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Synthesis, crystal structure of bis(arylamido)lanthanide methyl complexes and their catalytic behavior for the polymerization of methyl methacrylate

Yunjie Luo [a,b], Yingming Yao [a], Wenjing Li [b], Jinglei Chen [a], Zhenqin Zhang [a], Yong Zhang [a], Qi Shen [a,*]

[a] *Department of Chemistry and Chemical Engineering, Suzhou University, Shizi Street, Suzhou 215006, China*

[b] *Department of Biological and Pharmaceutical Engineering, Ningbo Institute of Technology, Zhejiang University, Ningbo 315104, China*

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Abstract

Reactions of $[N(C_6H_3{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2LnCl(THF)$ (Ln = Nd, Yb) with two equivalents of MeLi in a mixture solution of toluene and Et_2O gave $[N(C_6H_3{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2LnCH_3(\mu\text{-}CH_3)Li(THF)_3 \cdot PhCH_3$ (Ln = Nd (**1**), Yb (**2**)) in good isolated yields as crystalline solids. The single-crystal structural analysis of **2** revealed that the coordination geometry of ytterbium ion is best described as a distorted pseudo-tetrahedron. Both **1** and **2** are active for the polymerization of methyl methacrylate to give syndiotactic-rich and high molecular weight polymers ($M_n > 10^4$) with relatively narrow molecular weight distributions ($M_w/M_n < 2$).

Keywords: Organolanthanide; Arylamide ligand; Crystal structure; Polymerization

1. Introduction

Over the past two decades, derivatives of the cyclopentadienyl anions played an especially important role in organolanthanide chemistry [1], since lanthanocene complexes have shown high activity for a variety of olefin transformations including hydrogenation [2,3], polymerization [4], hydroaminiation [3,5], hydrosilylation [6], and hydroboration [7]. Recently, alternative ligand environments other than cyclopentadienyl, for example, bidentate nitrogen-based ligands such as amidinates [8], guanidinates [9], and diketiminates [10] have been developed to understand their organolanthanide chemistry. In contrast, to date little effort has been devoted to explore the chemistry of monodentate arylamido lanthanide complexes [11], which are somewhat analogous to aryoxide lanthanide complexes.

As our extensive study on the affect of the coordination sphere around lanthanide elements on their catalytic activity, we are now attracted to arylamido ligands, for their steric bulk can be easily turned by the variation of substituents in the aryl ring. We synthesized bis(*N*-(2,6-diisopropylphenyl)-trimethylsilylamido)lanthanide methyl complexes and tested their catalytic activity for the polymerization of methyl methacrylate (MMA). Furthermore, to the best of our knowledge, it is the first example with respect to the catalytic activity of arylamido lanthanide complex. Here we report these results.

2. Results and discussion

2.1. Synthesis and characterization of bis(arylamido)lanthanide methyl complexes

As an alternative ligand to cyclopentadienyl, $[N(C_6H_3{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]^-$ anion was first employed by Schumann in synthesizing arylamido lanthanide

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* Corresponding author. Tel.: +86-512-6511-2513; fax: +86-512-6511-2371.

E-mail address: qshen@suda.edu.cn (Q. Shen).

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chlorides in 1995 [11b]. However, little is known about the lanthanide derivatives with such ligand. We found $[N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2LnCl(THF)$ are one of the ideal precursors for preparation lanthanide methyl complexes as illustrated in the following equation:

$$\begin{aligned}&[N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2LnCl(THF)\\ &\quad + 2CH_3Li \xrightarrow{-78^\circ C} [N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)\\ &\quad \times (SiMe_3)]_2LnCH_3(\mu\text{-}CH_3)Li(THF)_3(PhCH_3)\\ &\quad + LiCl\end{aligned}$$

where Ln = Nd (**1**), Yb (**2**)

The formulae of **1** and **2** are confirmed by elemental analyses and IR spectra. Further X-ray structural determination of **2** reveals it to be an anionic complex possessing a methyl bridge as shown in above equation and Fig. 1.

