

Syntheses of $\{(\text{MeC}_5\text{H}_4)_2\text{Ln}[\text{O}=\overline{\text{C}}=\overline{\text{N}}(\text{CH}_2)_4\text{CH}_2]\}_2$ (Ln = Yb, Y, Er) and the X-ray crystal structure of the ytterbium complex

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Abstract

Reaction of $(\text{MeC}_5\text{H}_4)_2\text{LnCl}$ (Ln = Yb, Y, Er) with $\text{LiNCO}(\text{CH}_2)_4\text{CH}_2$ in toluene at 0°C yielded the complexes $\{(\text{MeC}_5\text{H}_4)_2\text{Ln}[\text{O}=\overline{\text{C}}=\overline{\text{N}}(\text{CH}_2)_4\text{CH}_2]\}_2$ [Ln = Yb (1), Y (2), Er (3)] in fairly good yields. The crystal structure of **1** [monoclinic, space group $P2_1/n$ ($\neq 14$); $a = 12.247(1)$, $b = 11.137(2)$, $c = 12.708(3)$ Å; $\beta = 107.36(1)^\circ$, $V = 1654.2(5)$ Å³, $Z = 2$, $D_{\text{calc}} = 1.780$ g cm⁻³] was identified by X-ray diffraction analysis. The two trivalent $(\text{MeC}_5\text{H}_4)_2\text{Yb}$ units are bridged by two deprotonated ϵ -caprolactam ligands via Yb–N (2.374(4) Å) and Yb–O* (2.277(3) Å) bonds. All the complexes exhibit good catalytic activity for the ring-opening polymerization of ϵ -caprolactone. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Synthesis; X-ray structure; Ytterbium; Yttrium; Erbium; Polymerization; ϵ -Caprolactone

1. Introduction

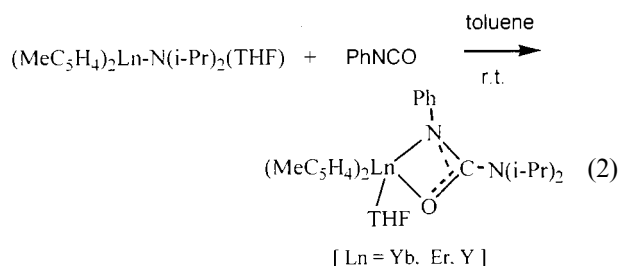
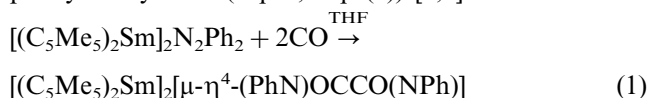
In recent years, the application of organolanthanide complexes as catalysts or precatalysts in polymer synthesis has attracted much attention [1–4]. Most of the complexes studied so far exhibit Ln–H, Ln–C, or Ln–N σ -bonds. All these complexes are very sensitive to the air and moisture.

The coordination chemistry of $[\text{RCONR}']^-$ is similar to that of carboxylate (RCO_2^-) ligands which have been known to donate four electrons to the metal and to yield stable, neutral, base-free complexes with a bidentate ($\text{O}=\overline{\text{C}}=\overline{\text{O}}$) coordination mode [5,6]. Therefore, it is supposed that the RCONR' group can be used as bidentate chelating ligand in organolanthanide complexes and such kind of the complexes should be considered to be interesting in their chemistry. However, investigations about synthesis, characterization and chemistry of the complexes are still limited [7–9]. Recently, we synthesized $\{(\text{MeC}_5\text{H}_4)_2\text{Ln}[\text{O}=\overline{\text{C}}=\overline{\text{N}}(\text{CH}_2)_4\text{CH}_2]\}_2$ (Ln = Yb, Y, Er) and found they all show good catalytic behavior for the polymerization of ϵ -caprolactone.

