Mono(amido-diphosphine) complexes of yttrium: synthesis and x-ray crystal structure of {Y(.eta.3-C3H5)[N(SiMe2CH2PMe2)2]}2(.mu.-Cl)2

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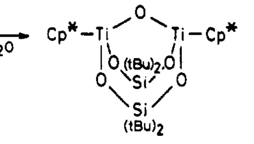
drogen atom is end-on bonded. The hydrogen bridge (H(2)–O(1)) gives an increase of the coordination number at O(1), resulting in an increase of the Ti(1)–O(1) bond length (187.2 (2) pm). Normally a water molecule would result and then be eliminated. In this case, however, steric factors prevent the reaction of the OH groups. A frozen situation results. We have not been able to eliminate water from 4 on a preparative scale to yield 5.[11] However, 5 has been prepared in 45% yield by reacting Cp*Li, $TiCl_3·3THF$, $(tBu)_2Si(OLi)_2$, and oxygen.[12]



5

The titanium atoms in 4 have a tetrahedral coordination, forming the well-known piano-stool structure. The distances within the eight-membered ring are comparable with those in known examples.[5] To demonstrate that the OH group in 3 does not migrate from the silicon to the titanium atom, an X-ray investigation was undertaken. The single-crystal structure shows clearly that in 3 the OH group is bonded to silicon.[13]

A few multinuclear hydroxide derivatives of zirconium have been reported;[14] however, none contain O–H–O bonds.

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Supplementary Material Available: Tables of crystallographic details, positional parameters, isotropic and anisotropic parameters, and all bond distances and angles and a figure showing the structure for 3 (8 pages). Ordering information is given on any current masthead page.

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[11] Heating 4 in the presence of a molecular sieve forms compound 5 in moderate yield.

[12] Compound 5: A solution of Cp*Li (0.71 g, 5 mmol) and $TiCl_3·3THF$ (1.85 g, 5 mmol) in THF (60 mL) is stirred for 2 days. A second solution of $(tBu)_2Si(OLi)_2$ (0.94 g, 5 mmol) in THF (40 mL) is added dropwise at −78 °C. The solution turns deep green. After the solution is stirred for 24 h, the solvent is removed in vacuo. The residue is treated with hexane (70 mL) and finally filtered. A small amount of O_2 is passed through the solution until it turns yellow. After the solution is concentrated (30 mL) and cooled in a refrigerator, 0.83 g (45%) of yellow crystals separated; mp 142–144 °C. MS (EI): m/z 673 ($M^+ - Bu$). IR: 1392 m, 1261 m, 1096 m, 1018 m, 903 s, 863 s, 824 s, 641 s, 572 m cm^{-1}.

[13] Crystallographic data for 3: monoclinic, $a = 877.6$ (1) pm, $b = 1309.5$ (3) pm, $c = 1067.4$ (1) pm, $\beta = 109.52$ (1) °, $V = 1.1561$ (3) nm^3, space group $P2_1$, $Z = 2$, $\rho_{calcd} = 1.23$ Mg m^{-3}, 1822 independent reflections $[F_o > 3\sigma(F_o)]$, $2\theta = 2–45°$, weighting scheme $w^{-1} = \sigma^2(F) + 0.0004F^2$, refinement $R = 4.18\%$, $R_w = 5.42\%$ for all data.[10b]

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Mono(amido–diphosphine) Complexes of Yttrium: Synthesis and X-ray Crystal Structure of $\{Y(\eta^3-C_3H_5)[N(SiMe_2CH_2PMe_2)_2]\}_2(\mu-Cl)_2$

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Summary: The reaction of allylmagnesium chloride with the bis(amido–phosphine) complex $YCl[N(SiMe_2CH_2PMe_2)_2]_2$ (**1**) results in displacement of one of the tridentate ligands rather than simple metathesis of the chloride to generate the new complex $\{Y(\eta^3-C_3H_5)[N(SiMe_2CH_2PMe_2)_2]\}_2(\mu-Cl)_2$. Careful product analysis showed that the identity of the magnesium byproduct was in fact the bis(amide) derivative $Mg[N(SiMe_2CH_2PMe_2)_2]_2$; improved procedures utilize the diallylmagnesium compound $Mg(C_3H_5)_2·(dioxane)$, which obviates the production of $MgCl_2$. Attempts to introduce hydrocarbyl groups on the mono(ligand) derivative $YCl_2[N(SiMe_2CH_2PMe_2)_2]$ by control of stoichiometry have so far failed, with the exception of the formation of the bis(allyl) derivative $[Y(\eta^3-C_3H_5)_2[N(SiMe_2CH_2PMe_2)_2]$ by the reaction with $Mg(C_3H_5)_2·(dioxane)$; this bis(allyl) derivative acts as a catalyst precursor for the polymerization of ethylene.

