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Yttrium Alkyl Complex with a Linked Bis(amidinate) Ancillary Ligand[†]

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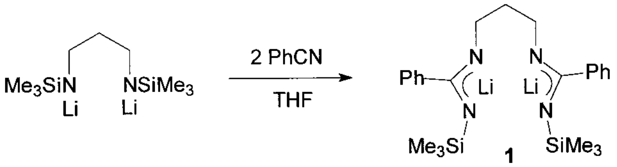
The dilithium salt of a linked bis(amidinate) dianionic ligand, $Li_2[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3]$ (**1**), was prepared by reaction of dilithiated *N,N′*-bis(trimethylsilyl)-1,3-diaminopropane with benzonitrile. Reaction of **1** with $YCl_3(THF)_{3.5}$ gave $[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3]YCl(THF)_2$ (**2**), which, by reaction with $Li[CH(SiMe_3)_2]$, was converted to the alkyl complex $[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3]Y[CH(SiMe_3)_2](THF)$ (**3**). A structure determination of **3** showed that linking together the amidinate functionalities opens up the coordination sphere to allow the bonding of an additional molecule of THF not present in the unbridged bis(amidinate) analogue $[PhC(NSiMe_3)_2]_2Y[CH(SiMe_3)_2]$.

Introduction

Linking two anionic ancillary ligands together by a covalent bridge as a means of determining complex geometry and limiting ligand mobility has been a very useful concept in transition-metal chemistry. The prime example for this is provided by the *ansa*-bis(indenyl) complexes first designed by Brintzinger et al.[1] and highly successful in the stereoregular catalytic polymerization of propene.[2] This approach has since been applied to various other anionic functionalities such as amides and aryloxides.[3] The *N,N′*-bis(trimethylsilyl) benzamidinate monoanionic ligand has been extensively used in the past as an ancillary in main group and transition-metal chemistry.[4] We recently developed a method to prepare *N*-functionalized *N′*-$SiMe_3$-benzamidinates by reaction of various lithium mono(trimethylsilyl)amides with benzonitrile.[5] This approach also is useful for the preparation of covalently linked bis(amidinate) ancillary ligands. Here we describe the synthesis of a yttrium alkyl complex with a linked bis(amidinate) ligand and show that the linking of the two amidinate moieties results in a different ligand geometry and a different coordination behavior of the metal center compared to analogous complexes with two unlinked amidinate ligands.

Scheme 1



Results and Discussion

The synthesis of the *N,N′*-bis(trimethylsilyl)amidinate ligand by reaction of the amide $Li[N(SiMe_3)_2]$ with benzonitrile (proceeding via attack of the amide on the nitrile carbon followed by a trimethylsilyl shift) was first reported by Sanger.[6] This method can be extended to a range of mono(trimethylsilyl) amides $Li[N(SiMe_3)R]$ to yield the lithium salts of functionalized benzamidinates $Li[PhC(NSiMe_3)(NR)]$.[5,7] This approach can also be used to prepare linked bis(amidinate) salts. Deprotonation of the *N,N′*-bis(trimethylsilyl)-1,3-diaminopropane $Me_3Si(H)N(CH_2)_3N(H)SiMe_3$ with 2 equiv of *n*-BuLi, followed by reaction with 2 equiv of benzonitrile in a THF/pentane mixture, yields the linked bis(benzamidinate) salt $Li_2[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3]$ (**1**, Scheme 1). The compound was isolated analytically pure in 69% yield as a white solid containing 0.5 equiv of THF per formula unit.