2. Results and discussion

2.1. Synthesis

There are only three lanthanocene complexes bearing RCONR' moiety published so far in the literature. One of them, $[(\text{C}_5\text{Me}_5)_2\text{Sm}]_2[(\text{PhN})\text{OCCO}(\text{NPh})]$, was reported by Evans et al. [8]. The complex was synthesized by the insertion of CO into corresponding amide complex $[(\text{C}_5\text{Me}_5)_2\text{Sm}]_2\text{N}_2\text{Ph}_2$ (Eq. (1)). The other two complexes were synthesized by the stoichiometric reactions of lanthanocene amides or divalent lanthanides with phenyl isocyanate (Eq. 2, Eq. (3)) [7,9].



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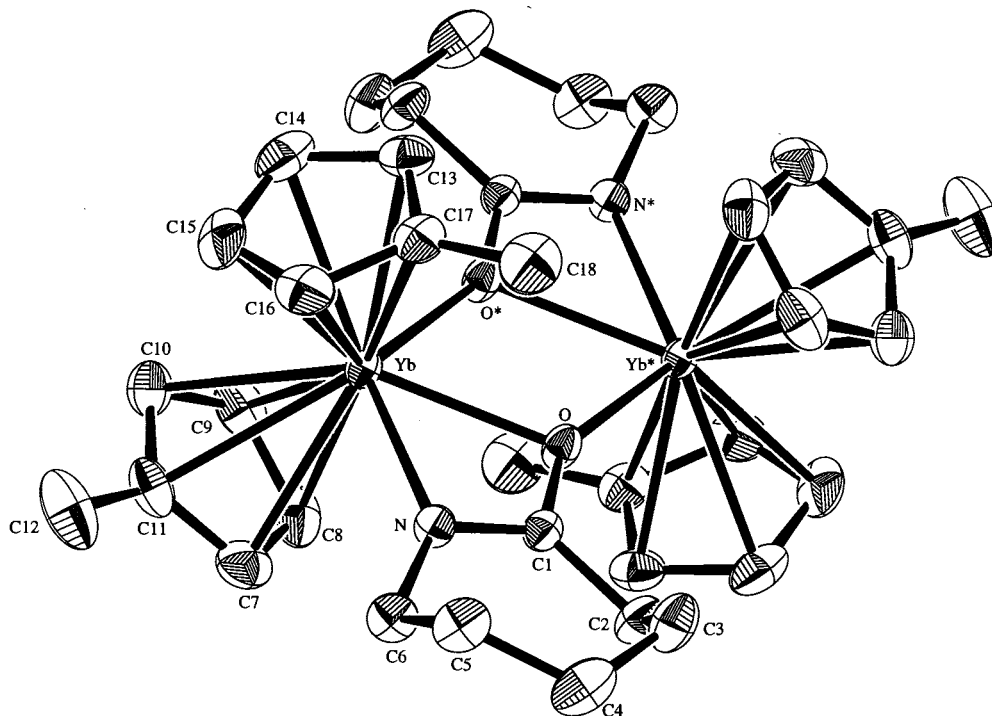
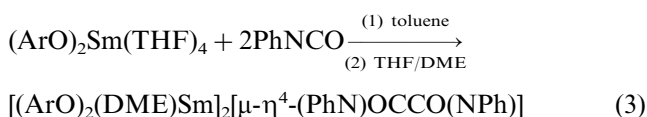
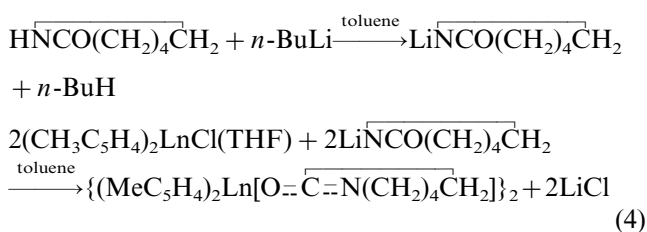


Fig. 1. Molecular structure of complex **1** with atomic numbering (hydrogen atoms have been omitted for clarity).



We considered that the complex could be synthesized straightforward by reaction of lanthanocene chloride with corresponding alkali metal salts. The expected complexes were conveniently obtained when ϵ -caprolactam reacts with one equivalent of *n*-BuLi in toluene, followed by the treatment with $(\text{MeC}_5\text{H}_4)_2\text{LnCl}$ in toluene at 0°C .