Phosphine ligands are still found infrequently in complexes in the group 3[1-6] and lanthanide metals.[1] We have recently described derivatives of yttrium(III) containing two hybrid ligands that incorporate an amide ligand flanked by two phosphine donors into a potentially tridentate chelate array; complexes of the formula $YCl[N(SiMe_2CH_2PMe_2)_2]_2$ (**1**) have been shown to be seven-coordinate and fluxional.[5] Although the hydrocarbyl derivatives $YR[N(SiMe_2CH_2PMe_2)_2]_2$ (**2**; R = Ph, $PhCH_2$) could be prepared by metathesis of the chloride of **1** with organolithium or potassium reagents,[5] the chemistry of these complexes was dominated by hydrocarbon elimination and

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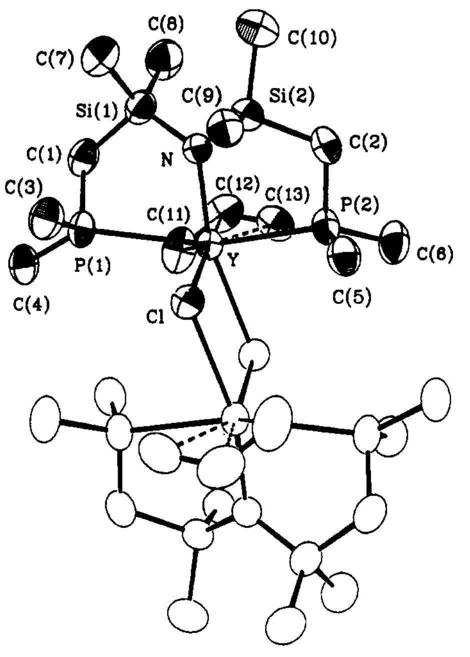

Figure 1. ORTEP view of 4 showing the atom-labeling scheme. Selected bond lengths (Å): Y–P1, 2.931 (1); Y–P2, 2.892 (1); Y–N, 2.292 (4); Y–Cl, 2.746 (1); Y–Cl′, 2.795 (1); Y–C11, 2.587 (5); Y–C12, 2.609 (5); Y–C13, 2.621 (5); C11–C12, 1.387 (9); C12–C13, 1.356 (8). Selected bond angles (deg): P1–Y–P2, 148.38 (4); N–Y–Cl′, 162.37 (10); N–Y–Cl, 97.88 (9); N–Y–P1, 76.01 (10); N–Y–P2, 80.75 (10); N–Y–allyl centroid, 100.0 (1); Cl′–Y–Cl, 76.11 (4); Cl′–Y–P1, 118.52 (4); Cl′–Y–P2, 81.94 (4); Cl′–Y–allyl centroid, 87.5 (1); C11–C12–C13, 126.1 (6).

formation of the very stable cyclometalated product $\overline{\mathrm{Y[N(SiMe_2CH}}\mathrm{PMe_2)(SiMe_2CH_2PMe_2)[N(SiMe_2CH_2PMe_2)_2]}$ (**3**). Interestingly, when organomagnesium reagents were used in an attempt to generate hydrocarbyl complexes similar to the bis(ligand) derivatives **2**, a new class of phosphine complexes of yttrium(III) was generated having only one tridentate amido–phosphine unit per yttrium. In this communication we describe our initial results in this area.

