Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

{2,6-Bis[(dimethylamino)methyl]phenyl-κ^2N,C^1,N'}-chloronickel(II)

Annie Castonguay,* Fabien Charbonneau, André L. Beauchamp and Davit Zargarian

Département de Chimie, Université de Montréal, CP 6128, Succ. Centre-ville, Montréal, Québec, Canada H3C 3J7

Correspondence e-mail: a.castonguay@umontreal.ca

Key indicators

Single-crystal X-ray study
T = 473 K
Mean σ(C–C) = 0.002 Å
R factor = 0.034
wR factor = 0.093
Data-to-parameter ratio = 15.8

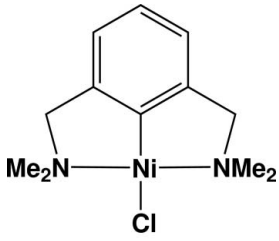
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title complex, [Ni($C_{12}H_{19}N_2$)Cl], consists of a slightly distorted square-planar Ni^{II} center coordinated by an anionic terdentate NCN pincer-type ligand, where both N atoms are *trans* to each other, and by a chloride ion. It is isostructural with two platinum analogues.

Received 27 September 2005
Accepted 4 October 2005
Online 12 October 2005

Comment

The title complex, (I), is known to be one of the most effective catalysts for the Kharash addition reaction (Grove *et al.*, 1989), and the rigidity of its framework allows further oxidation of the metal center to an air-stable five-coordinate Ni^{III} species (Grove *et al.*, 1983). Single crystals of (I) were isolated in the course of our study of the preparation and the reactivity of different pincer-type complexes. A view of the complex is presented in Fig. 1.



(I)

In the solid-state structure reported here, the Ni atom adopts a slightly distorted square-planar coordination. The

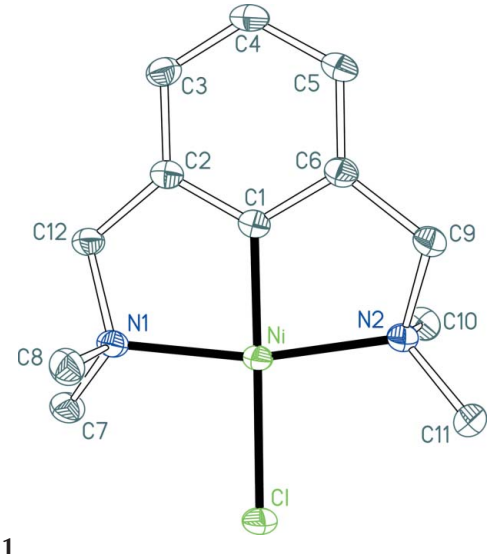


Figure 1
Top view of the title complex, (I), showing 30% displacement ellipsoids. H atoms have been omitted for clarity.

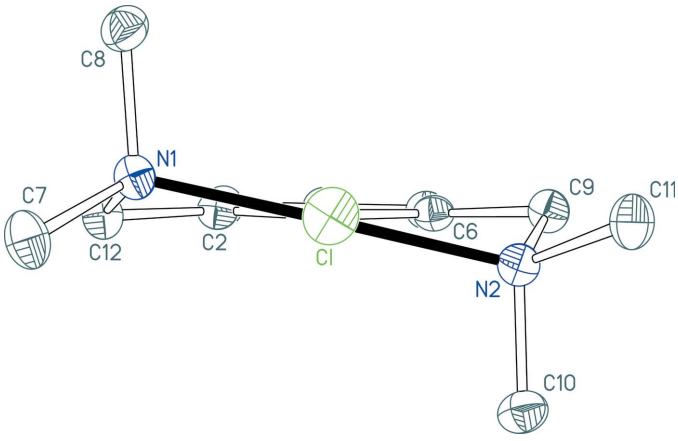


Figure 2
Side view of the title complex, (I), showing the approximate C_2 axis of the molecule, and showing 30% displacement ellipsoids. H atoms have been omitted for clarity.

molecule has an approximate C_2 axis but does not have a plane of symmetry since the two amine groups are located on opposite sides of the plane defined by the aromatic ring (Fig. 2).