(Ln = Yb (**1**), Y (**2**), Er (**3**))

All these complexes are moderately soluble in THF and toluene, and insoluble in aliphatic hydrocarbons. They are thermally stable and melting points are around 290°C .

2.2. Characteristics of the structure of **1**

The molecular structure of **1** is shown in Fig. 1. The complex is an unsolvated binuclear complex with two unsymmetrical bridging oxygen atoms. The ytterbium

atom is coordinated by two methylcyclopentadienyl rings, two oxygen atoms and one nitrogen atom. The formal coordination number of the central metal is nine and Yb, N, O, Yb*, N*, O* atoms form a distorted octagon. Selected bond distances and angles are listed in Table 1.

The N–C1 distance (1.323(7) Å) is shorter than 1.462(7) Å for N–C6(sp³) single bond distance and longer than the value accepted for a N(sp²)=C(sp²) double bond (1.26 Å). The O–C1 distance (1.300(6) Å)

Table 1
Selected bond distances(Å) and angles(°) for complex **1**^a

Bond distances			
Yb–O	2.370(3)	Yb–O*	2.277(3)
Yb–N	2.374(4)	Yb–C(7)	2.657(6)
Yb–C(8)	2.608(7)	Yb–C(9)	2.605(6)
Yb–C(10)	2.653(5)	Yb–C(11)	2.698(5)
Yb–C(13)	2.645(6)	Yb–C(14)	2.607(6)
Yb–C(15)	2.622(6)	Yb–C(16)	2.647(7)
Yb–C(17)	2.684(6)	O–C(1)	1.300(6)
N–C(1)	1.323(7)	N–C(6)	1.462(7)
Yb–Cent(1)	2.362	Yb–Cent(2)	2.357
Angles			
O–Yb–O*	67.0(1)	O–Yb–N	55.2(1)
O*–Yb–N	122.2(1)	O–C(1)–N	113.9(4)
Yb–O–C(1)	95.7(3)	Yb–N–C(1)	94.8(3)
Yb–O–Yb	113.0(1)	Cent(1)–Yb–Cent(2)	126.79

^a Cent(1) is the centroid of the C(7)–C(11) ring; Cent(2) is the centroid of the C(13)–C(17) ring

Table 2
Polymerization of ϵ -caprolactone by complexes **1–3**^a

No.	Cat.	Time (h)	Temperature (°C)	Yield (%)	Mn $\times 10^{-4}$	Mw/Mn
1	1	3	60	91.3	7.52	2.02
2	2	3	60	89.8	10.3	2.14
3	3	3	60	76.2	3.82	1.70
4	2	24	60	96.2	6.42	2.19
5	2	2	60	57.6	7.52	1.81
6	2	2	70	74.3	4.26	2.17
7	2	2	80	100	8.99	3.29

^a Reaction condition: initiator concentration, 0.2 mol% of monomer; solvent, toluene; solvent/monomer = 10 v/v.

falls between 1.324(6) and 1.41(1) Å observed for O–C(sp³) single bonds [10,11] and 1.192 and 1.256 Å for O=C(sp²) double bonds [12]. These bond parameters indicate some electronic delocalization within the OC1N moiety. Similar structures are also found in the analogous lanthanide complexes (MeC₅H₄)₂Ln(THF)[O=CN(*i*-Pr)₂=NPh] [7], [(C₅Me₅)₂Sm]₂[μ - η^4 -(PhN)OCCO(NPh)] [8] and [(ArO)₂(DME)Sm]₂[μ - η^4 -(PhN)OCCO(NPh)] [9]. The ytterbium is out of the plane defined by the O, C1, N atoms by 0.219 Å.