The allyl Grignard reagent $(C_3H_5)MgCl$ reacts with the bis(ligand) complex $YCl[N(SiMe_2CH_2PMe_2)_2]_2$ (**1**)[5] to produce the dimeric mono(amido–diphosphine) compound $\{Y(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]\}_2(\mu\text{-}Cl)_2$ (**4**); the structure of **4** was determined by single-crystal X-ray crystallography and is shown in Figure 1.[7] Assignment of two coordination sites to the η^3-allyl moiety makes the complex seven-coordinate at each yttrium center. This arrangement is best described as two distorted pentagonal bipyramids fused together via two bridging chlorides; each chloride is axial on one metal and equatorial on the other. In the allyl complex **4** the N–Y–Cl′ bond angle is 162.37°, a deviation of 17.6° from the ideal. The N–Y–equatorial bond angles range from 76.01 to 118.52° but average to 89.8°, close to the expected 90°. The Y–P bond lengths of 2.892 (1) and 2.931 (1) Å are similar to those found in the two other yttrium–phosphine complexes structurally characterized.[3,5]

The allyl complex **4** is fluxional and undergoes fast syn–anti allyl exchange. In the high-temperature limit, the ^1H and $^{13}C\{^1H\}$ NMR spectra[8] each show five resonances: one signal for the central allyl proton (or its carbon), one signal for the equivalent syn and anti allyl hydrogens (or their carbons), and three signals for the amido–diphosphine ligand. However, syn–anti exchange does not completely explain the high symmetry of these NMR spectra. A fluxional process which equates both sides of the amido–diphosphine ligand must also be invoked. If the compound maintains its dimeric structure, then the simplest explanation is that phosphine donors exchange sites via dissociation, rotation about the metal–amide bond, and phosphine recoordination. Pseudorotation or stereochemical nonrigidity, well-known processes for seven-coordinate complexes, are also possible, but phosphine dissociation seems simpler and has been invoked previously[5] for the bis(amido–diphosphine) complexes $YCl[N(SiMe_2CH_2PR_2)_2]_2$.

At the low-temperature limit, the NMR spectra of **4** are in agreement with the X-ray structure. The ^1H and $^{13}C\{^1H\}$ NMR spectra[8] reflect the inequivalent environments on either side of the tridentate ligand plane. There are two signals for the phosphorus methyl protons and carbons, two for the silyl methyl hydrogens and carbons, two resonances for the methylene protons, and one signal for the methylene carbons. The syn–anti exchange of the allyl moiety is also frozen out such that two carbon and three proton resonances (showing resolved $^3J_{H-H}$ couplings) are present. Only a doublet ($^1J_{Y-P}$ = 81.8 Hz) is observed in the $^{31}P\{^1H\}$ NMR spectrum, regardless of the temperature.

The formation of the mono(amido–diphosphine) compound $\{Y(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]\}_2(\mu\text{-}Cl)_2$ (**4**) from the reaction of allylmagnesium chloride and the bis(ligand) complex $YCl[N(SiMe_2CH_2PMe_2)_2]_2$ (**1**) was unexpected. When this reaction was monitored by $^{31}P\{^1H\}$ NMR spectroscopy, the spectrum showed the presence of only two phosphorus-containing compounds: a doublet at −39.7 ppm ($^1J_{Y-P}$ = 81.8 Hz), which was assigned to the allyl dimer **4**, and a singlet at −57 ppm presumably due to the magnesium chloro amide "$MgCl[N(SiMe_2CH_2PMe_2)_2]$"; this last assignment seemed reasonable on the basis of mass balance (eq 1).

$$\underset{\mathbf{1}}{YCl[N(SiMe_2CH_2PMe_2)_2]_2} + C_3H_5MgCl \xrightarrow{THF} 1/2\,\{Y(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]\}_2(\mu\text{-}Cl)_2\ \mathbf{4} + \text{"}MgCl[N(SiMe_2CH_2PMe_2)_2]\text{"} \qquad (1)$$

The two products formed in eq 1 could be separated by fractional crystallization. Unfortunately, the elemental analyses for the isolated allyl dimer **4** did not agree with the indicated formulation; in addition, on standing, toluene solutions of this material deposited some insoluble white compound. The isolated crystals of "$MgCl[N(SiMe_2CH_2PMe_2)_2]$" gave an elemental analysis consistent with the bis(amide) formula $Mg[N(SiMe_2CH_2PMe_2)_2]_2$. The analysis of the recrystallized allyl complex was then reinterpreted as indicating the presence of $MgCl_2$; a for-

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(7) $\{Y(\eta^3\text{-}C_3H_5)[N(SiMe_2CH_2PMe_2)_2]\}_2(\mu\text{-}Cl)_2$ (4): monoclinic, $P2_1/a$, a = 12.0949 (9) Å, b = 14.2647 (11) Å, c = 13.278 (2) Å, β = 101.555 (8)°, V = 2244.3 (4) Å^3, Z = 2, D_c = 1.320 g cm^{-3}, $\mu(MoK\alpha)$ = 29.74 cm^{-1}. The structure was solved by the Patterson method and was refined by full-matrix least-squares procedures to R = 0.035 and R_w = 0.038 for 2272 reflections with $I \geq 3\sigma(I)$. The data were corrected for absorption (analytical method); transmission factors ranged from 0.49 to 0.70. Complete details of the structure determination are included as supplementary material.

