

De-Zhong Niu,<sup>a</sup> Bai-Wang Sun,<sup>a</sup>  
Zai-Sheng Lu,<sup>a</sup> Zhe-Ming Wang<sup>b</sup>  
and Chun-Hua Yan<sup>b\*</sup>

<sup>a</sup>Department of Chemistry, Xuzhou Normal University, Jiangsu 221009, People's Republic of China, and <sup>b</sup>State Key Laboratory of Rare Earth Materials Chemistry and Applications, and PKU–HKU Joint Laboratory in Rare Earth Materials and Bioinorganic Chemistry, Peking University – Nonius BV Demo Lab For X-Ray Diffraction, Department of Chemistry, Peking University, Beijing 100871, People's Republic of China

Correspondence e-mail:  
chyan@chem.pku.edu.cn

#### Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$   
R factor = 0.038  
wR factor = 0.100  
Data-to-parameter ratio = 17.1

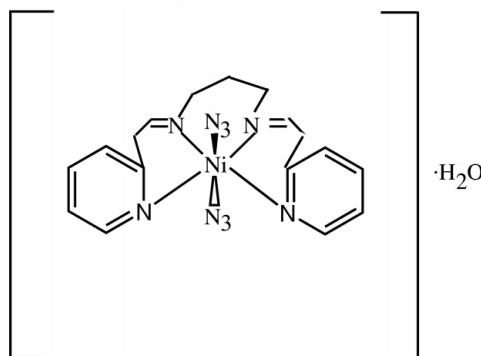
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

## Diazido{*N,N'*-bis[2-(2-pyridyl)ethylene]-1,3-diaminopropane}nickel(II) monohydrate

The crystal structure of yellow  $[\text{Ni}(L)(\text{N}_3)_2] \cdot \text{H}_2\text{O}$ , where *L* is *N,N'*-bis[2-(2-pyridyl)ethylene]-1,3-diaminopropane ( $\text{C}_{15}\text{H}_{16}\text{N}_4$ ), containing six-coordinate nickel(II) with an octahedral  $[\text{Ni}^{\text{II}}\text{N}_6]$  core, is reported. Four N atoms of the Schiff base ligand form the equatorial plane and two N atoms of two azide ligands occupy the axial positions.

#### Comment

In an attempt to prepare Haldane gap compounds with  $S = 1$ , we obtained the mononuclear nickel(II) complex  $[\text{Ni}(L)(\text{N}_3)_2(\text{H}_2\text{O})]$ , (I), where *L* = *N,N'*-bis[2-(2-pyridyl)ethylene]-1,3-diaminopropane.



(I)

The  $\text{Ni}^{\text{II}}$  octahedron deviates slightly from  $O_h$  symmetry, with the four N atoms of the Schiff base ligand in the equatorial plane, and two N atoms of different azide ligands at the axial positions. The Ni–N distances are in the range 2.050 (2)–2.138 (2) Å, the *cis*-N–Ni–N angles in the range 78.93 (8)–109.65 (7)° and the *trans*-N–Ni–N angles in the range 171.21 (7)–174.70 (8)°. These values are in good agreement with those reported in the literature (Asokan *et al.*, 1998). Fig. 1 shows an ellipsoid plot (Sheldrick, 1998) of the first coordination sphere of the  $\text{Ni}^{\text{II}}$  site and the atom labeling.

The dihedral angles are 2.49 (2)° between plane I (atoms N1, N2, N3, N4 and Ni1) and plane II (atoms N1, C1, C2, C3, C4 and C5) and 3.99 (2)° between planes I and III (atoms N4, C11, C12, C13, C14 and C15).

#### Experimental

0.214 g (2.0 mmol) of 2-pyridylaldehyde and 0.074 g (1.0 mmol) of 1,3-diaminopropane were stirred in 20 ml of ethanol, 0.237 mg (1.0 mmol)  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  was added, and the mixture was stirred to obtain a clear solution. To this, a solution of 130 mg (2 mmol) of  $\text{NaN}_3$  in a minimum amount of water was added, and the solution was

Received 19 October 2001  
Accepted 15 November 2001  
Online 24 November 2001

filtered after 1 h. Yellow polyhedral crystals of  $[\text{Ni}(\text{L})(\text{N}_3)_2]$  were separated from the mother liquor by slow evaporation at room temperature after two weeks. The crystals were filtered off, washed with a small amount of water, and dried in air. The yield was 55%. Analysis calculated for  $\text{C}_{15}\text{H}_{18}\text{N}_{10}\text{NiO}$ : C 43.62, H 4.39, N 33.91%; found: C 43.55, H 4.56, N 34.21%.