The Yb–N distance, 2.374(4) Å, is intermediate between the Yb–NR₂ bonds, 2.286(7) Å for (MeC₅H₄)₂YbNPh₂(THF) [13], 2.161(5) Å for (MeC₅H₄)₂YbNC₅H₁₀(HNC₅H₁₀) [14], and Yb←:NR₃ donor bond distances, 2.446(4) Å for (MeC₅H₄)₂YbNC₅H₁₀(HNC₅H₁₀), 2.586(7) Å and 2.544(6) Å for (C₅Me₅)₂Yb(C₅H₅N)₂ [15], 2.55(3) Å for (C₅Me₅)₂Yb(NH₃)(THF) [16] even if subtraction of the differences between eight- and nine-coordinate Yb³⁺, eight-coordinate Yb²⁺ and nine-coordinate Yb³⁺ in radii is made [17]. The Yb–O* and Yb*–O distances, 2.277(3) Å, are comparable to those in the related bimetallic ytterbium alkoxides (2.195–2.22 Å) [10], but much shorter than Yb←:OR₂ donor bond distances in (*t*-BuC₅H₄)₂YbCl(THF), 2.333(6) Å [18], (C₅Me₅)₂YbCl(THF), 2.362 Å [19], (MeC₅H₄)₂YbNPh₂(THF), 2.329(6) Å [13] after the corrections are made from the difference (0.057 Å) in radii for eight- and nine-coordinate Yb³⁺ [17]. It is obvious that the two [O=C–N(CH₂)₄CH₂][–] anions bond to two ytterbium atoms forming a binuclear molecule. The coordinate feature is very similar to that in the complex [(C₅Me₅)₂Sm]₂[μ - η^4 -(PhN)OCCO(NPh)](**4**) [8]. In complex **4**, the [(PhN)OCCO(NPh)]^{2–} dianion attaches to the samarium atoms and the distances of Sm–N and Sm–O are 2.49(1) and 2.30(1) Å, respectively. The Yb–O* or Yb*–O bond distance in complex **1** is similar to the Sm–O bond distance in **4**. However, the distance of Yb–N in complex **1** is much shorter than that of Sm–N in complex **4** when corrections are made from the difference (0.037 Å) in the ionic radii for nine-coordinate Yb³⁺ and eight-coordinate Sm³⁺ [17]. The Yb–O and Yb*–O* bond dis-

tances, 2.370(3) Å, are in agreement with Yb←:OR₂ donor bonds. The bond angles of Yb–N–C1 94.8(3)° and O–C1–N 113.9(4)° are much smaller than the corresponding values, 118.7(6)° and 130.4(10)°, respectively, in complex **4**, which may result from the presence of the Yb←:OR₂ donor bond in complex **1**.

The Yb–C(ring) distances in complex **1** range from 2.605(6) to 2.698(5) Å. The Yb–C(ring) average distance of 2.643 Å is in the range of values found in other ytterbocene complexes: [(C₅H₅)₂Yb(μ -O(Me)C=CH-CH₃)₂], 2.629 Å [10]; [(C₅H₅)₂Yb(μ -OCH=C=CH₂)₂], 2.601 Å [20]; [(C₅H₅)₂Yb(OCH₂CH=CHCH₂)₂], 2.643 Å [21]; (*t*-BuC₅H₄)₂YbCl(THF), 2.628 Å [18]; (MeC₅H₄)₂YbNPh₂(THF), 2.625 Å [13].

2.3. Catalytic behavior

The catalytic behavior of complexes **1–3** for the ring-opening polymerization of ϵ -caprolactone was tested. The preliminary results were summarized in Table 2. All complexes show good catalytic activity and the satisfied polymerization yields at 60°C for 3 h in the case of 0.2% catalyst concentration. Regardless of the use of Yb or Er complex, they all give polymers with high molecular weight (Mn > 30 000) and moderate polydispersities (Mw/Mn < 2.20). The different catalytic activity among these complexes has been observed. The increasing order is Yb > Y > Er.

