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Synthesis, characterization of homoleptic guanidino lanthanide complexes and their catalytic activity for the ring-opening polymerization of ε-caprolactone

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Abstract

A series of homoleptic lanthanide guanidinate $(guan)_3Ln\cdot((C_2H_5)_2O)_n$ (Ln = Yb, $n = 1$ guan = $(CyN)_2CN^iPr_2$, (**1**); Ln = Nd, $n = 0$, guan = $(CyN)_2CN^iPr_2$, (**2**); $(^iPrN)_2CN^iPr_2$, (**3**); $(^iPrN)_2CN(CH_2)_5$, (**4**)); (^{i}Pr = isopropyl, Cy = Cyclohexyl) were synthesized by the reaction of THF solution of lithium guanidinate with anhydrous lanthanide trichlorides in THF in 3:1 molar ratio. The molecular structures of **2** and **3** were determined to be monomeric in solid state with a six coordinate lanthanide metal ligated by six nitrogens of three guanidinate groups. All the complexes exhibited extremely high activity for the ring-opening polymerization of ε-caprolactone and the polymerization gave the polymers with high molecular weights. The different substituents at guanidino ligands have great effect on the catalytic activity. The mechanism of the polymerization was presented.

Keywords: Organolanthanides; Guanidino ligands; Polymerization; ε-Caprolactone; Crystal structure

1. Introduction

As an alterative to cyclopentadienyl-based ligands, guanidinate anions, $[(RN)_2CNR_2]^-$, for their steric bulk and electronic properties can be facilely modified through adjusting the organic substituents on the nitrogen atoms, have attracted considerable attention in the organometallic chemistry of main and transition metals [1]. However, this ligand has not been used in lanthanide chemistry until the first series of guanidino complexes including alkyl and amido lanthanide complexes were reported in 1998 [2]. Then lanthanum monoguanidino aryloxides [3] and yttrium guanidino complexes [4] were synthesized and the catalytic behaviors of the former were demonstrated. Very recently, bisguanidino lanthanide methyl complexes were found to be the effective initiators for the polymerization of nonpolar monomer styrene in our laboratory [5], which is quite difficult to polymerize with corresponding lanthanocene complexes. Both biguanidino lanthanide methyl complexes [6] and diisopropylamido complexes [7] showed high activities for the polymerizations of ε-caprolactone and MMA. Considering the fact that the resulting polymers in the polymerization of ε-caprolactone with bisguanidinate lanthanide methyl complexes or diisopropylamido complexes had broader molecular weight distribution than those obtained with the corresponding lanthanocene complexes [8,9]. One of the reasons for it might be the polyactive species existed in the system, that is to say that besides the Ln–CH_3 or Ln–N bond, the bond of metal to guanidinate might be also active. In order to confirm it and to understand the chemistry of the homoleptic triguanidino lanthanide, in this paper we first prepared a series of homoleptic guanidino lanthanide complexes via metathesis reaction of anhydrous $LnCl_3$ with corresponding lithium

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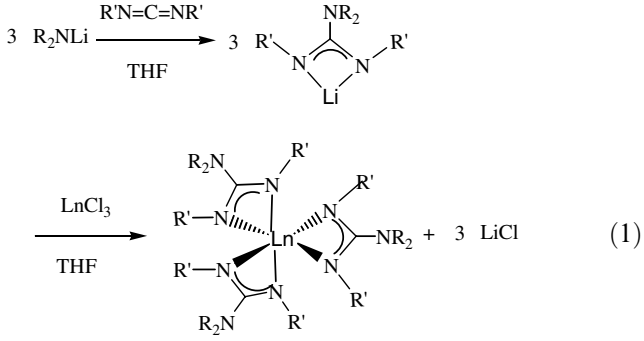
guanidinate, and tested their catalytic behavior for ε-caprolactone polymerization. It was first found that these new homoleptic complexes really showed very high activity for the ring-opening polymerization of ε-caprolactone. Here we would like to report the results.

