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Three different functions of a phosphinic amidato ligand in *L*Ni(*L*Li)(*L*H) {*L* = [^{t}Bu$_2$P(O)NEt]$^-$}

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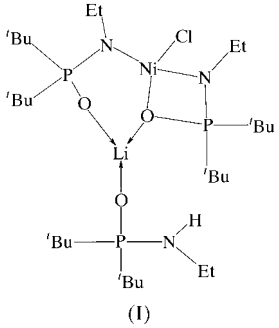
The title paramagnetic compound, chloro-2κ*Cl*-[di-*tert*-butylphosphinic ethylamide-1κ*O*]bis[μ-di-*tert*-butylphosphinic ethylamidato(1−)-1:2κ^4*O*:*N*]lithium(I)nickel(II), [NiLiCl(C$_{10}$H$_{23}$NOP)$_2$(C$_{10}$H$_{24}$NOP)], revealed an incomplete bischelation of Ni^{2+} by L^- {L = [^{t}Bu$_2$P(O)NEt]$^-$}, with the formation of a pseudo-tetrahedral NiON$_2$Cl chromophore. The Ni atom is coordinated by Cl$^-$, bidentate L^- and monodentate *L*Li (*via* N).

Comment

The paramagnetic (μ_{eff} = 3.3 BM at 300 K) title compound, (I), revealed an incomplete bischelation of Ni^{2+} by L^- {L = [^{t}Bu$_2$P(O)NEt]$^-$} with formation of a pseudo-tetrahedral NiON$_2$Cl chromophore. The Ni atom is coordinated by Cl$^-$, bidentate L^- and monodentate *L*Li (*via* N). The coordination



(I)

is pseudo-tetrahedral with a dihedral angle of 68.8 (1)° between the planes defined by Ni/O2/N2 and Ni/Cl/N1. A trigonal–planar LiO$_3$ coordination [angles at Li of 107.9 (3), 123.4 (3) and 128.7 (3)°] is formed by bonds between Li and the pendant ligand [1.801 (6) Å], L^- [1.842 (6) Å] and protonated L^- [1.825 (6) Å]. Preliminary results have been reported elsewhere (Wunderlich, 1996).

Experimental

The title compound, (I), was isolated from the products obtained in a metathesis reaction of *L*Li and (PPh$_3$)$_2$NiCl$_2$ {L = [^{t}Bu$_2$P(O)NEt]$^-$} (Brück, 1995; Brück *et al.*, 1996).

Crystal data

[NiLiCl(C$_{10}$H$_{23}$NOP)$_2$-(C$_{10}$H$_{24}$NOP)]
M_r = 714.90
Monoclinic, $P2_1/n$
a = 17.304 (4) Å
b = 13.055 (3) Å
c = 19.444 (4) Å
β = 105.80 (2)°
V = 4226.5 (16) Å^3
Z = 4
D_x = 1.123 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 40 reflections
θ = 10.0–12.6°
μ = 0.665 mm^{-1}
T = 300 (2) K
Square bipyramid, metallic dark brown
0.50 × 0.50 × 0.50 mm

Data collection

Siemens/Bruker *P*3 diffractometer
ω–2θ scans
7746 measured reflections
7461 independent reflections
4206 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.05°
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 15$
$l = -23 \rightarrow 22$
3 standard reflections
every 100 reflections
intensity decay: 19%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.133
S = 0.933
7468 reflections
421 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0736P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

All H atoms have been calculated and included in a riding mode except for H3 which has been refined using a restrained N—H distance [N3—H3 0.853 (14) Å].

Data collection: *P*3 *Software* (Siemens, 1989); cell refinement: *P*3 *Software*; data reduction: *SHELXTL-Plus* (Sheldrick, 1990); program(s) used to solve structure: *SHELXTL-Plus*; program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97.

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