(8) NMR spectral data for 4 (reported in $C_6D_5CD_3$) are as follows. **^1H NMR** (δ in ppm): at +80 °C 6.15 (1 H, p, J = 13 Hz, H_m), 3.15 (4 H, broad, H_s, H_a), 1.18 (12 H, s, PMe), 0.70 (4 H, s, PCH_2Si), and 0.14 (12 H, s, SiMe); at +20 °C, 6.19 (1 H, sept, J = 12 Hz, H_m), 3.64 (2 H, broad, H_s), 2.83 (2 H, broad, H_a), 1.13 (12 H, s, PMe), 0.65 (4 H, s, PCH_2Si), and 0.18 (12 H, s, SiMe); at −50 °C, 6.30 (1 H, t of t, J = 8.6, 15.6 Hz, H_m), 3.75 (2 H, d, J = 8.6, H_s), 2.95 (2 H, d, J = 15.6 H_a), 1.14 (6 H, s, PMe), 1.04 (6 H, s, PMe), 0.69 (2 H, s, PCH_2Si), 0.64 (2 H, s, PCH_2Si), 0.31 (6 H, s, SiMe), and 0.23 (6 H, s, SiMe). **$^{13}C\{^1H\}$ NMR** (δ, ppm [$^1J_{C-H}$, Hz]): 144.24 (s, C_i) [d, 143.5], 72.68 (s, C_o) [t, 150.1], 20.09 (s, PCH_2Si) [t, 120.9], 15 (broad, PMe), 6.34 (s, SiMe) [q, 117.1]. **$^{31}P\{^1H\}$ NMR** (δ, ppm): −39.7 (d, ^1J_Y = 81.8 Hz).

mulation of $\{\mathrm{YCl(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]}\}_2\mathrm{(MgCl_2)_{0.7}}$ better fits the analytical data[9] and implies that $\mathrm{MgCl_2}$ was the insoluble material that slowly precipitated from solutions of **4**. Therefore, the products are actually $\{\mathrm{Y(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]}\}_2(\mu\text{-Cl})_2$, $\mathrm{MgCl_2}$, and $\mathrm{Mg[N(SiMe_2CH_2PMe_2)_2]_2}$ (eq 2).

$$\underset{\mathbf{1}}{2\,\mathrm{YCl[N(SiMe_2CH_2PMe_2)_2]_2}} + 2\,\mathrm{C_3H_5MgCl} \xrightarrow{\text{THF}} \{\mathrm{Y(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]}\}_2(\mu\text{-Cl})_2\ \mathbf{4} + \mathrm{Mg[N(SiMe_2CH_2PMe_2)_2]_2} + \mathrm{MgCl_2} \qquad (2)$$

It is possible that the reaction proceeds initially via an exchange of ligands between the magnesium and yttrium metals (an allyl moiety for an amido–diphosphine ligand) followed by disproportionation of $\mathrm{MgCl[N(SiMe_2CH_2PMe_2)_2]}$ to give $\mathrm{Mg[N(SiMe_2CH_2PMe_2)_2]_2}$ and $\mathrm{MgCl_2}$. The monomeric yttrium allyl complex $\mathrm{YCl(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]}$ could then either dimerize to give **4** or coordinate the $\mathrm{MgCl_2}$ to give $\mathrm{YCl(C_3H_5)[N(SiMe_2CH_2PMe_2)_2](MgCl_2)}$; another possibility is that the $\mathrm{MgCl_2}$ associates weakly with the dimer **4**.