2. Results and discussion

2.1. Synthesis and characterization of the guanidino lanthanide complexes

In order to synthesize the homoleptic lanthanide guanidinates with different steric bulk, various amines, carbodiimides and rare earth metals were selected.

The title complexes were prepared by the metathesis reaction. The fresh solution of lithium guanidinate in THF, which were prepared from *N*,*N*′-dicyclohexylcarbodiimide (or *N*,*N*′-diisopropylcarbodiimide) with an equivalent molar amount of lithium amide, was added into a slurry of $LnCl_3$ in 3:1 molar ratio. After workup the expected air- and moisture-sensitive complexes were isolated. As shown in Eq. (1), the complexes **1**, **2**, **3** and **4** are $[(CyN)_2CN^iPr_2]_3Yb$ (**1**), $[(CyN)_2CN^iPr_2]_3Nd$ (**2**), $[(^iPrN)_2CN^iPr_2]_3Nd$ (**3**) and $[(^iPrN)_2CN(CH_2)_5]_3Nd$ (**4**) (^iPr = isopropyl, Cy = Cyclohexyl), respectively.



R = ^{i}Pr, R' = Cy, Ln = Yb(**1**), Nd(**2**)

R = ^{i}Pr, R '= iPr, L n= Nd (**3**)

R = 1/2$(CH_2)_5$, R'= ^{i}Pr, Ln = Nd (**4**)

They have good solubility in THF, diethyl ether and even toluene. The formulation of these homoleptic guanidino lanthanide species is supported by elemental analysis, ^{1}H NMR and IR spectroscopy. In the IR spectra, there are strong absorptions of C=N stretch in the range of 1600–1636 cm^{-1} for each of the lanthanide complexes, which indicates the existence of the delocalized double bond of the N–C–N linkage in all these complexes. X-ray structure analysis confirms further the structural feature.

The molecular structures of complexes **2** and **3** are presented in Figs. 1 and 2, respectively. Tables 1 and 2 list the selected bond distances and angles of **2** and **3**. Table 3 gives the details of their crystallographic data.

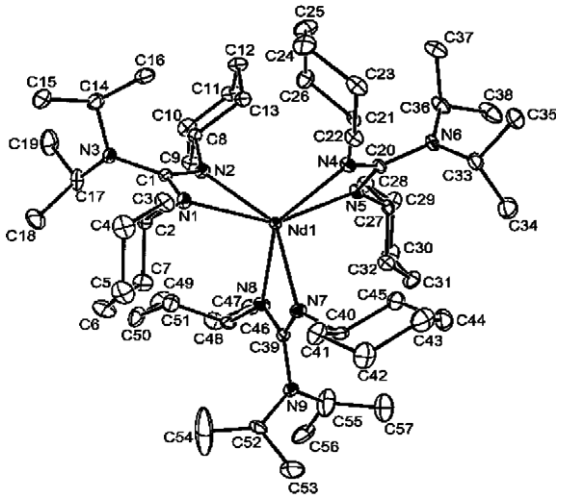


Fig. 1. The Molecular structure of complex **2**.

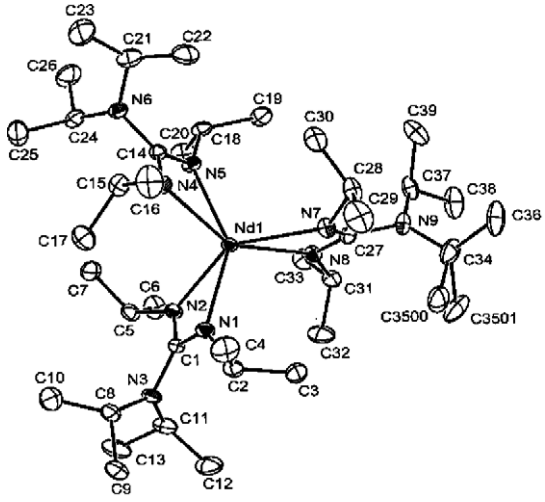


Fig. 2. The Molecular structure of complex **3**.