Reaction of the dilithium salt **1** with $YCl_3(THF)_{3.5}$ in THF, followed by extraction with pentane, afforded the corresponding yttrium bis(amidinate) monochloride complex with two coordinated molecules of THF, $[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3]YCl(THF)_2$ (**2**, Scheme 2), as analytically pure material in 57% isolated yield. By reaction with $Li[CH(SiMe_3)_2]$ the chloride and one of the

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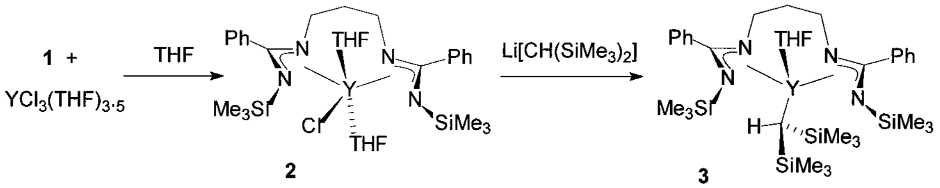
Scheme 2



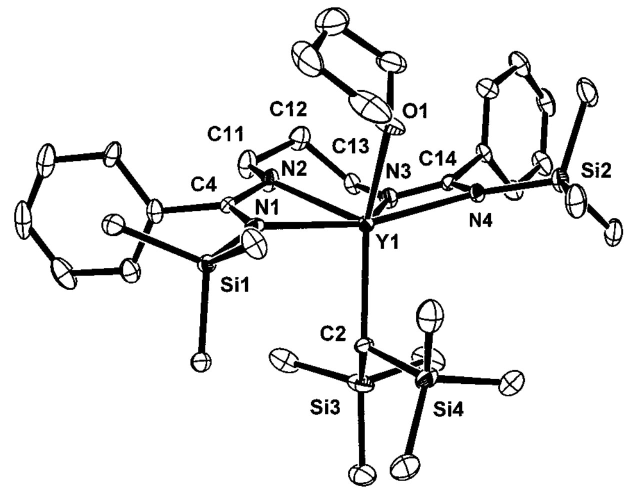


Figure 1. Molecular structure of [$Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3$]Y[$CH(SiMe_3)_2$](THF) (**3**). Thermal ellipsoids are drawn at the 30% level.

THF molecules in **2** can be replaced by the sterically demanding bis(trimethylsilyl)methyl group to give the alkyl complex [$Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3$]Y[$CH(SiMe_3)_2$](THF) (**3**, Scheme 2). The presence of a coordinated molecule of THF in **3** is a marked contrast with the nonbridged bis(amidinate) yttrium alkyl [$PhC(NSiMe_3)_2]_2Y[CH(SiMe_3)_2$] (**4**) reported previously by our group,[8] which is free from coordinated base or salt. The low-temperature ^1H NMR spectrum of **3** (−60 °C, toluene-d_8 solvent) shows two multiplets (2H each) for the diastereotopic NCH_2 methylene protons and two multiplets (1H each, one overlapped by THF) for the diastereotopic central CH_2 group of the ligand backbone, as well as one singlet each for the alkyl and amidinate $SiMe_3$ groups and two well-separated doublets for the phenyl *o*-protons. These data indicate an averaged C_s symmetry of the complex and slow rotation around the ligand C−Ph bond at this temperature. The ^1H and ^{13}C NMR resonances of the Y−CH group in **3**, δ −0.34 ppm ($^2J_{YH}$ = 1.8 Hz) and δ 35.8 ppm ($^1J_{YC}$ = 32 Hz, $^1J_{CH}$ = 93 Hz), are downfield by 0.6 ppm and upfield by 7.7 ppm, respectively, relative to the resonances in unbridged, base-free **4**.