Both complexes are extremely air- and moisture-sensitive, they have good solubility in toluene, tetrahydrofuran (THF), and insoluble in hexane.

Crystals of **2** suitable for X-ray diffraction were grown from toluene. An ORTEP diagram depicting the molecular structure of **2** is shown in Fig. 1. Detailed crystal and structural refinement data are listed in Table 1, selected bond lengths and angles are given in Table 2. As shown in Fig. 1, complex **2** consists of two moieties, i.e., $[N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2Yb(CH_3)_2$ and $Li(THF)_3$, they are bonded through a single μ-methyl, while the other methyl group is bonded to ytterbium ion independently. In this case, the molecular structure of **2** is quite unique, different from those anionic lanthanide methyl complexes, in which lanthanide metal and lithium would rather be bonded through two μ-methyl groups [1a]. The coordination geometry of ytterbium ion is best described as a distorted pseudo-tetrahedron with two nitrogen atoms of arylamido ligands and two carbon atoms of methyl groups forming the four vertexes, ytterbium ion lying in the heart of the tetrahedron. The lithium ion also adopts a distorted pseudo-tetrahedron with three oxygen atoms of THF molecules and one carbon atom of methyl group forming the four vertexes.

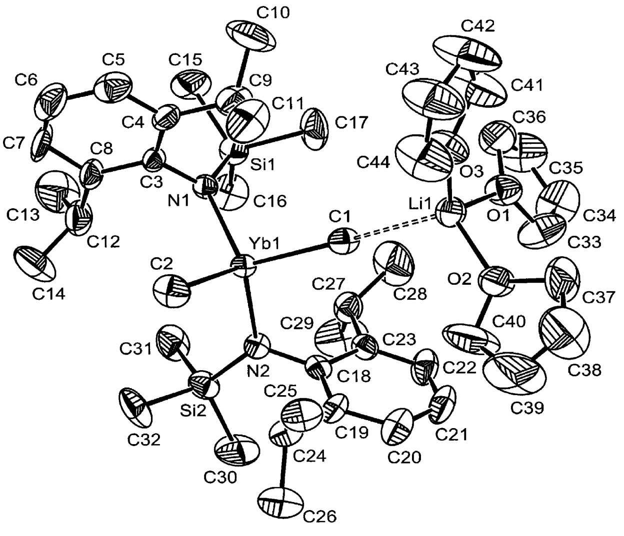

Fig. 1. The molecular structure of **2**.

The bond lengths of Yb(1)–N(1) and Yb(1)–N(2) are nearly equal (2.224(6) and 2.227(6) Å, respectively), which are identical to those in $[N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2NdCl(THF)$ (2.276(2) and 2.264(2) Å) [11b], after the subtraction of the difference in ionic radii. However, the Yb–N bond lengths in **2** are apparently longer than those in $[(Me_3Si)_2N]_2Yb(\mu\text{-}Cl)(THF)]_2$ (2.174(5) and 2.198(5) Å), although the latter is five-coordinate species

Table 1
Experimental data for X-ray diffraction study of **2**

	2
Empirical formula	$C_{51}H_{90}LiN_2O_3Si_2Yb$
Formula weight	1015.41
Temperature (K)	298.1 K
Wavelength (Å)	0.71070
Crystal system	Orthorhombic
Space group	*Pbca* (#61)
Unit cell dimensions	
a (Å)	38.207(3)
b (Å)	16.5249(14)
c (Å)	18.3477(16)
V (Å^3)	11 584.0(17)
Z	8
D_{calc} (g cm^{-3})	1.164
Absorption coefficient (mm^{-1})	1.692
F(0 0 0)	4280
Theta range for data collection (°)	3.00–26.01
Reflections collected	97 664
Independent reflections	11 143
Data/restraints/parameters	11 143/19/556
Final *R* [$I > 2\sigma(I)$]	0.0876
R_w	0.1468
Goodness-of-fit on F^2	1.425
Largest difference peak and hole (e A^{-3})	1.894, −1.401

