



Heterobimetallic complexes of lanthanide and lithium metals with dianionic guanidinate ligands: Syntheses, structures and catalytic activity for amidation of aldehydes with amines

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ABSTRACT

Reactions of triguanidinate lanthanide complexes $\text{Ln}[(^i\text{PrN})(\text{NC}_6\text{H}_4p\text{-Cl})\text{C}(\text{NH}^i\text{Pr})]_3$ ($\text{Ln} = \text{Nd}, \text{Y}$) with 3 equiv. of *n*-BuLi gave $[\text{Li}(\text{THF})(\text{DME})]_3\text{Ln}[\mu\text{-}\eta^2\eta^1(^i\text{PrN})_2\text{C}(\text{NC}_6\text{H}_4p\text{-Cl})]_3$, which represents the first structurally characterized complexes of lanthanide and lithium metals with dianionic guanidinate ligands. The Nd complex was found to be an effective catalyst for amidation of aldehydes with amines under mild conditions with a wide scope of substrates.

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1. Introduction

Heterobimetallic complexes of lanthanide and alkali metals are of interest because they have proven to enable transformations that have never been accomplished using monometallic analogs of lanthanide by virtue of cooperative effects between the two different metals [1–3]. For example, the heterobimetallic binaphthoxide and phenoxide complexes of lanthanide and alkali metals have been explored to be versatile asymmetric catalysts in organic syntheses [1–9]. Various organometallic complexes of trivalent lanthanide and alkali metals [10–17], as well as the complexes of divalent lanthanide and sodium metals [18,19] have proven to be more effective catalysts in polymerization of nonpolar and polar monomers than their corresponding lanthanide complexes without alkali metals. Recently, we have addressed that phenoxides [20] and bridged bisamidinate complexes [21] of lanthanide and alkali metals are much more active catalysts for amidation of aldehydes with amines under mild conditions in comparison with their monolanthanide partner. Now we are particularly interested in synthesis and reactivity of bimetallic complexes with guanidinate ligands because monoanionic guanidinates have widely been used

in organolanthanide chemistry as ancillary ligands [22–32]. Moreover, trisguanidinate lanthanides complexes can also serve as effective catalysts for polymerization of lactone [33–35] and for amidation of aldehydes with amines [36]. Considering the ability to generate dianionic species by deprotonating of a second N–H bond which provides the possibility to construct a new kind of heterobimetallic complex [37], the *N,N,N'*-trialyl guanidinate ligands were chosen. While various complexes of d-block transition metals and s-block main metals with dianionic *N,N,N'*-trialyl guanidinates ligands were synthesized [37–44], no example of lanthanide metals has been reported up to date. Here we report the syntheses and molecular structures of the first heterobimetallic complexes of lanthanide and lithium metals with dianionic guanidinate ligands $[\text{Li}(\text{THF})(\text{DME})]_3\text{Ln}[\mu\text{-}\eta^2\eta^1(^i\text{PrN})_2\text{C}(\text{NC}_6\text{H}_4p\text{-Cl})]_3$ ($\text{Ln} = \text{Nd}, \text{Y}$). Their high activity for mild amidation of aldehydes with amines was also presented.

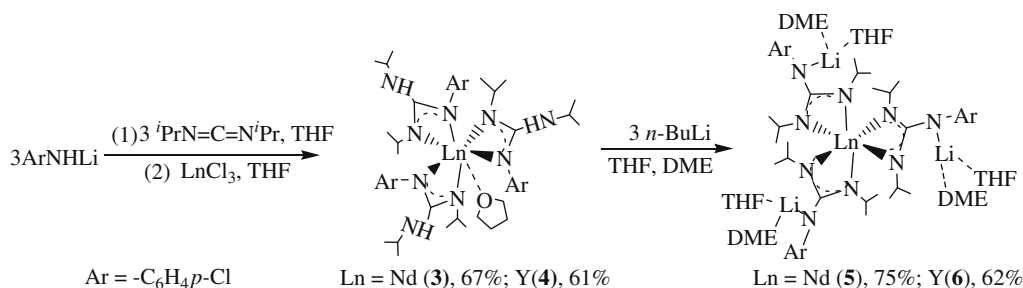
2. Results and discussion

2.1. Syntheses of complexes 1–6 and molecular structures of complexes 3–6

Reaction of anhydrous LnCl_3 ($\text{Ln} = \text{Nd}, \text{Y}$) with 3 equiv. of in situ formed lithium guanidinate $\text{Li}[(^i\text{PrN})(\text{NC}_6\text{H}_4p\text{-Cl})\text{C}(\text{NH}^i\text{Pr})]$ **1** by reaction of lithium amide $\text{LiNHC}_6\text{H}_4p\text{-Cl}$ **2** with *N,N*-diisopropylcarbodiimide $^i\text{PrN}=\text{C}=\text{N}^i\text{Pr}$ in THF at room temperature, afforded the

