

Yu-Xi Sun,\* Gen-Zhi Gao, Rui  
Zhang and Hong-Xia PeiDepartment of Chemistry, Qufu Normal  
University, Qufu 273165, People's Republic of  
China

Correspondence e-mail: yuxisun@163.com

## Key indicators

Single-crystal X-ray study  
 $T = 298\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.043  
 $wR$  factor = 0.121  
Data-to-parameter ratio = 17.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Azido[1-(isobutylaminomethyliminomethyl)-  
2-naphtholato]nickel(II)The title compound,  $[\text{Ni}(\text{C}_{15}\text{H}_{17}\text{N}_2\text{O})(\text{N}_3)]$ , is a mononuclear  
nickel(II) complex. The  $\text{Ni}^{\text{II}}$  atom is four-coordinated by two  
N atoms and one O atom of the Schiff base ligand, and another  
N atom from an azide anion, forming a slightly distorted  
square-planar coordination configuration.

Received 10 January 2005

Accepted 18 January 2005

Online 29 January 2005

## Comment

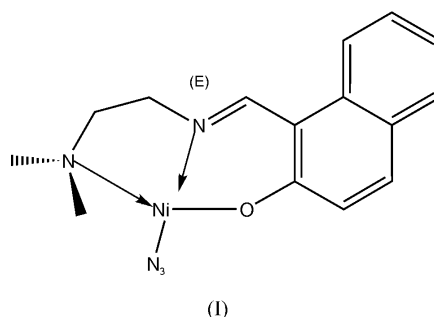
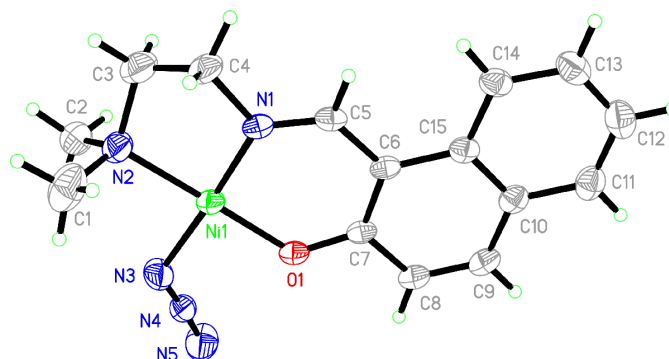
Transition metal compounds are present in the active sites of  
several important classes of metalloproteins. The study of  
Schiff base compounds is of great interest in various aspects of  
chemistry (Downing & Urbach, 1969; Ganeshpure *et al.*, 1996;  
Bosnich, 1968; Costes *et al.*, 1995).The title complex, (I), is a mononuclear nickel(II)  
compound (Fig. 1). The  $\text{Ni}^{\text{II}}$  atom is four-coordinated by two  
N atoms and one O atom of the Schiff base ligand, and another  
N atom from an azide anion, forming a slightly distorted square-  
planar coordination configuration. The four coordinating  
atoms around the Ni centre are approximately coplanar,  
giving a square-planar geometry with an average deviation of  
0.014 (6) Å; the Ni atom lies 0.020 (3) Å above this plane. The

Figure 1

The molecular structure of (I), showing the atom-numbering  
scheme. Displacement ellipsoids are drawn at the 30% probability  
level.

Ni1—O1 bond length [1.817 (2) Å; Table 1] is a little greater than the corresponding value [1.837 (3) Å] observed in another nickel(II) complex (Zhu *et al.*, 2004). The Ni1—N1 bond length in (I) [1.838 (2) Å] is a little less than the corresponding value [1.845 (3) Å] observed in the same complex.

As illustrated in Fig. 2, in the crystal structure of (I), the molecules stack along the *a* axis and are linked by C—H···O hydrogen bonds (Table 2).

### Experimental

2-Hydroxy-1-naphthaldehyde (0.2 mmol, 17.2 mg) and *N,N*-dimethylethane-1,2-diamine (0.2 mmol, 17.6 mg) were dissolved in ethanol (10 ml). The mixture was stirred for 15 min to give a clear yellow solution. To this solution was added an aqueous solution (5 ml) of NaN<sub>3</sub> (0.1 mmol, 5.5 mg) and an ethanol solution (5 ml) of Ni(NO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O (0.1 mmol, 24.4 mg), with stirring. The mixture was stirred at room temperature for about 30 min and then filtered. After allowing the green filtrate to stand in air for 11 d, green block-shaped crystals of (I) were formed at the bottom of the vessel on slow evaporation of the solvent.

