Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Tetrakis(3,5-lutidine)dichloronickel(II)

Xiang-Sheng Xu,[a] Ya-Ping Lü,[b] Dong-Shun Deng,[b] Mao-Lin Hu[c] and Zhi-Min Jin[b]*

[a]College of Chemical Engineering and Materials Science, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China, [b]College of Pharmaceutical Sciences, Zhejiang University of Technology, Hangzhou 310014, People's Republic of China, and [c]School of Chemistry and Materials Science, Wenzhou Normal College, Zhejiang, Wenzhou 325027, People's Republic of China

Correspondence e-mail: zimichem@sina.com

Received 29 March 2005
Accepted 11 April 2005
Online 16 April 2005

Key indicators

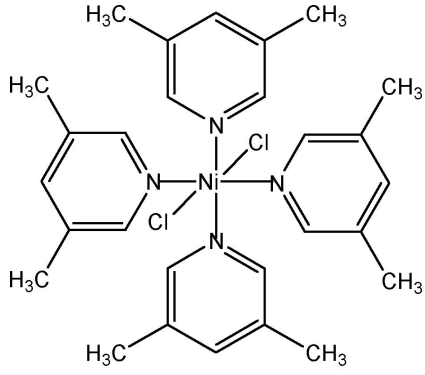
Single-crystal X-ray study
T = 289 K
Mean σ(C–C) = 0.003 Å
R factor = 0.028
wR factor = 0.075
Data-to-parameter ratio = 16.8

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

In the title compound, $[NiCl_2(C_7H_9N)_4]$, where C_7H_9N is 3,5-lutidine, the Ni^{II} atom is coordinated by two Cl atoms and four N atoms from 3,5-lutidine groups. The geometry around the Ni^{II} atom, which is located at a special position of symmetry 422, is octahedral. Molecules of the title compound are connected by C—H···Cl intermolecular hydrogen bonds and C—H···π interactions to form a three-dimensional structure.

Comment

Various 3,5-lutidine compounds have been reported previously (Abu-Youssef *et al.*,1999; Bolte *et al.*, 2000; Carmalt *et al.*, 2000; Goher *et al.*, 1997; Hu & Englert, 2002; Maunder & Sella, 1998; Minghetti *et al.*, 1998; Modec *et al.*, 2000; Nogai *et al.*, 2003; Nyman *et al.*, 1997; Tessier & Rochon, 2001; van Poppel *et al.*, 2001; Vries *et al.*, 2001). We have synthesized the title compound, (I), and report its structure here.



(I)

In (I), the Ni^{II} atom is coordinated octahedrally by four 3,5-lutidine ligands through four N atoms and two Cl atoms (Fig. 1). The Ni^{II} atom is located at a special position of symmetry 422. The dihedral angle between the plane of each pyridine ring and the N_4 plane is 46.1 (2)°. The geometrical outline of (I) resembles a screw propeller; a similar feature is also observed in an ytterbium compound, $(3,5\text{-lutidine})_4YbI_2$ (Maunder & Sella 1998). The two axial positions are filled by two Cl atoms.

There are five kinds of C—H···Cl hydrogen bonds (Table 2), with H···Cl distances shorter than the sum of the van der Waals radii [r(H) 1.16 (Zefirov & Zorkii, 1974) and 1.2 Å (Bondi, 1964); r(Cl) 1.90 Å (Zefirov & Zorkii, 1974)]. A C—H···π interaction [H4B···C_p($-x$, 1 $-$ y, $-z$) = 3.299 (3) Å, where C_p is the centroid of the pyridine ring] plays a minor role in the crystal structure (Fig. 2).

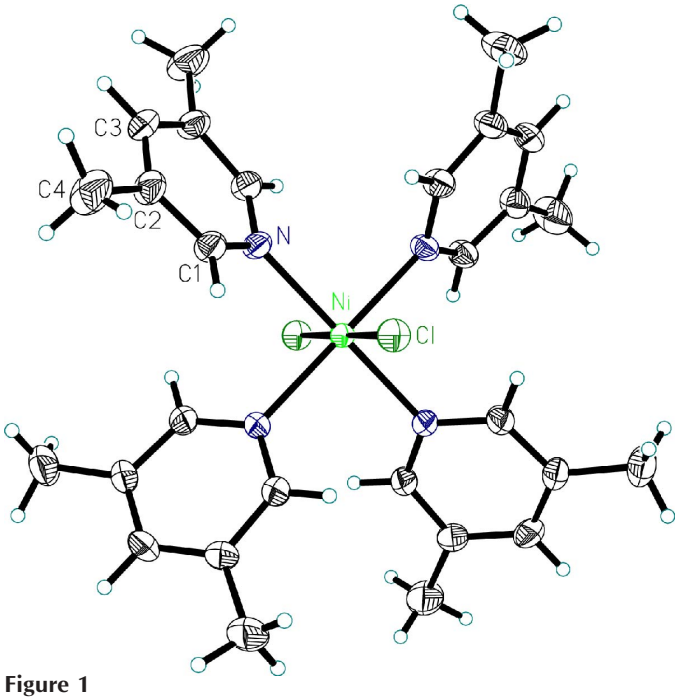


Figure 1
The formula unit with atom labels, showing 40% probability displacement ellipsoids.