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Scheme 1.

asymmetric triguanidinate complexes by a 1,3-H shift $\text{Ln}[(^i\text{PrN})(\text{NC}_6\text{H}_4\text{p-Cl})\text{C}(\text{NH}^i\text{Pr})]_3\text{THF}$ (Ln = Nd, **3**; Y, **4**) as blue crystals for **3** and as colorless crystals for **4** (Scheme 1). Complex **3** was further confirmed by single-crystal X-ray analysis (Fig. 1). The selected bond lengths and angles are listed in Table 1. The molecular structure of **3** is quite similar to that of analogous La complex [36], in which the central Nd ion is bound to three chelating asymmetric guanidinates and coordinated to one THF molecule in a capped octahedron. The average Nd–N bond distance of 2.492(6) Å is in the range for the published analogues [7] and the C–N distances within the chelate rings (av. 1.345(5) Å) are consistent with partial double bond character.

The reaction of **3** and **4** with 3 equiv. of *n*-BuLi, respectively, was then tried in an attempt to synthesize heterobimetallic complexes with dianionic guanidinate ligands by deprotonation of N–H bond. The reaction went rapidly at room temperature in THF. Removing the THF and extracting the residue with toluene resulted in the isolation of complexes **5** and **6** in moderate yields upon crystallization from a mixture of toluene and DME at room temperature. Single crystal X-ray analysis of these new products showed they to be the heterobimetallic complexes with formula $[\text{Li}(\text{THF})(\text{DME})]_3\text{Ln}[\mu\text{-}\eta^2\eta^1(^i\text{PrN})_2\text{C}(\text{NC}_6\text{H}_4\text{p-Cl})]_3$ (Ln = Nd, **5**; Y, **6**) (Scheme 1), not the asymmetric guanidinate complexes

$[\text{Li}(\text{THF})(\text{DME})]_3\text{Ln}[\mu\text{-}\eta^2\eta^1(^i\text{PrN})(\text{NC}_6\text{H}_4\text{p-Cl})\text{C}(\text{N}^i\text{Pr})]_3$ (Ln = Nd, Y). Obviously complexes **5** and **6** are derived from triple deprotonation followed by the rearrangement. The occurrence of the rearrangement may make these complexes more stable.

Complexes **5** and **6** are isostructural. The selected bond lengths and angles are listed in Table 1. The coordination sphere around each metal center is composed of six nitrogen atoms from the three dianionic ligands to form a distorted octahedron. The third nitrogen atom of each guanidinate (N3, N6 and N9) lies outside of the metal coordination sphere, but bonds to each lithium ion (Li1, Li2 and Li3) (Fig. 2). The three Li–N bond distances in both **5** and **6** are comparable and the average distances of 1.978(16) Å for **5** and 1.996(17) Å for **6** are in the range of single Li–N bond (1.96 Å). The values are also comparable to those found in lithium amides $[\text{Li}(\text{N}(\text{TMS})_2)_3]$ [45,46] and $[\{\text{Li}(\text{N}(\text{TMS})_2)(\text{OEt}_2)\}_2]$ [45]. Furthermore, the average C–N(Li) (C₆H₄p-Cl) bond distances of 1.405(9) Å for **5** and 1.396(9) Å for **6** are consistent with a CN single bond. Such a $\mu\text{-}\eta^2\eta^1$ coordination mode for a dianionic guanidinate has not been found previously. Each four-membered metallacycle Ln–N–C–N for both complexes is planar. The Ln–N bond distances vary from 2.437(6) to 2.505(6) Å for **5** and 2.329(6) to 2.410(6) Å for **6**, and the average distances of 2.471(6) Å for **5** and 2.338(6) Å for **6** are almost consistent with 2.492(6) Å in **3**