A crystal structure determination of **3** (Figure 1, pertinent interatomic distances and angles in Table 1) confirms the monomeric nature of the complex and the chelating bonding mode of the linked bis(amidinate) ligand. The unit cell contains two independent molecules that do not differ significantly in geometry; only one of the two molecules is shown, and its geometrical parameters are used for the discussion of the structure. The amidinate moieties are each dihapto bound through the nitrogen atoms, with the Y−N−C−N units only deviating slightly from planarity (dihedral angles Y(1)−N(1)−C(4)−N(2) = −15.6(7)°, Y(1)−N(4)−C(14)−N(3) = −7.0(7)°). By comparing the bis(amidinate)Y geometry in **3** with that of the structurally characterized *p*-methoxybenzamidinate derivative of the unbridged bis(amidinate) complex **4**, [(p-$MeOC_6H_4)C(NSiMe_3)_2]_2Y[CH(SiMe_3)_2$] (**5**),[8a] it can be seen that the amidinates respond to the tethering in two ways. In **3** there is now a substantial difference in Y−N distance for the amidinate nitrogens attached to the bridge compared to those attached to the $SiMe_3$ group (the former being 0.18 Å shorter). In **5**, all Y−N bond distances are around 2.33 ± 0.01 Å. In addition, the tethering of the amidinate functionalities in **3** restricts the angle between the two Y−N−C−N planes, which is now only 24.7(4)°, compared to 75.6(2)° in **5**. This opens up the coordination sphere of Y and allows for the coordination of an additional molecule of THF in **3**. One notable feature is the large C(24)−Y(1)−O(1) angle of 145.1(3)°. The gap left open in this way is occupied by a Si−Me group of the bis(trimethylsilylmethyl) ligand, which is distorted, Y(1)−C(24)−Si(4) = 106.1(4)° compared to Y(1)−C(24)−Si(3) = 121.3(4)°, to result in relatively short distances Y(1)···Si(4) = 3.415(3) Å and Y(1)···C(28) = 3.299(12) Å. This distortion of the alkyl group in **3** is less pronounced than similar types of distortions observed in mono- and bis-η^5-C_5Me_5 yttrium and lanthanide bis(trimethylsilyl)methyl complexes,[9] but such a distortion is completely absent in **5**.

Table 1. Selected Interatomic Distances (Å) and Angles (deg) for 3 (for one of the two independent molecules in the unit cell)

Y(1)−N(1)	2.488(6)	Y(1)−N(3)	2.298(7)
Y(1)−N(2)	2.308(8)	Y(1)−N(4)	2.469(7)
Y(1)−C(24)	2.410(8)	Y(1)−O(1)	2.395(7)
N(1)−Y(1)−N(2)	56.7(2)	N(1)−Y(1)−C(24)	89.6(2)
N(3)−Y(1)−N(4)	56.5(2)	N(2)−Y(1)−C(24)	110.8(3)
N(2)−Y(1)−N(3)	74.9(2)	N(3)−Y(1)−C(24)	101.3(3)
N(1)−Y(1)−N(4)	163.0(2)	N(4)−Y(1)−C(24)	104.4(2)
Y(1)−C(24)−Si(3)	121.3(4)	O(1)−Y(1)−C(24)	145.1(3)
Y(1)−C(24)−Si(3)	106.1(4)	Si(3)−C(24)−Si(4)	118.5(4)

The two 1,2-cyclohexanediyl-linked bis(amidinate) titanium compounds (including an unusual bis(amidinate) titanium arene complex) reported by Hagadorn and Arnold[10] and the compounds **2** and **3** described here are thus far the only complexes known with a chelating tethered bis(amidinate) ancillary ligand system. One bimetallic complex with a bridging linked bis(amidinate)

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ligand, derived from the reaction of an α,*ω*-bis(carbodiimide) with $CpTiMe_3$, has also been described.[11] These few examples already show that substantial differences in geometry and reactivity can be achieved by covalently linking amidinate functionalities. It is expected that the linked bis(amidinate) ligand system, together with the linked aminopyridinato[12] and aminotroponiminate[13] ligands, will form an interesting set of ancillary ligands for new transition-metal and lanthanide chemistry.