Table 2
Selected bond lengths (Å) and angles (°) for **2**

Bond lengths			
Yb(1)–N(2)	2.224(6)	O(1)–Li(1)	1.909(16)
Yb(1)–N(1)	2.227(6)	O(2)–Li(1)	1.917(16)
Yb(1)–C(2)	2.349(8)	O(3)–Li(1)	1.948(16)
Yb(1)–C(1)	2.382(7)	N(1)–C(3)	1.439(9)
Si(1)–N(1)	1.718(6)	N(2)–C(18)	1.421(10)
Si(2)–N(2)	1.723(6)	C(1)–Li(1)	2.424(14)
Bond angles			
N(2)–Yb(1)–N(1)	121.3(2)	Si(1)–N(1)–Yb(1)	129.8(4)
N(2)–Yb(1)–C(2)	106.1(3)	C(18)–N(2)–Yb(1)	110.1(4)
N(1)–Yb(1)–C(2)	106.7(3)	Si(2)–N(2)–Yb(1)	129.1(4)
N(2)–Yb(1)–C(1)	107.7(3)	O(1)–Li(1)–O(2)	107.4(8)
N(1)–Yb(1)–C(1)	108.0(3)	O(1)–Li(1)–O(3)	112.1(8)
C(2)–Yb(1)–C(1)	106.1(3)	O(2)–Li(1)–O(3)	103.7(7)
C(3)–N(1)–Yb(1)	112.7(4)	Yb(1)–C(1)–Li(1)	178.8(5)

[11d]. This is consistent with the difference in steric bulkiness between $N(C_6H_3{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)$ and $(Me_3Si)_2N$. Due to the different bonding modes, the bond length of Yb(1)–C(1) is 0.033 Å longer than that of Yb(1)–C(2) (2.349(8) Å), however, they both are shorter than those found in $[(SiMe_3)_2NC(N\,i\text{-}Pr)_2]_2Yb(\mu\text{-}Me)_2Li(TMEDA)$ (2.472(8) and 2.479(8) Å) [9e].

The bond angle of N(1)–Yb(1)–N(2) is 121.3(2)°, which is 4.5° smaller than that of $[N(C_6H_3{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2NdCl(THF)$ (116.60(7)°) [11b], and 5.7° larger than that of $[(Me_3Si)_2N]_2Yb(\mu\text{-}Cl)(THF)]_2$ (115.6(2)°) [11d]. The bond angle of C(1)–Yb(1)–C(2) (106.1(3)°) is much greater than that of $[(SiMe_3)_2NC(N\,i\text{-}Pr)_2]_2Yb(\mu\text{-}Me)_2Li(TMEDA)$ (91.6(3)°) [9e] for in the latter complex the existence of two μ-methyl bridges to constrain the geometry. Due to the steric bulk of two arylamido ligands and three coordinated THF molecules, the bond angle of Yb(1)–C(1)–Li(1) (178.8(5)°) is close to straight angle, and this deposition reduces the steric repulsion between the two moieties.

As can be seen in Fig. 1, the orientation of the $SiMe_3$ groups in the two ligand frameworks is approximately opposite, and this deposition reduces the steric congestion around the coordination sphere of the ytterbium ion.

2.2. The polymerization of methyl methacrylate

Both **1** and **2** are able to initiate the polymerization of MMA. The polymerization proceeds fluently at the temperatures below 0 °C to give the PMMA with high molecular weight ($M_n > 10^4$) and relatively narrow molecular weight distribution ($M_w/M_n < 2$) (see Table 3). The activity of the present polymerization system can be comparable with that of $[(SiMe_3)_2NC(N\,i\text{-}Pr)_2]_2Nd(\mu\text{-}Me)_2Li(TMEDA)$ [9e]. The effect of the temperature on the polymerization can be observed obviously. This may be the result of higher polymerization temperature favoring the nucleophilic attack of methyl group at the carbonyl-carbon atom of MMA.