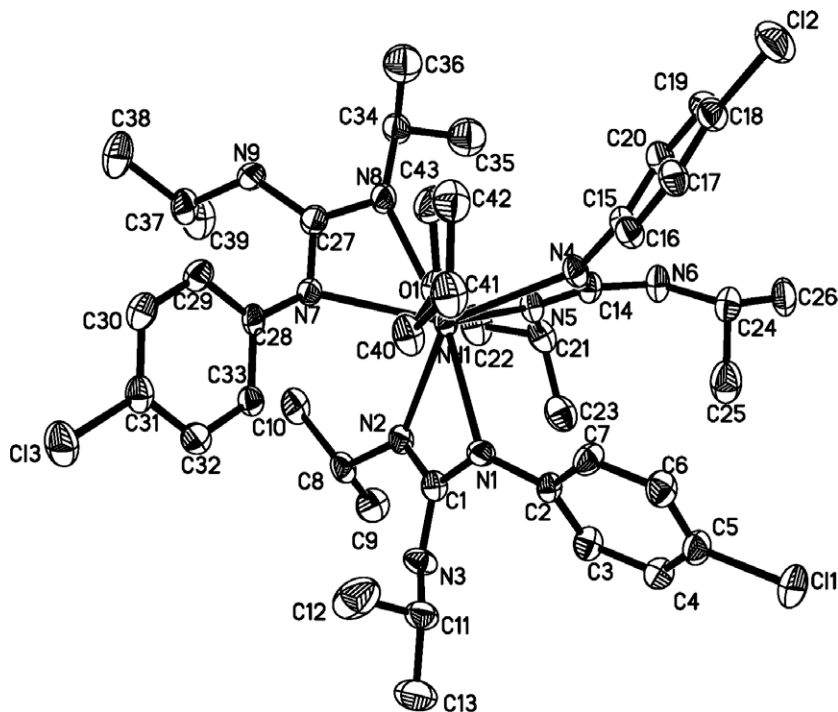


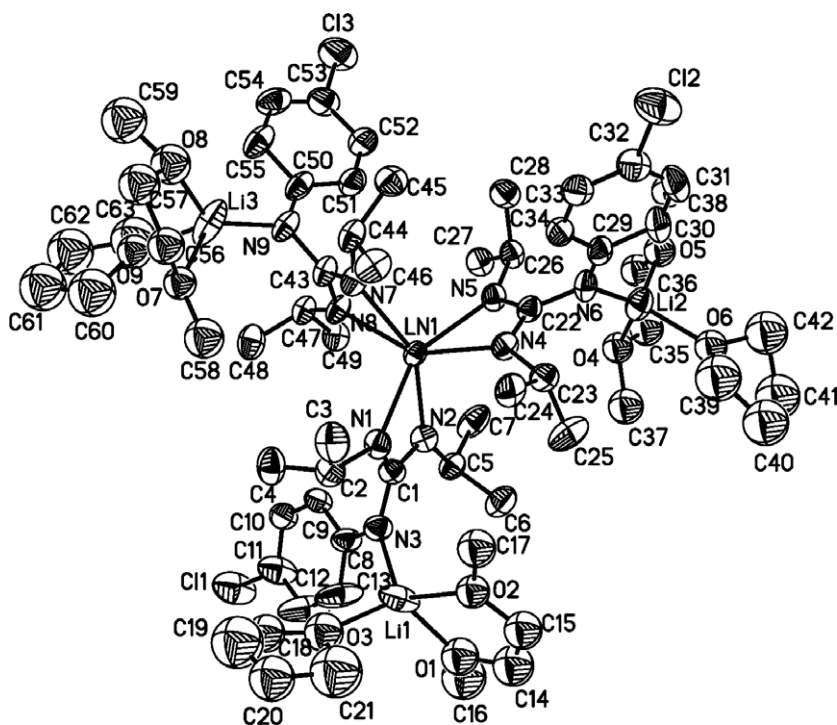
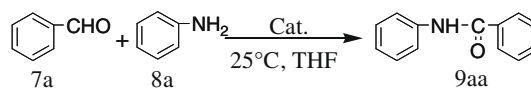
Fig. 1. The molecular structure of complex **3**. Hydrogen atoms are omitted for clarity, and thermal ellipsoids are drawn at the 30% probability level.

Table 1
Selected bond lengths (Å) and angles (°) for **3**, **5** and **6**.