Compound (I) was found to be isostructural with two complexes of platinum, [Pt*X*{$C_6H_3(CH_2NMe_2)_2$-N,C,N′}] (where *X* = Cl and Br) (Terheijden *et al.*, 1986; Albrecht *et al.*, 2000). All three structures crystallize in the space group and their respective cells, which display similar parameters, contain four molecules.

Experimental

A solution of *n*-BuLi (0.21 ml of a 2.5 *M* solution of *n*-BuLi in hexanes, 0.53 mmol) was added to a cooled solution of 1,3-bis-[(dimethylamino)methyl]benzene (100 mg, 0.52 mmol) in hexanes (2 ml) and the mixture was stirred at room temperature for 24 h. Removal of the solvent under reduced pressure produced an oily solid, to which was added a tetrahydrofuran solution (10 ml) of $Ni(PMe_3)_2Cl_2$ (147 mg, 0.52 mmol), and the resulting mixture was stirred for 72 h. Addition of hexanes (15 ml) and filtration produced a yellow filtrate which was evaporated to give a solid residue. Recrystallization of this material from a cooled solution in benzene/hexanes gave a few orange crystals.

Crystal data

$[Ni(C_{12}H_{19}N_2)Cl]$	D_x = 1.478 Mg m^{-3}
M_r = 285.45	Cu $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 12652 reflections
a = 13.0386 (7) Å	θ = 3.7–68.9°
b = 9.0976 (5) Å	μ = 3.88 mm^{-1}
c = 11.6950 (7) Å	T = 200 (2) K
β = 112.329 (2)°	Block, orange
V = 1283.24 (12) Å^3	0.16 × 0.14 × 0.12 mm
Z = 4	

Data collection

Bruker SMART 2000 diffractometer	2347 independent reflections
ω scans	2222 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	R_{int} = 0.028
T_{min} = 0.546, T_{max} = 0.628	θ_{max} = 68.9°
17426 measured reflections	$h = -15 \rightarrow 15$
	$k = -10 \rightarrow 10$
	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.3203P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.034	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.093	$\Delta\rho_{max}$ = 0.28 e Å^{-3}
S = 1.09	$\Delta\rho_{min}$ = −0.80 e Å^{-3}
2347 reflections	
149 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ni—C1	1.8298 (17)	Ni—N2	1.9914 (13)
Ni—N1	1.9904 (13)	Ni—Cl	2.2388 (5)
C1—Ni—N1	83.28 (6)	C1—Ni—Cl	177.41 (5)
C1—Ni—N2	83.17 (6)	N1—Ni—Cl	96.17 (4)
N1—Ni—N2	166.41 (5)	N2—Ni—Cl	97.41 (4)

The H atoms were placed in calculated positions (C—H = 0.93–0.98 Å) and refined as riding with the constraint U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(methyl carrier) applied.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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, 1997); software used to prepare material for publication: *UdMX* (Maris, 2004).

The authors are grateful to the Natural Sciences and Engineering Research Council of Canada and the Fonds Québécois de la Recherche sur la Nature et les Technologies for financial support.

References

Albrecht, M., Lutz, M., Schreurs, A. M. M., Lutz, E. T. H., Spek, A. L. & van Koten, G. (2000). *J. Chem. Soc. Dalton Trans.* pp. 3797–3809.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SMART* (Version 5.059), *SAINT* (Version 6.06) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Grove, D. M., Verschuuren, A. H. M. & van Koten, G. (1989). *J. Organomet. Chem.* **372**, C1–C6.
Grove, D. M., van Koten, G. & Zoet, R. J. (1983). *J. Am. Chem. Soc.* **105**, 1379–1380.
Maris, T. (2004). *UdMX*. Université de Montréal, Canada.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Terheijden, J., van Koten, G., Muller, F., Grove, D. M., Vrieze, K., Nielsen, E. & Stam, C. H. (1986). *J. Organomet. Chem.* **315**, 401–407.