Table 1
Selected bond distances (Å) and angles (°) for **2**

Bond distance			
Nd–N1	2.465(4)	Nd–N2	2.447(4)
Nd–N4	2.440(4)	Nd–N5	2.479(3)
Nd–N7	2.472(4)	Nd–N8	2.449(3)
N1–C1	1.327(6)	N2–C1	1.339(6)
N3–C1	1.437(6)	N4–C20	1.351(6)
N5–C20	1.334(6)	N6–C20	1.412(6)
N7–C39	1.342(6)	N8–C39	1.329(6)
N9–C39	1.419(6)		
Bond angles			
N1–Nd–N2	54.3(1)	N4–Nd–N5	54.6(1)
N7–Nd–N8	54.3(1)	C1–N1–Nd	95.3(3)
C1–N2–Nd	95.8(3)	C20–N4–Nd	96.0(3)
C20–N5–Nd	94.7(3)	C39–N7–Nd	94.9(3)
C39–N8–Nd	96.3(3)	N1–C1–N2	114.5(4)
N4–C20–N5	114.4(4)	N7–C39–N8	114.3(4)

Table 2
Selected bond distances (Å) and angles (°) for **3**

Bond distance			
Nd–N1	2.459(3)	Nd–N2	2.462(3)
Nd–N4	2.496(3)	Nd–N5	2.452(2)
Nd–N7	2.473(3)	Nd–N8	2.444(3)
N1–C1	1.344(4)	N2–C1	1.334(4)
N3–C1	1.412(5)	N4–C14	1.335(4)
N5–C14	1.328(4)	N6–C14	1.432(3)
N7–C27	1.336(4)	N8–C27	1.329(4)
N9–C27	1.416(5)		
Bond angles			
N1–Nd–N2	54.68(8)	N4–Nd–N5	53.86(9)
N7–Nd–N8	54.42(8)	C1–N1–Nd	95.0(2)
C1–N2–Nd	95.1(2)	C14–N4–Nd	94.5(2)
C14–N5–Nd	96.7(2)	C27–N7–Nd	94.2(2)
C27–N8–Nd	95.7(2)	N1–C1–N2	115.2(3)
N4–C14–N5	114.6(2)	N7–C27–N8	115.0(3)

Table 3
Details of the crystallographic data of **2** and **3**

	2	**3**
Empirical formula	$C_{57}H_{108}N_9Nd$	$C_{39}H_{84}N_9Nd$
Formula weight	1063.78	823.39
Temperature (K)	193.1	193.1
Wavelength (Å)	0.7107	0.7107
Size (mm)	$0.20 \times 0.28 \times 0.18$	$0.60 \times 0.60 \times 0.40$
Crystal system	Orthorhombic	Monoclinic
Space group	$P2_12_12_1$(#19)	$P2_1/c$ (#14)
a (Å)	12.9867(9)	13.3636(11)
b (Å)	15.0535(11)	20.062(1)
c (Å)	30.775(2)	18.773(1)
α (°)	90.00	90.00
β (°)	90.00	109.666(3)
γ (°)	90.00	90.00
V (Å^3)	6016.3(8)	4739.6(6)
Z	4	4
D_{calc} (g cm^{-3})	1.174	1.154
Absorption coefficient (mm^{-1})	0.904	1.128
F(0 0 0)	2292.00	1764.00
Theta range for collection (°)	3.0–27.5	3.0–27.5
Reflection collected	61308	50958
Independent reflections	13765 ($R_{int} = 0.095$)	10817 ($R_{int} = 0.025$)
Variables	714	531
$R[I > 3\sigma(I)]$	0.0410	0.032
wR	0.0810	0.099
Goodness-of-fit on F^2	0.904	1.042