The effect of reaction conditions on the polymerization was examined using complex **2** as an initiator. In the case of 0.2% catalyst concentration, the monomer conversion increased with the increase of reaction time at 60°C, while the molecular weight distribution of the polymer slightly broadened (Table 2, runs 2, 4). The effect of polymerization temperature on the activity was also investigated. When the polymerization temperature is elevated from 60 to 80°C the yield increased from 57.6 to 100% (Table 2, runs 5–7) and the molecular weight distribution (Mw/Mn) becomes wider. This may be caused by the increase of side reactions such as transesterifications at high temperature.

3. Experimental

All manipulations were carried out under an argon atmosphere using standard Schlenk techniques. Toluene and hexane were dried over Na or sodium benzophenone ketyl and distilled before use. ϵ -Caprolactone was dried over CaH_2 for 4 days and stored over molecular sieves 3 Å under argon after distillation. ϵ -Caprolactam was recrystallized from hexane before use. $(\text{MeC}_5\text{H}_4)_2\text{LnCl}$ (Ln = Yb, Y, Er) were prepared by the method reported in the literature [7].

Melting points were determined in sealed argon-filled capillaries and uncorrected. Lanthanide metal analyses were carried out by complexometric titration. Carbon, hydrogen, and nitrogen analyses were performed on an EA 110-CHNS-O spectrometer by direct combustion. The IR spectra were obtained as KBr pellets on a Magna-550 spectrometer. Gel permeation chromatographic analyses were run on a Shimadzu GPC apparatus equipped with two Ultrastayragel columns in THF at 30°C. Monodisperse polystyrene standards were used to obtain the universal curve.

3.1. Preparation of $\{(MeC_5H_4)_2Yb-[O=C=N(CH_2)_4CH_2]\}_2$ (**1**)

A hexane solution of *n*-BuLi (6.4 ml, 6.13 mmol) was added at 0°C to a solution of ϵ -caprolactam (0.694 g, 6.13 mmol) in toluene, and the mixture was stirred for 20 min. Then a solution of $(\text{MeC}_5\text{H}_4)_2\text{YbCl}$ in toluene (27.3 ml, 6.13 mmol) was added at 0°C. The mixture was stirred for 1 h at 0°C and then for another 48 h at room temperature. After centrifugation, the residue was extracted with 30 ml of toluene for three times. The combined toluene solution was concentrated and cooled to -10°C for crystallization. Orange crystals were formed. Yield: 1.49 g (54.8%). M.p. = 284–288°C. Anal. Calc. for $\text{C}_{36}\text{H}_{48}\text{N}_2\text{O}_2\text{Yb}_2$: C, 48.76; H, 5.46; N, 3.16; Yb, 39.02. Found: C, 48.4; H, 5.38; N, 3.03; Yb, 38.94%. IR (KBr pellet, cm^{-1}): 3063w, 2960w, 2931w, 2853m, 2694w, 1607s, 1429m, 1391s, 1348s, 1277s, 1204s, 1153m, 1130s, 1030s, 958m, 927m, 876m, 832s, 768m, 752m, 697m, 615m, 487s.

3.2. Preparation of $\{(MeC_5H_4)_2Y-[O=C=N(CH_2)_4CH_2]\}_2$ (**2**)

The complex was prepared from 5.90 mmol of *n*-BuLi, 0.668 g of ϵ -caprolactam (5.90 mmol), and 26 ml of a solution of $(\text{MeC}_5\text{H}_4)_2\text{YCl}$ (5.90 mmol) in toluene using the procedure described above. Colorless crystals were obtained. Yield: 0.86g (40.6%). M.p. = 286–289°C. Anal. Calc. for $\text{C}_{36}\text{H}_{48}\text{N}_2\text{O}_2\text{Y}_2$: C, 60.17; H, 6.73; N, 3.90; Y, 24.74. Found: C, 59.53; H, 6.75; N, 4.01; Y, 24.14%. IR (KBr pellet, cm^{-1}): 3242w, 3088m,

2931s, 2853m, 2694w, 1604s, 1439m, 1389s, 1348s, 1276s, 1204s, 1153s, 1127s, 1086s, 1030s, 958m, 927m, 871m, 831s, 766s, 702s, 610m, 482w.