The tacticity of the resultant PMMA was determined with reference to the reported triad [12]. These polymerization reactions give syndiotactic-rich PMMA. The stereoregularity of the resulting polymer varies greatly depending on the polymerization temperature. Table 3 indicates that the syndiotacticity increased from 46.5 to 65.3% when the polymerization temperature was decreased from 10 to −78 °C. This is in line with the general trend observed in MMA polymerizations. The somewhat complicated triad distributions of the PMMAs can be attributed to the presence of multiple active sites in the catalysts. However, comparison with the extremely high sydiotacticity of the resultant polymers using lanthanocene complexes as catalysts [13], the present system is not well controlled in stereo-specificity.

3. Conclusion

In summary, we have successfully synthesized a series of new bis(arylamido)lanthanide methyl complexes. Moreover, we found theses complexes can be served as single-component catalysts for the polymerization of MMA to give syndiotactic-rich and high molecular weight polymers ($M_n > 10^4$) with relatively narrow molecular weight distributions ($M_w/M_n < 2$).

4. Experimental

All manipulations were performed under pure argon with rigorous exclusion of air and moisture using standard Schlenk techniques. Solvents were distilled from Na/benzophenone ketyl prior to use. Anhydrous $LnCl_3$ [14], $[N(C_6H_3{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2LnCl(THF)$ [11b] were prepared according to the literature procedures. *N*,*N*′-Diisopropylaniline, $SiMe_3Cl$ were pur-

Table 3

Polymerization of MMA initiated by $[N(C_6H_3{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2LnCH_3(\mu\text{-}CH_3)Li(THF)_3\cdot PhCH_3$ (Ln = Nd (**1**), Yb (**2**))

Entry	Initiator	[M]/[*I*]	T_p (°C)	Yield [a] (%)	M_n ($\times 10^{-4}$) [b]	M_w/M_n [b]	*mm*	*mr*	*rr* [c]
1	**1**	235	−78	98.9	5.65	1.61			
2	**1**	235	0	70.3	2.24	1.92			
3	**2**	235	−78	95.1	1.63	1.58	15.8	18.9	65.3
4	**2**	235	0	76.8	2.31	1.86	22.1	23.1	54.8
5	**2**	235	10	66.2	1.90	1.93	19.0	34.5	46.5
6	**2**	500	0	53.4	2.95	1.80			
7	**2**	700	0	36.7	2.47	1.85			

Polymerization conditions: in toluene; 3 h, solvent/monomer = 2 v/v.

[a] Yield = weight of polymer obtained/weight of monomer used.

[b] Measured by GPC calibrated with standard polystyrene samples.

[c] Measured by ^{13}C-NMR in CD_3Cl.

chased from Acros and purified by distillation. MMA, a commercial reagent (chemically pure) of Beijing Chemical Factory, was distilled over fresh calcium hydride (CaH_2) powder and stored over molecular sieves 4 Å under argon. Melting points were determined in argon-filled capillary tubes and are uncorrected. Lanthanide metal analyses were carried out by complexometric titration. The content of lithium was determined on a Hitachi 180–80 polarized Zeeman atomic absorption spectrophotometer. Carbon, hydrogen, and nitrogen analyses were performed by direct combustion on a Carlo–Erba EA-1110 instrument. The IR spectra were recorded with a Nicolet Magna-IR 550 spectrometer. ^{1}H-NMR and ^{13}C-NMR spectra were obtained using a Unity Inova-400 spectrometer. Molecular weight and molecular weight distributions were determined against polystyrene standard by gel permeation chromatography (GPC) with a Waters 1515 apparatus equipped with a set of Waters Styragel HR columns (HR-1, HR-2 and HR-4 columns, effective molecular weight range, 100–5000, 500–20 000, and 5000–500 000, respectively). THF was used as an eluent at a flow rate of 1.0 ml min^{-1} at 30 °C.