Both complexes are monomeric in solid state and have similar structure, but space group of them is different (**2** in orthorhombic, **3** in monoclinic). Each neodymium ion is ligated by three guanidino moieties and the geometry of the center metal is best described as a trigonal planar with each chelating bidentate guanidino ligand to occupy one coordination vertex. The center metal ion and three center carbon atoms in guanidino moiety are almost in a plane (mean deviation from plane is 0.0660 Å in **2** and 0.0490 Å in **3**, respectively). The Nd–N bond lengths for each NdN_2C moieties differ by only approximately 0.03 Å and are almost as same as those in $[(SiMe_3)_2NC(N^iPr)_2]_2Nd(\mu\text{-}Me)_2Li(TMEDA)$ (mean value 2.49 Å), those in $[(SiMe_3)_2NC(N^iPr)_2]_2YN^iPr_2$ (mean value 2.38 Å) and those in $[MeC(NCy)_2]_3Yb$ and $[PhC(NCy)_2]_3Yb$ (mean value 2.33 Å), if the difference in ion radii are considered. The distances of two Ln–N bonds and two C–N bonds (average value 1.33 Å) [10] in each guanidinate moieties are nearly equal, which reflected the delocalization of the π bond in the N–C–N unit. The angles of these N–C–N bonds are almost equal with the same value (114.4° in **2**, 115.0° in **3**). The N–Nd–N angle in each NdN_2C moieties is about 54.5° in **2**, 54.2° in **3**, which is comparable with the value in guanidino lanthanide methyl complexes (54.0°) [5], but is lower than those in guanidino lanthanide diispropylamido complexes (56.7°) [7].

2.2. The ring-opening polymerization of ε-caprolactone

All the guanidino lanthanide complexes as an initiator showed extremely high activity in the ring-opening polymerizations of ε-caprolactone. For example, the polymerization gives 100% conversion in the case of $[M]/[I] = 2000$ at 35 °C for 5 min (Table 4, run 5). The conversion still reaches as high as 80%, even the amount of $[M]/[I]$ increased to 4000 under the conditions used.

The resulting polymers have high molecular weights and relatively broad molecular weight distributions (Mw/Mn = 1.87–2.51). No correlation could be found between the calculated Mn and the measured one by GPC under the different conditions. The rather high polydispersity might be caused from the fact that all the three bonds of Ln to guanidinos are active spaces, running with different speeds. The result also gives the explanation for why the system with guanidino lanthanide methyl or diisopropylamido complex as the initiator gives the polymer with higher polydispersities than that with corresponding lanthanocene complex.

The catalytic activity of homoleptic guanidino lanthanide complexes is lower than those of the guanidino lanthanide methyl or diisopropylamido complexes. It might be caused from the difference of the rates of migratory insertion step among the three initiating systems. In the comparison of the present results with those obtained by homoleptic amidino lanthanide complexes, the activity of guanidino complexes is also lower. For example, using complex **1** as the initiator at $[M]/[I] = 800$ only 22% of the yield was obtained (Table 4, run 2), while 100%, for the case of $[PhC(NCy)_2]_3Yb$ (**5**) under the same conditions (Table 4, run 9). This might be because guanidino group is bulkier than amidino group and the electronic effect is also different between the two ligands.

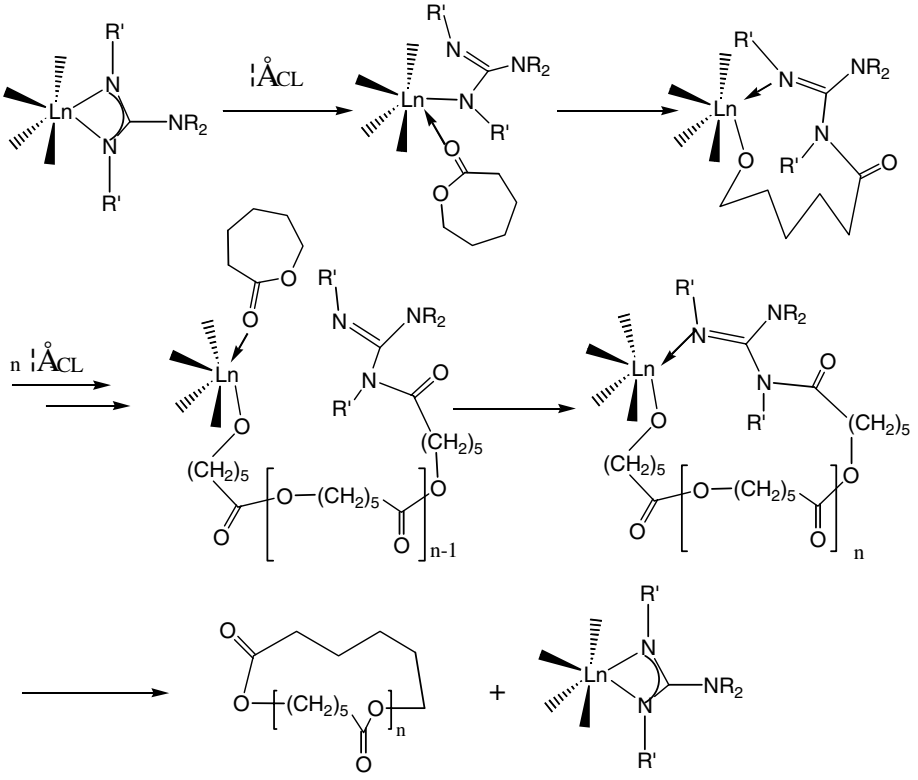
Table 4
The ring-opening polymerization of ε-caprolactone with homoleptic guanidino lanthanide complexes as the initiator[a]