**Crystal data**

$[\text{Ni}(\text{C}_{15}\text{H}_{16}\text{N}_4)(\text{N}_3)_2] \cdot \text{H}_2\text{O}$   
 $M_r = 413.10$   
 Orthorhombic,  $Pbca$   
 $a = 13.5783$  (3) Å  
 $b = 15.2608$  (3) Å  
 $c = 17.5449$  (4) Å  
 $V = 3635.58$  (14) Å<sup>3</sup>  
 $Z = 8$   
 $D_x = 1.509$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 48907 reflections  
 $\theta = 3.5\text{--}27.9^\circ$   
 $\mu = 1.10$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Block, yellow  
 0.24 × 0.20 × 0.18 mm

**Data collection**

Nonius KappaCCD diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multiscan (Blessing, 1995, 1997)  
 $T_{\text{min}} = 0.710$ ,  $T_{\text{max}} = 0.821$   
 48907 measured reflections  
 4317 independent reflections

2828 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.069$   
 $\theta_{\text{max}} = 27.9^\circ$   
 $h = -17 \rightarrow 17$   
 $k = -20 \rightarrow 20$   
 $l = -23 \rightarrow 23$

**Refinement**

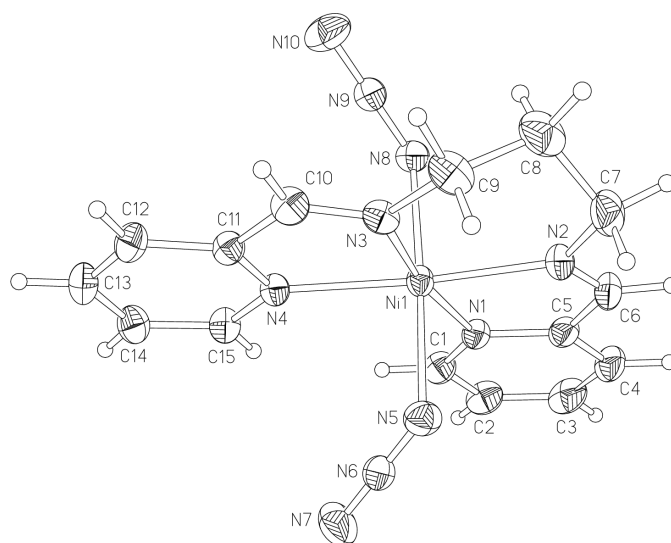
Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.038$   
 $wR(F^2) = 0.100$   
 $S = 1.02$   
 4317 reflections  
 252 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 1.9396P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.68$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.37$  e Å<sup>-3</sup>  
 Extinction correction: *SHELXL97*  
 Extinction coefficient: 0.0014 (3)

**Table 1**

Selected geometric parameters (Å, °).

Ni1—N3	2.0497 (18)	Ni1—N8	2.130 (2)
Ni1—N2	2.0569 (18)	Ni1—N1	2.1339 (18)
Ni1—N5	2.113 (2)	Ni1—N4	2.1380 (18)
N3—Ni1—N2	92.34 (8)	N5—Ni1—N1	88.13 (8)
N3—Ni1—N5	93.25 (8)	N8—Ni1—N1	88.35 (7)
N2—Ni1—N5	92.06 (8)	N3—Ni1—N4	79.07 (7)
N3—Ni1—N8	90.83 (7)	N2—Ni1—N4	171.39 (7)
N2—Ni1—N8	91.15 (8)	N5—Ni1—N4	88.96 (7)
N5—Ni1—N8	174.70 (8)	N8—Ni1—N4	88.50 (7)
N3—Ni1—N1	171.21 (7)	N1—Ni1—N4	109.65 (7)
N2—Ni1—N1	78.93 (8)		



**Figure 1**  
 The molecular structure with 30% probability displacement ellipsoids and the atom labeling.

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D\text{--}H \cdots A$	$D\text{--}H$	$H \cdots A$	$D \cdots A$	$D\text{--}H \cdots A$
O1—H1A <sup>i</sup> ⋯N5	0.94 (2)	1.96 (2)	2.892 (3)	171 (3)
O1—H1B <sup>i</sup> ⋯N8 <sup>i</sup>	0.94 (2)	1.98 (3)	2.901 (3)	169 (2)

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *maxus* (Mackay *et al.*, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXL97*.

**References**

Asokan, A., Varghese, B., Caneschi, A. & Manoharan, P. T. (1998). *Inorg. Chem.* **37**, 228–232.  
 Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.  
 Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.  
 Mackay, S., Gilmore, C. J., Edwards, C., Tremayne, M., Stuart, N. & Shankland, K. (1998). *maxus*. University of Glasgow, Scotland, Nonius BV, Delft, The Netherlands, and MacScience Co. Ltd, Yokohama, Japan.  
 Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.  
 Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter and R. M. Sweet, pp. 307–326. London: Academic Press.  
 Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. PC Version. University of Göttingen, Germany.  
 Sheldrick, G. M. (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.