

Azido{2,4-dichloro-6-[2-(diethylamino)-ethyliminomethyl]phenolato}nickel(II)

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Key indicators

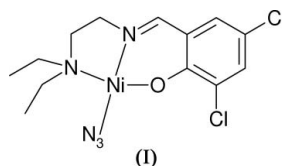
Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.035
 wR factor = 0.086
Data-to-parameter ratio = 18.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title mononuclear nickel(II) complex, $[\text{Ni}(\text{C}_{13}\text{H}_{17}\text{Cl}_2\text{N}_2\text{O})(\text{N}_3)]$, is isostructural with the copper(II) complex reported recently by Wei & Wang [*Acta Cryst.* (2006), **E62**, m309–m311]. The Ni^{II} ion is four-coordinate in a square-planar geometry defined by an imine N, an amine N, a phenolate O atom of the Schiff base and a terminal N atom of the azide anion.

Received 18 January 2006
Accepted 20 February 2006

Comment

Schiff base ligands have played an important role in the development of coordination chemistry as they readily form stable complexes with most transition metal ions (Ueno *et al.*, 2004; Pal *et al.*, 2005; Hou, 2005). Recently, we reported a mononuclear copper(II) complex, azido{2,4-dichloro-6-[2-(diethylamino)ethyliminomethyl]phenolato}copper(II), (II), derived from the Schiff base ligand 2,4-dichloro-6-[2-(diethylamino)ethyliminomethyl]phenol (Wei & Wang, 2006). As an extension of our work on the complexes derived from this ligand, the title nickel(II) complex, (I), has been investigated.



The Ni atom in mononuclear (I) is four-coordinated by an imine N, an amine N, a phenolate O atom derived from the Schiff base and a terminal N atom of the azide anion, giving a square planar geometry. The Ni–ligand bond lengths (Table 1) are shorter than the corresponding values observed in isostructural (II), and within the ranges expected for Ni–Schiff base structures (de Castro *et al.*, 2001; Chakraborty *et al.*, 2004; Wei, 2005). The bond angles around the central metal in (I) are comparable to those in (II), and also show some deviations from ideal square-planar geometry (Table 1). In the crystal structure, adjacent molecules are linked through weak $\text{Cl} \cdots \text{Cl}$ interactions, forming loosely associated dimers (Fig. 2), such that $\text{Cl}2 \cdots \text{Cl}2^i$ is 3.4908 (12) [symmetry code: (i) $1 - x, 1 - y, 2 - z$]. There are also weak $\text{C}-\text{H} \cdots \text{N}^{\text{iii}}$ interactions linking molecules into chains along the b axis (Table 2).

Experimental

3,5-Dichlorosalicylaldehyde (1.0 mmol, 192.1 mg), *N,N*-diethylethane-1,2-diamine (1.0 mmol, 116.2 mg), NaN_3 (1.0 mmol, 65.3 mg) and $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (1.0 mmol, 290.7 mg) were dissolved in an MeOH solution (150 ml). The mixture was refluxed at 340 K for

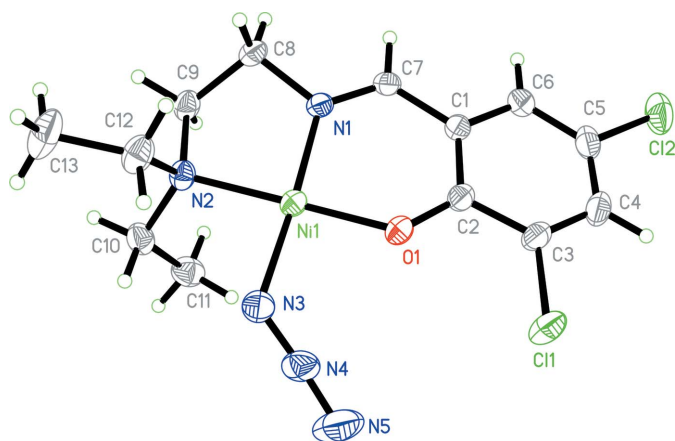


Figure 1
The structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

about 1 h to give a clear green solution. After keeping the cooled solution in the dark for 5 days, green block-shaped crystals were formed.