4.1. Synthesis of $[N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2NdCH_3(\mu\text{-}CH_3)Li(THF)_3\cdot PhCH_3$ (**1**)

A Schlenk flask was charged with $[N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2NdCl(THF)$ (3.74 g, 5.0 mmol) and 100 ml of toluene. The solution was cooled to −78 °C, and MeLi (5.6 ml, 10.0 mmol) was added by syringe. The reaction mixture was kept at −78 °C for an hour, then slowly warmed to room temperature and stirred overnight. After removal of volatiles under vacuum, the yellow residue was extracted with toluene and LiCl was removed by centrifugation. The extracts were concentrated to 10 ml and cooled at −15 °C overnight to give **1** as blue–purple cubic crystals. Yield, 3.4 g (3.4 mmol, 68%). M.p. 168–171 °C. Anal. Calc. for $C_{51}H_{90}LiN_2O_3Si_2Nd$: C, 62.08; H, 9.21; N, 2.84; Nd, 14.62; Li, 0.70. Found: C, 61.85; H, 9.16; N, 2.81; Nd, 14.39; Li, 0.68. IR (KBr pellet, cm^{-1}): 2962 (s), 2870 (s), 1587 (m), 1462 (s), 1381 (m), 1326 (s), 1250 (s), 1180 (m), 1103 (m), 1045 (m), 907 (s), 837 (s), 779 (s), 744 (m), 667 (m), 617 (m), 524 (m), 435 (m).

4.2. Synthesis of $[N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2YbCH_3(\mu\text{-}CH_3)Li(THF)_3\cdot PhCH_3$ (**2**)

Following the procedure similar to the synthesis of **1**, using 5.0 mmol of $[N(C_6H_3\text{-}i\text{-}Pr_2\text{-}2,6)(SiMe_3)]_2YbCl(THF)$, 10.0 mmol of MeLi, and 150 ml of toluene following by crystallization from toluene yielded orange cubic crystals of **2** (3.7 g, 72%). M.p. 156–159 °C. Anal. Calc. for $C_{51}H_{90}LiN_2O_3Si_2Yb$: C, 60.32; H, 8.95; N, 2.76; Yb, 17.04; Li, 0.68. Found: C, 59.93; H, 8.71; N, 2.54; Yb, 16.95; Li, 0.71. IR (KBr pellet, cm^{-1}): 2963 (s), 2870 (s), 1586 (m), 1462 (s), 1381 (m), 1327 (s), 1250 (s), 1181 (m), 1103 (m), 1046 (m), 906 (s), 837 (s), 779 (s), 667 (m), 617 (m), 524 (m), 436 (m).

4.3. A typical procedure for polymerization reactions

The procedures for the polymerization of MMA are the same (Table 3), and a typical polymerization reaction is given below (Entry 3, Table 3). A 50-ml Schlenk flask equipped with a magnetic stir bar was charged with a 40.4 mg of **2** and 2 ml of toluene. Then, the flask was removed to a dry ice bath (−78 °C) and added 1 ml of MMA using rubber septum and syringe. The contents of the flask were vigorously stirred for 3 h at −78 °C. The reaction mixture was quenched by the addition of 1 M HCl solution and then poured into a cold petroleum ether to precipitate the polymer, which was dried under vacuum and weighed.

4.4. X-ray structural determination of **2**

A suitable crystal was sealed in a thin-walled glass capillary for X-ray structural analysis. Diffraction data were collected on a Rigaku Mercury CCD area detector using ϕ and ω scans. The structures were solved by direct methods and refined by full-matrix least-squares procedures based on $|F|^2$. All non-hydrogen atoms were refined with anisotropic displacement coefficients. Hydrogen atoms were treated as idealized contributions. The structures were solved and refined using SHELXS-97 and SHELXL-97 programs, respectively.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 207200 for complex **2**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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