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Chloro[1-(dimethylaminoethyl)indenyl](triphenylphosphine)nickel(II)

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Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.060
wR factor = 0.168
Data-to-parameter ratio = 14.1

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

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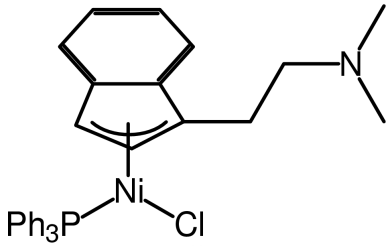
The title compound, $[\eta^5{:}\eta^0$–Ind$(CH_2)_2NMe_2](PPh_3)NiCl$ or $[NiCl(C_{13}H_{16}N)(C_{18}H_{15}P)]$, is a precatalyst for the polymerization of olefins. The present structure differs from a previously published polymorph of the same compound in the conformation of the amino tether (disordered over two positions with occupation factors 0.54/0.46) and in the cell parameters.

Comment

A recent review presents the many advantages and possibilities offered by transition-metal complexes of Cp-type ligands bearing a coordinating tether (Müller *et al.*, 2000). Our interest in the structural features and catalytic activities of the nickel indenyl complexes Ind$Ni(PR_3)X$ (Fontaine *et al.*, 1998; Dubois *et al.*, 2001) prompted us to explore the chemistry of analogous compounds bearing aminoalkyl side chains tethered to the indenyl ligand (Groux *et al.*, 2000). The combination of the title compound, $[\eta^5{:}\eta^0$–Ind$(CH_2)_2NMe_2](PPh_3)NiCl$, (I), and activators such as $AgBF_4$ and MAO (methylaluminoxane) catalyses the oligomerization of styrene and the polymerization of norbornene and ethylene (Groux & Zargarian, 2001; further publication in preparation).



(I)

The solid-state structure of (I) was determined from a single-crystal X-ray diffraction study carried out on crystals grown from a solution in Et_2O/hexanes at room temperature, and this structure analysis has already been published (Groux *et al.*, 2000). A new batch of crystals was obtained from a solution in CH_2Cl_2/hexanes at 253 K, and this paper reports a new solid-state structure of the same product (a different polymorph), differing only in the conformation of the disordered amino tether. No significant change in the main geometrical parameters is noted. Both crystal structures belong to space group $P\overline{1}$; the cell parameters for the first form were: a = 9.215 (2), b = 10.228 (4), c = 16.250 (6) Å; α = 76.60 (3), β = 87.94 (2), γ = 65.43 (2)°; V = 1351.8 (8) Å^3.

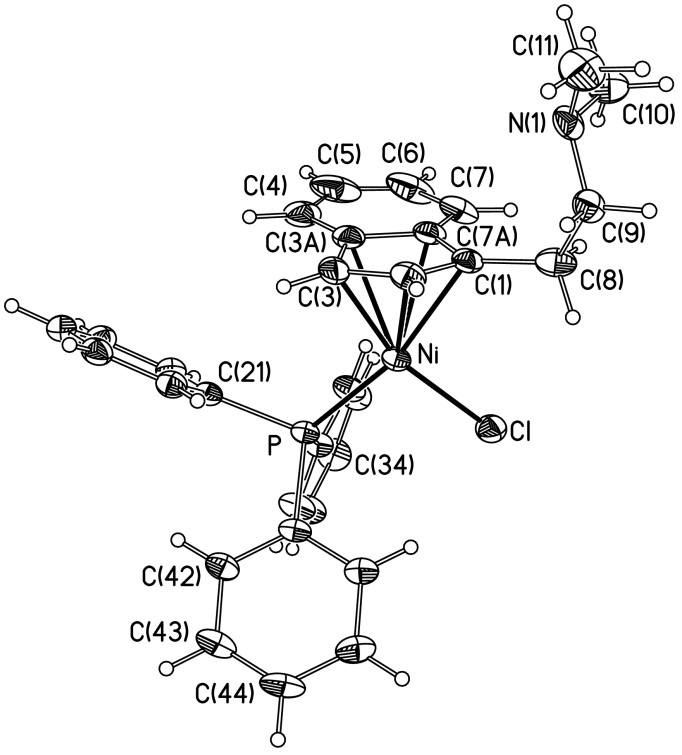


Figure 1
The molecular structure with ellipsoids at the 30% probability level. Disorder is not shown.

Experimental

The synthesis of (I) has been published elsewhere (Groux *et al.*, 2000). Single crystals suitable for X-ray diffraction study were obtained from a solution of (I) in CH_2Cl_2/hexanes at 253 K.

Crystal data

$[NiCl(C_{13}H_{16}N)(C_{18}H_{15}P)]$	$Z = 2$
$M_r = 542.70$	$D_x = 1.319$ Mg m^{-3}
Triclinic, $P\overline{1}$	Cu Kα radiation
$a = 9.0763$ (2) Å	Cell parameters from 1005 reflections
$b = 12.8096$ (3) Å	$\theta = 3.5$–$72.0°$
$c = 13.0012$ (3) Å	$\mu = 2.61$ mm^{-1}
$\alpha = 83.522$ (2)°	$T = 223$ (2) K
$\beta = 75.195$ (2)°	Block, dark red
$\gamma = 69.2873$ (14)°	0.65 × 0.65 × 0.34 mm
$V = 1366.51$ (5) Å^3	

Data collection

Bruker AXS SMART 2K/Platform diffractometer	5153 independent reflections
ω scans	4940 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$R_{int} = 0.084$
$T_{min} = 0.222$, $T_{max} = 0.411$	$\theta_{max} = 72.7°$
16367 measured reflections	$h = -11 \rightarrow 11$
	$k = -15 \rightarrow 15$
	$l = -14 \rightarrow 15$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.1192P)^2 + 0.404P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.060$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.168$	$\Delta\rho_{max} = 0.69$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.49$ e Å^{-3}
5153 reflections	Extinction correction: *SHELXL*97
366 parameters	Extinction coefficient: 0.0016 (5)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Ni—C3	2.025 (3)	C1—C8*a*	1.497 (10)
Ni—C2	2.071 (2)	C2—C3	1.417 (4)
Ni—C1	2.147 (2)	C3—C3A	1.453 (5)
Ni—Cl	2.1803 (7)	C3A—C7A	1.422 (4)
Ni—P	2.1817 (6)	N1*a*—C9*a*	1.416 (11)
Ni—C3A	2.314 (3)	N1*a*—C10*a*	1.451 (10)
Ni—C7A	2.349 (2)	N1*a*—C11*a*	1.460 (10)
C1—C2	1.399 (4)	C8*a*—C9*a*	1.541 (10)
C1—C7A	1.462 (3)		
C3—Ni—C1	66.10 (10)	C3—Ni—P	101.65 (8)
C3—Ni—Cl	161.82 (8)	C1—Ni—P	165.22 (7)
C1—Ni—Cl	95.94 (7)	Cl—Ni—P	96.53 (3)

H atoms were constrained with a riding model (*SHELXL*97 defaults); U_{iso}(H) was set at 1.5 (methyl) or 1.2 (others) times U_{eq} of the parent atom. Occupancy factors for the disordered amino tether were initially refined with fixed displacement parameters, then were fixed while displacement parameters were refined; restraints were applied to interatomic distances within the disordered group.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL*97.

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