Conclusion

The reaction of lithium trimethylsilylamides with benzonitrile to give lithium (mono)trimethylsilyl benzamidinates can also be used to obtain tethered bis(amidinate) ligands, as shown by the synthesis of the 1,3-propanediyl-bridged species **1**. The two yttrium complexes synthesized with this ligand, the chloride **2** and the alkyl **3**, represent only the second example of complexes with a chelating bis(amidinate) ancillary ligand. The linking of the two amidinate functionalities alters the relative orientation of the amidinates in the complex and opens up the metal coordination sphere relative to the analogous unbridged systems.

Experimental Section

General Information. All experiments were performed under nitrogen atmosphere using standard glovebox and Schlenk techniques. Deuterated solvents, C_6D_6, C_7D_8, and THF-d_8 (Aldrich), were dried over Na/K alloy and vacuum transferred before use. Pentane and THF were distilled from Na or Na/K alloy before use. The compounds $YCl_3(THF)_{3.5}$,[14] $Li[CH(SiMe_3)_2]$,[15] and $(CH_2)_3(NHSiMe_3)_3$[16] were prepared according to literature procedures. Benzonitrile (Aldrich) was degassed and dried over molecular sieves (4 Å) before use. The amines $H_2N(CH_2)_3NH_2$ and $HN(SiMe_3)_2$ (Aldrich) were used as purchased. NMR spectra were run on Varian VXR-300 or Unity 500 spectrometers. Elemental analyses were performed by the Microanalytical Department of the University of Groningen. Every value is the average of at least two independent determinations.

Synthesis of $Li_2[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3]\cdot(THF)_{0.5}$ (1). To a solution of $Me_3Si(H)N(CH_2)_3N(H)SiMe_3$ (7.5 g, 34.3 mmol) in 30 mL of pentane was slowly added a solution of *n*-BuLi in hexane (26.0 mL, 2.5 M, 70.0 mmol). The reaction mixture was stirred overnight; then 30 mL of THF was added to the yellow suspension. Benzonitrile (7.0 mL, 69.0 mmol) was then added dropwise to the solution. The reaction mixture was stirred for 5 h, after which the solvent was removed in vacuo. The remaining crystalline solid was rinsed with 20 mL of pentane and dried to give 11.2 g (23.7 mmol, 69%) of the title compound. ^1H NMR (300 MHz, C_6D_6, 25 °C): δ 7.40 (d, $^3J_{HH} = 6.9$ Hz, 4H, *o*-Ph), 7.27 (t, $^3J_{HH} = 7.3$ Hz, 4H, *m*-Ph), 7.09 (t, $^3J_{HH} = 7.3$ Hz, 2H, *p*-Ph), 3.59 (m, Hz, α-CH_2 THF), 3.23 (t, $^3J_{HH} = 4.67$ Hz, 4H, NCH_2), 1.66 (br p, 2H, CH_2), 1.38 (m, β-CH_2 THF), 0.10 (s, 18H, $NSiMe_3$). ^{13}C NMR (75.4 MHz, C_6D_6, 25 °C): δ 179.4 (s, N*C*N), 142.3 (s, Ph C), 128.4 (d, $J = 160$ Hz, *o*-Ph), 127.2 (d, $J = 159$ Hz, *m*-Ph), 126.7 (d, $J = 160$ Hz, p-Ph), 68.2 (t, $J = 148$ Hz, α-CH_2 THF), 51.0 (t, $J = 134$ Hz, NCH_2), 32.62 (t, $J = 123$ Hz, CH_2), 25.3 (t, $J = 132$ Hz, β-CH_2 THF), 3.3 (q, $J = 118$ Hz, $SiMe_3$). Anal. Calcd for $C_{27}H_{42}N_4LiO_{0.5}Si_2$: C, 63.53; H, 8.10; N, 11.85; Li, 2.94. Found: C, 63.45; H, 8.11; N, 11.68; Li, 2.99.