Run	Initiator	$[M]/[I]$	Time (min)	Temp (°C)	Yield (%)[b]	Mn × 10^{-4}	Mn/Mw[c]
1	1	500	20	35	95	33.68	1.87
2	1	800	20	35	22	13.80	1.35
3	2	1000	5	35	100	15.55	2.19
4	2	2000	5	35	81	45.56	2.43
5	3	2000	5	35	100	56.02	2.51
6	3	2000	5	10	97	65.71	2.22
7	3	2000	5	0	77	54.11	2.21
8	4	2000	5	35	74	44.87	2.33
9	5	800	20	35	100	–	–

[a] Polymerization condition: in toluene, solvent/monomer = 5 (V/V).
[b] Yield: weight of polymer obtained/weight of monomer used.
[c] Measured by GPC calibrated with standard polystyrene samples.

The structure of guanidinate has great effect on the activity of the complex. The activity of the complex ligated with bulkier group is lower than that with less bulk ligand. The active order is **4** < **2** < **3** (Table 4, runs 4, 5 and 8). The central metal has an impact on the polymerization. The order of reactivity for metal Nd > Yb (Table 4, runs 2, 3) is consistent with that found for metallocene-based organolanthanide catalysts [8] and lanthanide guanidino methyl complexes [5]. Polymerization temperature also has great effect on the polymerization. At lower temperature (0 °C), the polymerization with **3** becomes slower (Table 4, run 7).

According to the above experimental results, the reaction of the guanidino-catalyzed polymerizations would be supported to be a coordination mechanism. In order to confirm it further, the oligomerization was carried out under the condition of $[M]/[I] = 8$, and terminated by isopropyl alcohol. The resulting polymer has taken to ^{1}H NMR spectrum analysis. There were no peaks of isopropyl or guanidino group observed but the peaks of polyCL in ^{1}H NMR spectrum, which means the oligomer has no end group and should be a cyclic polymer. The mechanism was similar to that with the system of lanthanides benzimidinates [11]. Postulated mechanism was described in Scheme 1. As the initial



Scheme 1. Postulated mechanism of the ring-opening polymerization of ε-caprolactone.

step of the polymerization, ε-caprolactone coordinated to the central metal, then a nucleophilic attack by one of the guandinio-nitrogen atom at carbonyl-carbon atom of the lactone, followed by acyl bond cleavage and the formation of an alkoxide complex. The cyclic polymer was formed through intramolecular attack of the Ln–O bond to the N-bonded acyl carbon atom and the catalyst was regenerated.

3. Conclusion

A series of homoleptic guanidino lanthanide complexes were prepared in good yield and the molecular structures were determined by X-ray diffractometer. These complexes showed high catalytic activity for the ring-opening polymerization of ε-caprolactone. The guanidinate anions with different substituents and the central metal both have great effect on the catalytic activity.