3.3. Preparation of $\{(MeC_5H_4)_2Er-[O=C=N(CH_2)_4CH_2]\}_2$ (**3**)

The complex was prepared from 5.72 mmol of *n*-BuLi, 0.647 g of ϵ -caprolactam (5.72 mmol), and 40.5 ml of a solution of $(\text{MeC}_5\text{H}_4)_2\text{ErCl}$ (5.72 mmol) in toluene using the procedure described above. Pink crystals were obtained. Yield: 1.15 g (45.9%). M.p. = 289–292°C. Anal. Calc. for $\text{C}_{36}\text{H}_{48}\text{N}_2\text{O}_2\text{Er}_2$: C, 49.41; H, 5.53; N, 3.20; Er, 38.21. Found: C, 49.02; H, 5.51; N, 3.07; Er, 37.74%. IR (KBr pellet, cm^{-1}): 3088m, 3063w, 2960w, 2931s, 2853m, 2740w, 1644w, 1491w, 1434m, 1390s, 1348s, 1301s, 1277s, 1153s, 1127s, 1086s, 1030s, 958m, 876m, 831s, 766s, 607m, 492m.

3.4. Polymerization of ϵ -caprolactone

To a toluene solution (10 ml) of ϵ -caprolactone (1 ml, 9.45 mmol) was added at once the toluene solution (1 ml) of complex **1**, **2** or **3** (0.0189 mmol) with vigorous magnetic stirring at the desired temperature. After the polymerization was carried out for a fixed time, 5 ml of ethanol containing 2% HCl solution was added to terminate the reaction, then the viscous solution mixture was poured into a large excess of petroleum ether to induce the precipitation of the polymer. The polymer was washed with petroleum ether three times and dried at 30°C in vacuum.

3.5. X-ray crystallographic analysis of **1**

Suitable crystals were selected and mounted in thin-walled glass capillaries for X-ray structure analysis. Diffraction data were collected at 293 K on a Rigaku AFC7R diffractometer using graphite-monochromated $\text{Mo-K}\alpha$ ($\lambda = 0.71069$ Å) radiation. Unit cell parameters were determined from a least-squares refinement using the setting angles of 20 carefully centered reflections in the range $18.30 < 2\theta < 26.57^\circ$. During the course of the collection of the intensity data, no significant decays were observed. The intensity was corrected for Lorentz-polarization effects and empirical absorption. A summary of crystallographic data is given in Table 3.

The structure was solved by heavy-atom Patterson methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. All calculations were performed using the TEXSAN [22] crystallographic software package of Molecular Structure Corporation.

Table 3
Experimental data for the X-ray diffraction study of complex 1

Formula	C ₃₆ H ₄₈ O ₂ N ₂ Yb ₂
Fw	886.87
Temperature (°C)	20
Radiation	Mo–K _α (0.71069 Å)
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁ / <i>n</i> (# 14)
Unit cell dimensions	
<i>a</i> (Å)	12.247(1)
<i>b</i> (Å)	11.137(2)
<i>c</i> (Å)	12.708(3)
β (°)	107.36(1)
<i>V</i> (Å ³)	1654.2(5)
<i>Z</i>	2
<i>D</i> _{calc} (g cm ⁻³)	1.780
μ(Mo–K _α) (cm ⁻¹)	56.52
<i>F</i> (000)	868.00
Crystal size (mm)	0.20 × 0.20 × 0.30
Scan type	ω–2θ
Scan width	1.52 + 0.30 tan θ
Scan rate (° min ⁻¹ , in ω)	16.0
2θ _{max} (°)	55.0
No. of reflections collected	3445
No. of independent reflections	3267 (<i>R</i> _{int} = 0.020)
No. of observations (<i>I</i> > 3.00σ(<i>I</i>))	2729
<i>R</i>	0.029
<i>R</i> _w	0.039
Goodness-of-fit	1.78
Max shift/error in final cycle	0.00
Largest difference peak and hole (e Å ⁻³)	0.73 and –1.95

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 150283 for complex 1. 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 <http://www.ccdc.cam.ac.uk>).

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