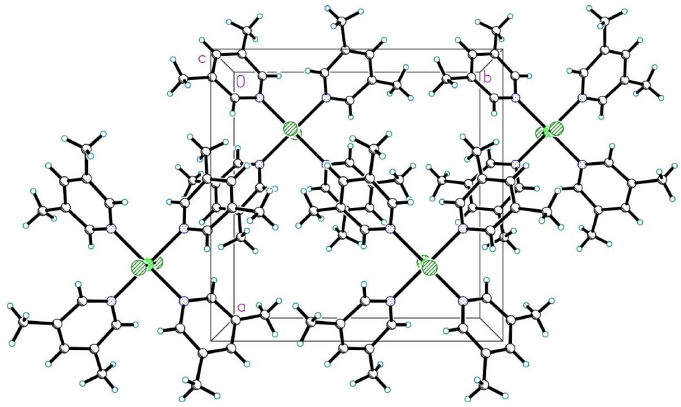


Figure 2
A packing diagram, viewed along the *c* axis.

Table 1
Selected bond lengths (Å).

Ni—N	2.136 (2)	C1—C2	1.383 (3)
Ni—Cl	2.4523 (11)	C2—C3	1.387 (3)
N—C1	1.336 (2)	C2—C4	1.499 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···Cl	0.93	2.90	3.361 (3)	112

Experimental

The title compound was prepared by a hydrothermal procedure from a mixture of 3,5-lutidine (5 mmol, 0.53 g), $NiCl_2{\cdot}6H_2O$ (1 mmol, 0.24 g) and water (30 ml) in a 30 ml Teflon-lined stainless steel reactor. The mixture was heated to 415 K for 3 d. The reaction system was then slowly cooled to room temperature. Green block-shaped crystals of (I), with a yield of 76% based on 3,5-lutidine, were collected and washed with distilled water. IR (KBr): 3583, 3391, 3267, 2922, 2846, 1708, 1445, 1416, 1331, 1135, 1049, 1028 cm^{-1}.

Crystal data

$[NiCl_2(C_7H_9N)_4]$
M_r = 558.20
Tetragonal, $P4/nnc$
a = 11.583 (1) Å
c = 10.747 (1) Å
V = 1441.8 (2) Å^3
Z = 2
D_x = 1.286 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 reflections
θ = 2.6–13.0°
μ = 0.88 mm^{-1}
T = 289 (2) K
Block, green
0.38 × 0.38 × 0.28 mm

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.723, T_{max} = 0.781
1894 measured reflections
757 independent reflections
502 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.5°
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = -1 \rightarrow 13$
3 standard reflections
every 97 reflections
intensity decay: 4.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.075
S = 0.91
757 reflections
45 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0408P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0049 (11)

All H atoms were placed in calculated positions (C—H = 0.93 Å for pyridyl CH groups and C—H = 0.96 Å for CH_3 groups) and allowed to ride on the parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Siemens, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Abu-Youssef, M. A. M., Escuer, A., Gatteschi, D., Goher, M. A. S., Mautner, F. A. & Vicente, R. (1999). *Inorg. Chem.* **38**, 5716–5723.
Bolte, M., Hensen, K. & Spangenberg, B. (2000). *J. Chem. Crystallogr.* **30**, 245–249.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Carmalt, C. J., Mileham, J. D., White, A. J. P. & Williams, D. J. (2000). *New J. Chem.* **24**, 929–930.
Goher, M. A. S., A1-Salem, N. A. & Mautner, F. A. (1997). *Polyhedron*, **16**, 3747–3755.
Hu, C. H. & Englert, U. (2002). *CrystEngComm*, **4**, 20–25.
Maunder, G. H. & Sella, A. (1998). *Polyhedron*, **17**, 63–68.
Minghetti, G., Cinellu, M. A., Pinna, M. V., Stoccoro, S., Zucca, A. & Manassero, M. (1998). *J. Organomet. Chem.* **568**, 225–232.
Modec, B., Brenëië, J. V., Golië, L. & Daniels, L. M. (2000). *Polyhedron*, **19**, 1407–1414.
Nogai, S., Schriewer, A. & Schmidbaur, H. (2003). *Dalton Trans.* pp. 3165–317.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Nyman, M. D., Hampden-Smith, M. J. & Duesler, E. N. (1997). *Inorg. Chem.* **36**, 2218–2224.

Poppel, L. G. van, Bott, S. G. & Barron, A. R. (2001). *J. Chem. Crystallogr.* **31**, 417–420.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1994). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1998). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Tessier, C. & Rochon, F. D. (2001). *Inorg. Chim. Acta*, **322**, 37–46.
Vries, E., Nassimbeni, L. R. & Su, H. (2001). *Eur. J. Org. Chem.* pp. 1887–1892.
Zefirov, Yu. V. & Zorkii, P. M. (1974). *Zh. Strukt. Khim.* **15**, 118–122.