Synthesis of $[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3]YCl(THF)_2$ (2). To a stirred suspension of $YCl_3(THF)_{3.5}$ (0.94 g, 2.1 mmol) in 30 mL of THF was added 0.99 g (2.1 mmol) of solid **1** in portions. The resulting yellowish solution was stirred overnight. Subsequently, the solvent was removed in vacuo and the remaining solid stripped of residual THF by stirring with 20 mL of pentane, which was then pumped off. The solid was extracted with pentane (5 × 40 mL), and the volume of the extract was reduced to 20 mL. Cooling to −30 °C yielded colorless crystals of **2** (0.82 g, 1.19 mmol, 57%). ^1H NMR (300 MHz, C_6D_6, 25 °C): δ 7.42 (br, 4H, *o*-Ph), 7.17 (t, 4H, $^3J_{HH} = 7.5$ Hz, *m*-Ph), 7.07 (t, 2H, $^3J_{HH} = 6.9$ Hz, *p*-Ph), 3.94 (br m, 8 H, α-CH_2 THF), 3.13 (m, 2 H, NC*H*H), 2.94 (m, 2 H, NCH*H*), 1.57 (m, 1H, C*H*H), 1.43 (br m, 8H, β-CH_2 THF), 1.39 (m, 1H, CH*H*), 0.30 (s, 18H, $SiMe_3$). ^{13}C NMR (75.4 MHz, C_6D_6, 25 °C): δ 178.9 (s, N*C*N), 138.7(s, Ph C), 128.3 (d, $J = 154$ Hz, *o*-Ph), 127.2 (d, $J = 158$ Hz, *m*-Ph), 126.4 (d, $J = 154$ Hz, *p*-Ph), 69.9 (t, $J = 148$ Hz, α-CH_2 THF), 48.4 (t, $J = 133$ Hz, NCH_2), 32.6 (br, CH_2), 25.5 (t, $J = 132$ Hz, β-CH_2 THF), 3.6 (q, $J = 118.3$ Hz, $SiMe_3$). Anal. Calcd for $C_{31}H_{50}N_4YClO_2Si_2$: C, 53.86; H, 7.29; N, 8.10; Y, 12.86. Found: C, 54.23; H, 7.40; N, 8.25; Y, 12.93.

Synthesis of $[Me_3SiNC(Ph)N(CH_2)_3NC(Ph)NSiMe_3]Y[CH(SiMe_3)_2]$ (THF) (3). To a stirred solution of **2** (0.48 g, 0.7 mmol) in 10 mL of THF was added 0.17 g (0.7 mmol) of $Li[CH(SiMe_3)_2]$. The resulting colorless solution was stirred for 3 h, after which the solvent was removed in vacuo. The remaining solid was stripped of residual THF by stirring with 5 mL of pentane, which was then pumped off. The residue was extracted with pentane (2 × 40 mL) and the volume of the extract reduced to 10 mL. Cooling to −20 °C overnight yielded 0.33 g (0.44 mmol, 64%) of colorless crystalline **3**. ^1H NMR (500 MHz, C_7D_8, −60 °C): δ 7.48 (d, 2H, $^3J_{HH} = 7.3$ Hz, *o*-Ph), 7.11−6.99 (m, 6 H, *m*- and *p*-Ph), 6.91 (d, 2H, $^3J_{HH} = 7.3$ Hz, *o*-Ph), 3.77 (m, 4 H, α-CH_2 THF), 3.06 and 2.96 (m, 2H each, NCH_2), 1.39 (m, 1H, C*H*H), 1.25 (m, 5H, CH*H* and β-CH_2 THF), 0.68 (s, 18H, $CSiMe_3$), 0.14 (s, 18H, $NSiMe_3$), −0.34 (d, $^2J_{YH} = 1.8$, Hz, 1H, YCH). ^{13}C NMR (75.4 MHz, C_6D_6, 25 °C): δ 179.5 (s, N*C*N), 138.6 (s, Ph C), 128.3 (d, $J = 157$ Hz, *o*-Ph), 128.1 (d, $J = 160$ Hz, *m*-Ph), 126.8 (d, $J = 160$ Hz, *p*-Ph), 68.9 (t, $J = 145$ Hz, α-CH_2 THF), 47.8 (t, $J = 134$ Hz, NCH_2), 35.8 (dd, $^1J_{YC} = 32$ Hz, $^1J_{CH} = 93$ Hz, YCH), 33.3 (t, $J = 125$ Hz, CH_2), 25.5 (t, $J = 131$ Hz, β-CH_2 THF), 5.6 (q, $J = 117$ Hz, CSi*Me*$_3$), 3.8 (q, $J = 118$ Hz, NSi*Me*$_3$). Anal. Calcd for $C_{34}H_{61}N_4YOSi_4$: C, 54.95; H, 8.27; N, 7.54; Y, 11.96. Found: C, 54.03; H, 8.08; N, 7.75; Y, 11.76. The carbon content determined for **3** is consistently and reproducibly too low, whereas reasonable values are obtained for the other elements. We have observed this behavior previously in other $Y[CH(SiMe_3)_2]$ compounds.[7] It may be associated with the formation of inert carbide species.