In an effort to improve this reaction and simplify the isolation of products, the diallylmagnesium reagent $\mathrm{Mg(C_3H_5)_2{\cdot}(dioxane)}$ was utilized in the reaction with 2 equiv of $\mathrm{YCl[N(SiMe_2CH_2PMe_2)_2]_2}$. The only products detected were $\{\mathrm{Y(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]}\}_2(\mu\text{-Cl})_2$ (**4**) and $\mathrm{Mg[N(SiMe_2CH_2PMe_2)_2]_2}$ (eq 3). Both complexes gave satisfactory elemental analyses[10] and NMR data[8] identical with those recorded for the compounds isolated using eq 2.

$$\underset{\mathbf{1}}{2\,\mathrm{YCl[N(SiMe_2CH_2PMe_2)_2]_2}} + \mathrm{Mg(C_3H_5)_2{\cdot}C_4H_8O_2} \xrightarrow{\text{THF}} \{\mathrm{Y(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]}\}_2(\mu\text{-Cl})_2\ \mathbf{4} + \mathrm{Mg[N(SiMe_2CH_2PMe_2)_2]_2} \qquad (3)$$

This reaction appears to be quite general, although not in a synthetic sense. The reaction of a number of magnesium reagents does result in ligand displacements, as evidenced by the formation of $\mathrm{Mg[N(SiMe_2CH_2PMe_2)_2]_2}$ ($^{31}\mathrm{P}\{^1\mathrm{H}\}$ NMR spectroscopy); however, the yttrium-containing products have so far resisted purification except for the allyl derivative **4**. For example, the reaction of $\mathrm{Mg(CH_2Ph)_2(THF)_2}$[11] with 2 equiv of $\mathrm{YCl[N(SiMe_2CH_2PMe_2)_2]_2}$ does generate what is presumed to be $\{\mathrm{Y(CH_2Ph)[N(SiMe_2CH_2PMe_2)_2]}\}_2(\mu\text{-Cl})_2$ (**5**), but we have not been able to separate it from the magnesium bis(amide) reagent.[12] As expected, the bis(ligand) benzyl complex $\mathrm{Y(CH_2Ph)[N(SiMe_2CH_2PMe_2)_2]_2}$[5] was not detected in this reaction.

So far, this is the only method we have found to cleanly prepare hydrocarbyl complexes of yttrium(III) which contain only one of the tridentate ligands $^-\mathrm{N(SiMe_2CH_2PMe_2)_2}$. The reaction of the mono(ligand) dichloride derivative $\mathrm{YCl_2[N(SiMe_2CH_2PMe_2)_2]}$ (**6**; prepared[13] from $\mathrm{YCl_3}$ and 1 equiv of $\mathrm{KN(SiMe_2CH_2PMe_2)_2}$ in THF) with a variety of organometallic reagents leads to intractable products with the exception of the reaction of $\mathrm{Mg(C_3H_5)_2{\cdot}C_4H_8O_2}$. Although none of the allyl dimer **4** was produced, we have identified the only product (eq 4) as

$$\underset{\mathbf{6}}{\mathrm{YCl_2[N(SiMe_2CH_2PMe_2)_2]}} + \mathrm{Mg(C_3H_5)_2{\cdot}C_4H_8O_2} \xrightarrow[(-\,\mathrm{MgCl_2})]{\text{THF}} \underset{\mathbf{7}}{\mathrm{Y(C_3H_5)_2[N(SiMe_2CH_2PMe_2)_2]}} \qquad (4)$$

being the bis(allyl)complex $\mathrm{Y(C_3H_5)_2[N(SiMe_2CH_2PMe_2)_2]}$ (**7**), on the basis of spectroscopic data.[14] Preliminary results indicate that the bis(allyl) complex polymerizes ethylene, whereas neither the allyl dimer **4** nor the bis(ligand) derivatives $\mathrm{YR[N(SiMe_2CH_2PMe_2)_2]_2}$ (**3**) show any reactivity with added ethylene. The further reactivity patterns of these mono(ligand)yttrium derivatives are currently under investigation, as are extensions to the lanthanide elements.

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Supplementary Material Available: Text giving full experimental details for the preparation of all new complexes, including details of the X-ray structure analysis of **4**, and tables of crystallographic data, positional parameters for non-hydrogen atoms, bond distances and angles, hydrogen atom parameters, anisotropic thermal parameters, and torsion angles for **4** (15 pages). Ordering information is given on any current masthead page.

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[9] Anal. Calcd for $\{\mathrm{Y}(\mu\text{-Cl})\mathrm{(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]}\}_2{\cdot}0.7\mathrm{MgCl_2}$, $\mathrm{C_{26}H_{66}N_2P_4Si_4Y_2Cl_2(MgCl_2)_{0.7}}$: C, 32.58; H, 6.94; N, 2.92. Found: C, 32.68; H, 7.07; N, 2.86.

