ (10) Å
$\alpha = 101.291$ (7)°
$\beta = 93.077$ (7)°
$\gamma = 92.768$ (7)°
$V = 1620.2$ (13) Å^3
$Z = 2$
$D_x = 1.356$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2229 reflections
$\theta = 2.6$–22.3°
$\mu = 0.65$ mm^{-1}
$T = 298$ (2) K
Prism, green
0.25 × 0.18 × 0.13 mm

Data collection

Siemens SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.855$, $T_{max} = 0.921$
8609 measured reflections
5647 independent reflections
3698 reflections with $I > \sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 25.0°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 10$
$l = -25 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.103$
$S = 0.86$
5647 reflections
409 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.33$ e Å^{-3}

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

$D-\mathrm{H}\cdots A$	$D-\mathrm{H}$	$\mathrm{H}\cdots A$	$D\cdots A$	$D-\mathrm{H}\cdots A$
$\mathrm{N2-H2}A\cdots\mathrm{N5}^{\mathrm{i}}$	0.90	2.08	2.967 (4)	170
$\mathrm{N2-H2}B\cdots\mathrm{O1}^{\mathrm{ii}}$	0.90	1.80	2.698 (3)	175
$\mathrm{N7-H7}A\cdots\mathrm{N10}^{\mathrm{iii}}$	0.90	2.00	2.898 (4)	177
$\mathrm{N7-H7}B\cdots\mathrm{O2}^{\mathrm{iv}}$	0.90	1.78	2.673 (3)	169

Symmetry codes: (i) $1-x, 1-y, -z$; (ii) $2-x, 1-y, -z$; (iii) $x-1, y, z$; (iv) $1-x, 1-y, 1-z$.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms; N—H = 0.90 Å, C—H = 0.93–0.97 Å and $U_{\mathrm{iso}}(\mathrm{H}) = 1.2U_{\mathrm{eq}}$(C or N).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*b*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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References

Choi, K.-Y., Chun, K.-M., Lee, K.-C. & Kim. J. (2002). *Polyhedron*, **21**, 1913–1920.
Karplus, P. A. & Pearson, M. A. (1997). *Acc. Chem. Res.* **30**, 330–337.
Li, L. C., Liao, D. Z., Jiang, Z. H. & Yan, S. P. (2003). *J. Mol. Struct.* **649**, 37–42.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Yamada, S. (1999). *Coord. Chem. Rev.* **190–192**, 537–555.
Zhu, H.-L., Tong, Y.-X., Chen, X.-M. & Ren, C.-X. (2001). *Transition Met. Chem.* **26**, 528–531.
Zhu, H.-L., Zeng, Q.-F., Xia, D.-S., Liu, X.-Y. & Wang, D.-Q. (2003). *Acta Cryst.* E**59**, m777–m779.
Zhu, H.-L., Zheng, L.-M., Zhao, J., Bu, W.-M. & Tang, W.-X. (1999). *Transition Met. Chem.* **24**, 131–134.