Crystal Structure Determination. A summary of crystallographic data is given in Table 2. A suitable crystal of **3** was obtained by cooling a pentane solution to −20 °C, mounted using inert handling techniques into the cold nitrogen stream of an Enraf-Nonius CAD-4F diffractometer. The unit cell parameters were obtained from a least-squares treatment of 22 reflections in the range $16.77° < \theta < 21.56°$. The $|E|$ distribution statistics indicated a non-centrosymmetric space group. Space group $P2_1$ was determined from considerations of the unit cell parameters, statistical analyses of intensity distributions, and (where appropriate) systematic absences. Examination of the final atomic coordinates with respect to molecular symmetry and coordinate equivalence suggested a centrosymmetric space group ($P2_1/c$), but the systematic extinction condition $h0l$: $l = 2n+1$ was heavily violated.

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Table 2. Crystallographic Data and Details of Structure Refinement Procedure for 3

formula	$C_{34}H_{61}N_4OSi_4Y$
fw	743.12
cryst dimens, mm	0.20 × 0.25 × 0.50
space group	$P2_1$, No. 4
temp, K	130
a, Å	18.763(1)
b, Å	10.205(1)
c, Å	22.391(2)
β, deg	106.383(7)
D_c, g cm^{-3}	1.198
formula Z	4
radiation (λ, Å)	0.710 73
no. of total rflns	9745
no. of unique rflns	9453
no. of obsd rflns ($F_o > 4.0\sigma(F_o)$)	7119
no. of params	818
μ, cm^{-1}	15.6
$wR(F_o^2)^a$	0.1128
weighting scheme:^{b} a, b	0.0322, 8.0825
Flack's x, twin ratio	0.72(2)
$R(F)^c$	0.0487
min, max resid density, e/Å^3	−0.54, 0.60(9)

[a] $wR(F^2) = [\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$ for $F_o^2 > 0$. [b] $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$ and $P = [\max(F_o^2,0) + 2F_c^2]/3$. [c] $R(F) = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$ for $F_o > 4.0\ \sigma\ (F_o)$.

Intensity data were corrected for Lorentz and polarization effects, scale factor and absorption (DIFABS)[17] and reduced to F_o^2. The structure was solved by Patterson methods and the model extended by direct methods applied to difference structure factors (DIRDIF).[18] Hydrogen atoms were included in the final refinement, introduced in calculated positions and riding on their carrier atoms. Flack's x refinement[19] gave an ambiguous result, probably due to enantiomorphic twinning. Enantiomorph twin refinement resulted in a value of 0.72(2). All calculations were performed using the SHELXL[20] and PLATON[21] programs.

Acknowledgment. This work was generously supported by ExxonMobil Chemical Company.

Supporting Information Available: Full details of the crystal structure determination of **3** including positional and thermal parameters. This material is available free of charge via the Internet at http://pubs.acs.org.

OM0008124

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