	3	5	6
Nd(1)–N(1)	2.486(6)	Ln(1)–N(1)	2.486(6)
Nd(1)–N(2)	2.507(6)	Ln(1)–N(2)	2.437(6)
Nd(1)–N(4)	2.487(6)	Ln(1)–N(4)	2.447(6)
Nd(1)–N(5)	2.455(6)	Ln(1)–N(5)	2.505(6)
Nd(1)–N(7)	2.496(6)	Ln(1)–N(7)	2.488(6)
Nd(1)–N(8)	2.518(6)	Ln(1)–N(8)	2.461(5)
Nd(1)–N(8)	1.358(9)	N(1)–C(1)	1.360(9)
N(1)–C(1)	1.331(9)	N(2)–C(1)	1.340(9)
N(2)–C(1)	1.382(9)	N(4)–C(22)	1.341(9)
N(4)–C(14)	1.382(9)	N(5)–C(22)	1.338(9)
N(5)–C(14)	1.319(9)	N(7)–C(43)	1.339(9)
N(7)–C(27)	1.348(9)	N(8)–C(43)	1.344(9)
N(8)–C(27)	1.332(9)	N(3)–C(1)	1.399(9)
Nd(1)–O(1)	2.551(5)	N(6)–C(22)	1.409(9)
N(3)–C(1)	1.376(9)	N(9)–C(43)	1.406(9)
N(6)–C(14)	1.372(9)	N(3)–Li(1)	1.977(16)
N(9)–C(27)	1.385(9)	N(6)–Li(2)	1.968(16)
		N(9)–Li(3)	1.99(2)
N(1)–Nd(1)–N(2)	54.1(2)	N(2)–Ln(1)–N(1)	54.45(19)
N(5)–Nd(1)–N(4)	54.5(2)	N(4)–Ln(1)–N(5)	53.93(19)
N(7)–Nd(1)–N(8)	53.08(19)	N(8)–Ln(1)–N(7)	54.01(19)
C(1)–N(1)–Nd(1)	95.3(5)	C(1)–N(1)–Ln(1)	94.4(4)
C(1)–N(2)–Nd(1)	95.1(5)	C(1)–N(2)–Ln(1)	97.1(4)
C(14)–N(4)–Nd(1)	93.7(4)	C(22)–N(4)–Ln(1)	97.3(4)
C(14)–N(5)–Nd(1)	96.8(5)	C(22)–N(5)–Ln(1)	94.8(4)
C(27)–N(7)–Nd(1)	96.9(4)	C(43)–N(7)–Ln(1)	95.0(4)
C(27)–N(8)–Nd(1)	96.3(5)	C(43)–N(8)–Ln(1)	96.0(4)
N(2)–C(1)–N(1)	115.1(7)	N(2)–C(1)–N(1)	113.1(6)
N(5)–C(14)–N(4)	113.8(7)	N(5)–C(22)–N(4)	113.9(6)
N(8)–C(27)–N(7)	113.5(7)	N(7)–C(43)–N(8)	113.8(6)

when the differences in ionic radius was considered [47]. The C–N bond distances within the guanidinate fragment are indicative of delocalized π -bond (av. 1.344(9) Å for **5** and 1.350(9) Å for **6**).

The coordination geometry around each Li ion is a tetrahedron by one nitrogen atom and additional three oxygen atoms from one THF and one DME molecules with a normal Li–O bond distance (av. 2.056 Å for **5** and 2.050 Å for **6**).

**Fig. 2.** The molecular structure of complex **5** (LN1 = Nd) and **6** (LN1 = Y). Hydrogen atoms are omitted for clarity, and thermal ellipsoids are drawn at the 30% probability level.**Table 2**
Optimization of complexes **1–6** catalyzed amidation of aldehydes with amines.^a

Entry	Cat. (mol%)	Yield (%)	Entry	Cat. (mol%)	Yield (%) ^b
1	1 (3%)	30	4	4 (2%)	30
2	2 (3%)	27	5	5 (1%)	92
3	3 (2%)	42	6	6 (1%)	80

^a Starting amine, aldehyde, and catalyst concentrations are 0.02 mmol/mL in each experiment; amine/aldehyde = 1/3.

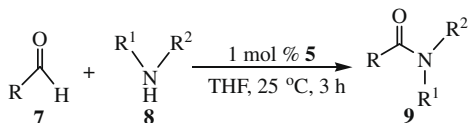
^b Isolated yields based on aniline.