[10] Anal. Calcd for $\{\mathrm{Y(C_3H_5)[N(SiMe_2CH_2PMe_2)_2]}\}_2(\mu\text{-Cl})_2$, $\mathrm{C_{13}H_{33}NP_2Si_2YCl}$: C, 35.02; H, 7.46; N, 3.14; Cl, 7.95. Found: C, 35.40; H, 7.52; N, 3.16; Cl, 7.71. Anal. Calcd for $\mathrm{Mg[N(SiMe_2CH_2PMe_2)_2]_2}$, $\mathrm{C_{20}H_{56}N_2P_4Si_4Mg}$: C, 41.05; H, 9.64; N, 4.79. Found: C, 40.77; H, 9.44; N, 4.78.

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[12] NMR spectral data for **5** (reported in $\mathrm{C_6D_6}$) are as follows. ^{1}**H NMR** (δ, ppm): 7.10 (2 H, m, $\mathrm{H_o}$), 6.40 (2 H, m, $\mathrm{H_m}$), 6.55 (1 H, m, $\mathrm{H_p}$), 2.05 (2 H, m, $\mathrm{YC}H_2\mathrm{Ph}$), 0.90 (12 H, s, PMe), 0.60 (4 H, s, $\mathrm{PCH_2Si}$), and 0.25 (12 H, s, SiMe). ^{31}**P{^{1}H} NMR** (δ, ppm): −40.84 (d, $^1J_{\mathrm{Y}}$ = 81.3 Hz).

[13] NMR spectra for $\mathrm{YCl_2[N(SiMe_2CH_2PMe_2)_2]}$ are reported in $\mathrm{C_6D_5CD_3}$/THF. ^{1}**H NMR** (δ, ppm): 1.04 (12 H, t, J = 1.9 Hz, PMe), 1.02 (4 H, t, J = 2.8, $\mathrm{PCH_2Si}$), and 0.30 (12 H, s, SiMe). ^{13}**C{^{1}H} NMR** (δ, ppm): 19.75 (s, $\mathrm{PCH_2Si}$), 14.06 (s, PMe), and 6.34 (s, SiMe). ^{31}**P{^{1}H} NMR** (δ, ppm): −41.8 (d, $^1J_{\mathrm{Y}}$ = 77 Hz). Anal. Calcd for $\mathrm{C_{10}H_{28}NP_2Si_2YCl_2}$: C, 27.28; H, 6.41; N, 3.18; Cl, 16.11. Found: C, 27.50; H, 6.50; N, 3.08; Cl, 15.95. The low solubility of this material may be due to an oligomeric structure, although we have not confirmed this.

[14] NMR spectral data and elemental analyses are given for **6**; although this is the only material detected by $^{31}\mathrm{P}\{^1\mathrm{H}\}$ NMR spectroscopy, so far only a yellow oil has been isolated. ^{1}**H NMR** (δ, ppm): at 20 °C, 6.09 (2 H, p, J = 11.5 Hz), 3.4 (4 H, broad, $\mathrm{H_s}$), 2.6 (4 H, broad, $\mathrm{H_a}$), 0.91 (12 H, t, J = 1.8, PMe), 0.58 (4 H, t, J = 3.9, $\mathrm{PCH_2Si}$), and 0.18 (12 H, s, SiMe); in $\mathrm{C_6D_5CD_3}$ at −40 °C, 6.11 (2 H, t of t, J = 8.8, 15.4), 3.44 (4 H, d, J = 8.8, $\mathrm{H_s}$), 2.56 (4 H, d, J = 15.4, $\mathrm{H_a}$), 0.88 (12 H, broad, PMe), 0.49 (4 H, broad, $\mathrm{PCH_2Si}$), and 0.20 (12 H, s, SiMe). ^{13}**C{^{1}H} NMR** (δ, ppm [$^1J_{\mathrm{C-H}}$, Hz]): 143.54 (s, $\mathrm{C_i}$) [d, 142.6], 67.71 (s, $\mathrm{C_o}$) [t, 150.0], 19.56 (t, J = 3, $\mathrm{PCH_2Si}$) [t, 120.1], 14.65 (t, J = 3, PMe), and 6.74 (s, SiMe) [q, 117.4]. ^{31}**P{^{1}H} NMR** (δ, ppm): −38.3 (d, J = 80.8 Hz).