Crystal data

[Ni(C ₁₃ H ₁₇ Cl ₂ N ₂ O)(N ₃)]	<i>D_x</i> = 1.603 Mg m ⁻³
<i>M_r</i> = 388.93	Mo K α radiation
Monoclinic, <i>P</i> 2 ₁ / <i>c</i>	Cell parameters from 3623 reflections
<i>a</i> = 16.361 (2) Å	θ = 2.4–26.7°
<i>b</i> = 9.735 (1) Å	μ = 1.54 mm ⁻¹
<i>c</i> = 10.165 (1) Å	<i>T</i> = 298 (2) K
β = 95.656 (1)°	Block, green
<i>V</i> = 1611.1 (3) Å ³	0.23 × 0.20 × 0.12 mm
<i>Z</i> = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3618 independent reflections
ω scans	2772 reflections with <i>I</i> > 2 σ (<i>I</i>)
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	<i>R</i> _{int} = 0.028
<i>T</i> _{min} = 0.718, <i>T</i> _{max} = 0.836	θ _{max} = 27.5°
10944 measured reflections	<i>h</i> = -20 → 21
	<i>k</i> = -12 → 12
	<i>l</i> = -13 → 13

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 0.2299P]$
$R[F^2 > 2\sigma(F^2)] = 0.035$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.086$	(Δ/σ) _{max} < 0.001
<i>S</i> = 1.02	$\Delta\rho$ _{max} = 0.40 e Å ⁻³
3618 reflections	$\Delta\rho$ _{min} = -0.21 e Å ⁻³
201 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ni1—O1	1.840 (2)	Ni1—N2	1.967 (2)
Ni1—N1	1.850 (2)	Ni1—N3	1.894 (2)
O1—Ni1—N1	93.87 (7)	N1—Ni1—N2	86.83 (8)
O1—Ni1—N2	178.84 (7)	N1—Ni1—N3	176.76 (8)
O1—Ni1—N3	89.33 (8)	N2—Ni1—N3	89.97 (9)

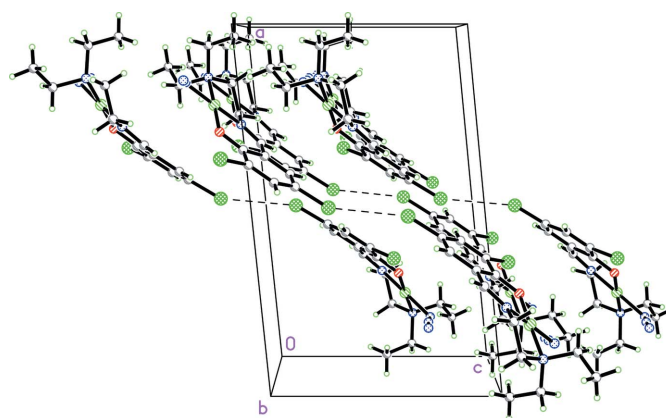


Figure 2
The crystal packing of (I), highlighting the Cl...Cl interactions (dashed lines).

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>
C10—H10B...N3	0.97	2.56	2.915 (2)	102
C12—H12B...N3	0.97	2.59	3.125 (2)	115
C13—H13A...N5	0.97	2.66	3.576 (4)	159

Symmetry code: (i) *x*, 1 + *y*, *z*.

All H atoms were placed in 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*_{iso}(H) = 1.2 or 1.5*U*_{eq}(C).

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

The authors acknowledge the Natural Science Foundation of Anhui Provincial Universities, People’s Republic of China, for research grant No. 2005 K J019ZD.

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