2.2. Catalytic activity of complexes **1–6** for amidation reactions of aldehydes with amines

To further address the reactivity of these new heterobimetallic complexes, amidation of aldehydes with amines under mild conditions were examined with them as amides are important structural units in many biologically and pharmaceutically active compounds. The catalytic activity of **5** and **6** for amidation of benzaldehyde **7a** with aniline **8a** was first tested in THF at 25 °C. For control the same reaction with complexes **1–4**, respectively, was also conducted. As shown in Table 2, bimetallic complexes **5** and **6** are much more active than the corresponding monometallic complex **3** and **4**: the amide **9aa** is produced in the yield of 92% for **5** and 80% for **6** at the catalyst loading of 1 mol% while only 42% for **3** and 30% yield for **4** at the catalyst loading of 2 mol%. The two lithium complexes **1** and **2** are both less active (Table 2, entries 1 and 2). The high activity of heterobimetallic complexes may result from the cooperative effects by lanthanide and Li metals. Preliminary results also showed that complex **5** is a very robust and effective

catalyst with good functional group tolerance and a wide scope of substrates (Table 3). Less electrophilic cyclohexanal and less nucleophilic secondary cyclic amines, pyrrolidine, piperidine, and morpholine, also offered good to excellent yields at 1 mol% catalyst loading (Table 3), indicating that **5** is the most effective catalyst ever known [20,21,36,48]. It was supposed that one of the intermediates for the amidation might be an amide by the amination of complex **5(6)** with amine according to the mechanism presented

Table 3
Complex **5** catalyzed amidation of aldehydes with amines.^a



Entry	Aldehyde	Amine	Acylamide	Yield (%) ^b
1				92
2				88
3				90
4				80
5				87
6				91
7				97
8				91
9				90
10				90
11				90

^a Starting amine, aldehyde, and catalyst concentrations are identical in each experiment; amine/aldehyde = 1/3.

^b Isolated yields based on amine.

[48]. An attempt to isolate the amide was not successful yet. The work on it is going on.

3. Conclusion

We have described the syntheses and structures of the first heterobimetallic complexes of Ln and Li with dianionic guanidates [Li(THF)(DME)]₃Ln[μ-η²η¹ (i^{Pr}N)₂C(NC₆H₄p-Cl)]₃ (Ln = Nd, Y) via deprotonation of second N–H bond followed by the rearrangement. The Nd complex can serve as an effective catalyst for mild amidation of aldehydes with amines.

4. Experimental section

4.1. General procedures

All manipulations and reactions were performed under a purified argon atmosphere using standard Schlenk techniques. Solvents were degassed and distilled from sodium benzophenone ketyl prior to use. [(ArN)C(NⁱPr)(HNⁱPr)]₃Ln [Ar = *p*-ClPh, Ln = Nd (**3**), Ln = Y (**4**)] [(ArNLi)C(NⁱPr)₂]₃Ln [Ar = *p*-ClPh, Ln = Nd (**5**), Ln = Y (**6**)] were prepared. All aldehydes and amines were predried, sublimed, recrystallized or distilled before use. Melting points were determined in sealed Ar-filled capillary tube, and uncorrected. ¹H and ¹³C NMR spectra were recorded on a Unity Inova-400 spectrometer. Chemical shifts (δ) were reported in ppm.

4.2. Synthesis of [i^{Pr}NHCNⁱPr(NC₆H₄p-Cl)]₃Nd·THF (**3**)

A Schlenk flask was charged with *p*-ClC₆H₄NH₂ (1.905 g, 15.0 mmol), THF (30 mL), and a stir bar. The solution was cooled to 0 °C, and *n*-BuLi (8.6 mL, 15.0 mmol, 1.75 M in hexane) was added, then slowly warmed to room temperature and stirred for 1 h. To this solution was added *N,N*-diisopropylcarbodiimide (2.4 mL, 15.3 mmol) at 0 °C. The resulting solution was slowly warmed to room temperature and stirred for 1 h and then added slowly to a blue slurry of NdCl₃ (1.22 g, 5.0 mmol) in 20 mL THF. The color of the solution immediately changed to blue. The resulting solution was then stirred for another 24 h and evaporated to dryness in vacuo. The residue was extracted with Et₂O and LiCl was removed by centrifugation. The extracts were concentrated and cooled to 0 °C for crystallization, to afford blue crystals. Yield: 3.26 g (67%). Mp: 180–182 °C. Anal. Calc. for C₄₃H₆₅Cl₃N₉NdO (974.63): C, 52.99; H, 6.72; N, 12.93; Nd, 14.80. Found: C, 53.00; H, 6.61; N, 13.01; Nd, 14.71%. IR (KBr pellet): 3320 (s) cm⁻¹, 3061 (s), 2981 (s), 2910 (s), 2888 (s), 1635 (s), 1577 (s), 1520 (s), 1421 (s), 1370 (s), 1328 (s), 1165 (m), 1123 (m), 1034 (m), 940 (m).