#### Crystal data

[Ni(C <sub>15</sub> H <sub>17</sub> N <sub>2</sub> O)(N <sub>3</sub> )]	Mo <i>K</i> α radiation
<i>M<sub>r</sub></i> = 342.05	Cell parameters from 7800 reflections
Orthorhombic, <i>Pbca</i>	$\theta = 2.7\text{--}27.5^\circ$
<i>a</i> = 7.576 (1) Å	$\mu = 1.28\text{ mm}^{-1}$
<i>b</i> = 13.300 (2) Å	<i>T</i> = 298 (2) K
<i>c</i> = 30.306 (2) Å	Block, green
<i>V</i> = 3053.7 (6) Å <sup>3</sup>	0.29 × 0.12 × 0.10 mm
<i>Z</i> = 8	
<i>D<sub>x</sub></i> = 1.488 Mg m <sup>-3</sup>	

#### Data collection

Bruker APEX area-detector diffractometer	3488 independent reflections
$\varphi$ and $\omega$ scans	2859 reflections with <i>I</i> > 2σ( <i>I</i> )
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	<i>R<sub>int</sub></i> = 0.026
<i>T<sub>min</sub></i> = 0.708, <i>T<sub>max</sub></i> = 0.883	$\theta_{\text{max}}$ = 27.5°
24 371 measured reflections	<i>h</i> = -9 → 9
	<i>k</i> = -16 → 16
	<i>l</i> = -39 → 39

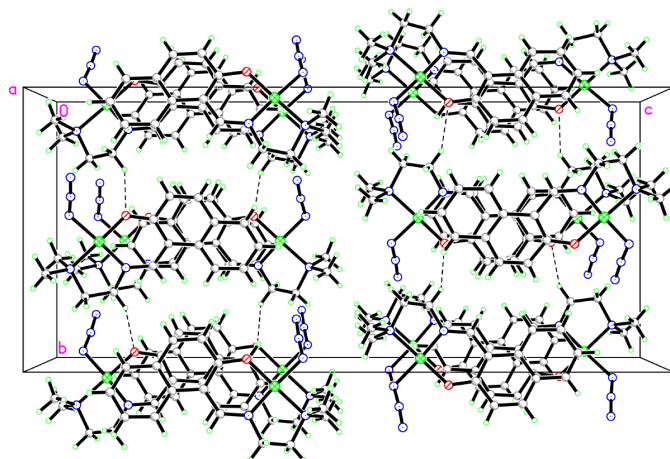
#### Refinement

Refinement on <i>F</i> <sup>2</sup>	$w = 1/[\sigma^2(F_o^2) + (0.0564P)^2 + 2.2975P]$
$R[F^2 > 2\sigma(F^2)] = 0.043$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.121$	( $\Delta/\sigma$ ) <sub>max</sub> < 0.001
<i>S</i> = 1.08	$\Delta\rho_{\text{max}} = 0.97\text{ e \AA}^{-3}$
3488 reflections	$\Delta\rho_{\text{min}} = -0.31\text{ e \AA}^{-3}$
201 parameters	
H-atom parameters constrained	

**Table 1**

Selected geometric parameters (Å, °).

Ni1—O1	1.817 (2)	Ni1—N3	1.900 (3)
Ni1—N1	1.838 (2)	Ni1—N2	1.952 (2)
O1—Ni1—N1	94.0 (1)	O1—Ni1—N2	179.1 (1)
O1—Ni1—N3	89.5 (1)	N1—Ni1—N2	86.8 (1)
N1—Ni1—N3	176.0 (1)	N3—Ni1—N2	89.7 (1)



**Figure 2**

The crystal packing of (I), viewed along the *a* axis. The C—H···O hydrogen bonds are shown as dashed lines (Table 2).

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C4—H4B···O1 <sup>i</sup>	0.97	2.56	3.327 (3)	136

Symmetry code: (i)  $-x + \frac{3}{2}, +y - \frac{1}{2}, z$ .

The H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.97 Å and with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C).

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2002); software used to prepare material for publication: SHELXTL.

The authors thank Qufu Normal University for funding this study.

### References

- Bruker. (2002). SMART (Version 5.628), SAINT (Version 6.02) and SHELXTL (Version 5.1), Bruker AXS Inc., Madison, Wisconsin, USA.
- Bosnich, B. (1968). *J. Am. Chem. Soc.* **90**, 627–632.
- Costes, J. P., Dominguez-Vera, J. M. & Laurent, J. P. (1995). *Polyhedron*, **14**, 2179–2187.
- Downing, R. S. & Urbach, F. L. (1969). *J. Am. Chem. Soc.* **91**, 5977–5983.
- Ganeshpure, P. A., Tembe, G. L. & Satish, S. (1996). *J. Mol. Catal. A*, **113**, L423–L425.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
- Zhu, B., Ruang, W. & Zhu, Z. (2004). *Acta Cryst.* **E60**, m634–m636.