4. Experimental

All manipulations were performed under pure Ar with rigorous exclusion of air and moisture using standard Schlenk techniques. Solvents were distilled from Na/benzophenone ketyl prior to use. Anhydrous $LnCl_3$ were prepared according to the literature procedures. *N*,*N*′-dicyclohexylcarbodiimide and *N*,*N*′-diisopropylcarbodiimide was purchased from Aldrich and used as received without further purification. ε-Caprolactone was purchased from Acros, dried by stirring with CaH_2 for 48 h, and then distilled under reduced pressure. Melting points were determined in sealed Ar-filled capillary tubes and are uncorrected. Metal analyses were carried out by complexometric titration. Carbon, hydrogen and nitrogen analyses were performed by direct combustion on a Carlo-Erba EA = 1110 instrument. The IR spectra were recorded on a Magna-IR 550 spectrometer. Molecular weight and molecular weight distributions were determined against polystyrene standard by gel permeation chromatography (GPC) on a waters 1515 apparatus with three HR columns (HR-1, HR-2 and HR-4). THF was used as an eluent at 30 °C. ^{1}H NMR spectra were measured on a Unity Inova-400 spectrometer.

4.1. Synthesis of $[(CyN)_2CN^iPr_2]_3Yb\cdot((C_2H_5)_2O)$ (1)

A Schlenk flask was charged with $^iPr_2NH$ (2.1 ml, 15.0 mmol), THF (30 ml), and a stir bar. The solution was cooled to 0 °C, and *n*-BuLi (9.6 ml, 15.0 mmol, 1.56 M in hexane) was added. The solution was slowly warmed to room temperature and stirred for 1 h. Then to this solution was added *N*,*N*′-dicyclohexylcarbodiimide (3.15 g, 15.3 mmol in 10 ml THF) at 0 °C. The resulting solution was slowly warmed to room temperature and stirred for 1 h and then added slowly to a pale-gray slurry of $YbCl_3$ (1.42 g, 5.08 mmol) in 20 ml THF. The color of the solution immediately changed to yellow. The resulting solution was then stirred for another 24 h and evaporated to dry in vacuo. The residue was extracted with Et_2O and LiCl was removed by centrifugation. When the extracts were concentrated and cooled to −30 °C for crystallization, pale-yellow crystals were formed. Yield: 4.0 g (76%) M.p. 273–275 °C. ^{1}H NMR (C_6D_6, δ): 3.37–3.16 (m, 12H, C*H*$_2$, unique Cy-*H*, overlap), 3.05 (m, 4H, ($CH_3CH_2)_2O$), 2.13–1.28 (m, 60H, C_6H_{10}), 1.16–0.93 (m, 42H, CH(C*H*$_3$)$_2$, ($CH_3CH_2)_2O$, overlap). Anal. Calc. for $C_{61}H_{118}N_9YbO$: C, 62.80; H, 10.19; N, 10.80; Yb, 14.83. Found: C, 62.47; H, 10.01; N, 10.62; Yb, 14.56; IR (KBr pellet, cm^{-1}): 3175 (m), 2932 (s), 2854 (s), 1601 (s), 1554 (s), 1450 (s), 1338 (m), 1246 (m), 1149 (m), 1099 (m), 895(m).

4.2. Synthesis of $[(CyN)_2CN^iPr_2]_3Nd$ (2)

Following the procedure similar to the synthesis of **1**, using 15.0 mmol of $[(CyN)_2CN^iPr_2]Li$, 1.28 g of $NdCl_3$ (5.11 mmol), and 60 ml THF following by crystallization from diethyl ether yielded pale yellow crystals. Yield: 4.3 g (79%). M.p. 251–253 °C. ^{1}H NMR (C_6D_6, δ): 3.61–3.16 (m, 12H, C*H*$_2$, unique Cy-*H*, overlap), 1.91–1.16 (m, 60H, C_6H_{10}), 1.14–0.91 (m, 36H, CH(C*H*$_3$)$_2$). Anal. Calc. for $C_{57}H_{108}N_9Nd$: C, 62.32; H, 9.84; N, 11.48; Nd, 15.85. Found: C, 62.47; H, 10.01; N, 11.62; Nd, 14.56; IR (KBr pellet, cm^{-1}): 3182 (m), 2928 (s), 2854 (s), 1631 (s), 1554 (s), 1450 (s), 1373 (m), 1207 (m), 1141 (m), 1103 (s), 1003 (m), 892 (s), 860 (m), 744 (m).

4.3. Synthesis of $[(^iPrN)_2CN^iPr_2]_3Nd$ (3)

Following the procedure similar to the synthesis of **1**, using 15.0 mmol of $[(^iPrN)_2CN^iPr_2]Li$, 1.30 g of $NdCl_3$ (5.08 mmol), and 60 ml THF following by crystallization from diethyl ether yielded blue-purple crystals. Yield: 3.3 g (82%). M.p. 206–208 °C. ^{1}H NMR (C_6D_6, δ): 2.98–3.66 (m, 12H, C*H*$(CH_3)_2$), 0.94–1.35 (m, 72H, CH(C*H*$_3$)$_2$). Anal. Calc. for $C_{39}H_{84}N_9Nd$: C, 56.89; H, 10.28; N, 15.31; Nd, 17.52. Found: C, 57.31; H, 10.24; N, 15.51; Nd, 17.2. IR (KBr pellet, cm^{-1}): 2967 (s), 2932 (s), 2870 (s), 1632 (s), 1470 (s), 1416 (s), 1377 (s), 1327 (s), 1162 (m), 1123 (m), 1034 (m), 939 (m), 860 (m), 698 (m), 575 (m).

4.4. Synthesis of $[(^iPrN)_2CN(CH_2)_5]_3Nd$ (4)

Following the procedure similar to the synthesis of **1**, using 15.0 mmol of $[(^iPrN)_2C(CH_2)_5]Li$, 1.23 g of $NdCl_3$ (4.90 mmol), and 60 ml THF following by crystallization

from diethyl ether yielded blue-purple crystals. Yield: 3.8 g (72%). M.p. 208–210 °C. ^{1}H NMR (C_6D_6, δ): 3.04–3.76 (m, 18H, C*H*$(CH_3)_2$, C*H*$_2$–N–C*H*$_2$, overlap); 1.155–1.39 (m, 18H, (C*H*$_2$)$_3$); 0.95 (s, 36H, CH(C*H*$_3$)$_2$). Anal. Calc. for $C_{36}H_{72}N_9Nd$: C, 55.79; H, 9.29; N, 16.27; Nd, 18.62. Found: C, 55.61; H, 9.79 N, 15.87; Nd, 18.50. IR (KBr pellet, cm^{-1}): 2967 (s), 2932 (s), 2855 (s), 1636 (s), 1454 (s), 1385 (s), 1277 (s), 1169 (s), 1127 (s), 1034 (s), 995 (s), 926 (s), 857 (s), 729 (s), 667 (s), 617 (s), 555 (s).

4.5. A typical procedure for polymerization reactions

The procedures for the polymerization of ε-caprolactone are the same (Table 4). And a typical polymerization reaction is given below (Entry 1, Table). A 50 ml Schlenk flask equipped with a magnetic stir bar was charged with a solution of 0.5 ml ε-caprolactone in 3.5 ml toluene. To this solution was added 1.5 ml solution of 1 in toluene (1.0×10^{-2} M) using rubber septum and syringe. The contents of the flask were then vigorous stirred for 20 min at 35 °C. The reaction mixture was quenched by the addition of alcohol then poured into a cold alcohol to precipitate the polymer, which was dried under vaccum and weighed.

4.6. X-ray structural determination of **2** and **3**

A suitable crystal was mounted in a thin-walled glass capillary for X-ray structural analysis. Diffraction data were collected on a Bruker SMART CCD area detector using phi and omega scans. The structures were solved by Patterson Methods (DIRDIF99 PATTY) 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 Crystal Structure programs.

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Cryatallographic Data Center, CCDC No. 223138 for complex **2** and No. 223139 for complex **3**. Copies of this information may be obtained free of charge from 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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