4.3. Synthesis of [i^{Pr}NHCNⁱPr(NC₆H₄p-Cl)]₃Y·THF (**4**)

Following the procedure similar to that for the synthesis of **3**, complex Li[i^{Pr}NHCNⁱPr(NC₆H₅)] (15.0 mmol), which was formed in situ by the reaction of LiNHC₆H₅ with ⁱPrN=C=NⁱPr, reacted with YCl₃ (0.975 g, 5.0 mmol), in THF (60 mL) to yield the colorless crystals (**4**) upon crystallization from ether. Yield: 2.80 g (61%). ¹H NMR (400 MHz, C₆D₆): 7.35–7.31 (m, 6H, *m*-H-Ph), 6.93–6.74 (m, 6H, *o*-H-Ph), 3.64–3.50 (m, 7H, α-H-THF and N-H), 3.32–3.17 (m, 6H, H-C(N)Me₂), 1.48–1.23 (m, 4H, β-H, THF), 0.95–0.78 (d, 36H, CH₃) ppm. Anal. Calc. for C₄₃H₆₅Cl₃N₉YO (919.33): C, 56.18; H, 7.13; N, 13.71; Y, 9.67. Found: C, 56.07; H, 7.08; N, 13.69; Y, 9.60%. IR (KBr pellet): 3377 (s) cm⁻¹, 3079 (s), 2963 (s), 2924 (s), 2874 (s), 1636 (s), 1578 (s), 1516 (s), 1423 (s), 1363 (s), 1327 (s), 1160 (m), 1122 (m), 1014 (m), 944 (m).

4.4. Synthesis of [(p-CIPhNli)C(NⁱPr)₂]₃Nd·3THF·3DME (**5**)

A Schlenk flask was charged with [ⁱPrNHCNⁱPr(NC₆H₄p-Cl)]₃Nd·THF (2.92 g, 3.0 mmol), THF (30 mL), and a stir bar. The solution was cooled to 0 °C, and *n*-BuLi (5.1 mL, 9.0 mmol, 1.75 M in hexane) was added, then slowly warmed to room temperature and stirred for 24 h. The resulting solution was then evaporated to dryness in vacuo. DME (15 mL) and toluene (5 mL) was added to the residue and then heated until to the solution being clear. The solution was cooled to room temperature for crystallization and the blue crystals (**5**) was isolated. Yield: 3.17 g (75%). Mp: 230–228 °C. Anal. Calc. for C₆₃H₁₀₈Cl₃Li₃N₉O₉Nd (1406.99): C, 53.78; H, 7.74; N, 8.96; Nd, 10.25. Found: C, 53.67; H, 7.72; N, 8.95; Nd, 10.21%. IR (KBr pellet): 3074 (s) cm⁻¹, 2974 (s), 2926 (s), 2869 (s), 1636 (s), 1609 (s), 1585 (s), 1546 (s), 1499 (s), 1485 (s), 1365 (m), 1165 (m), 1091 (m), 1007 (m), 952 (m), 859(m).

4.5. Synthesis of [(p-CIPhNli)ⁱPrNCNⁱPr]₃Y·3THF·3DME (**6**)

Following the procedure for the synthesis of **5**, the reaction of [ⁱPrNHCNⁱPr(NC₆H₄p-Cl)]₃Y·THF (3.0 mmol) in THF (20 mL) with *n*-BuLi (5.1 mL, 9.0 mmol, 1.75 M in hexane) afforded **6** as colorless crystals upon crystallization from DME and toluene. Yield: 2.51 g (62%). Mp: 212–210 °C. ¹H NMR (400 MHz, TDF): δ = 7.01–6.89 (m, 6H, *m*-H-Ph), 6.55–6.46 (m, 6H, *o*-H-Ph), 3.66–3.58 (m, 12H, *α*-H-THF), 3.41–3.38 (m, 12H, O-CH₂-), 3.33–3.30 (m, 6H, H-C(N)Me₂), 3.24 (s, 18H, O-CH₃), 1.77–1.70 (m, 4H, β-H, THF), 1.10–0.97 (d, 36H, CH₃) ppm. ¹³C NMR (100 MHz, TDF): δ = 155.7, 127.7, 123.3, 121.2, 115.8, 71.9, 67.4, 58.1, 45.5, 25.6, 23.3 (Anal. Calc. for C₆₃H₁₀₈Cl₃Li₃N₉O₉Y (1351.66 ppm): C, 55.98; H, 8.05; N, 9.33; Y, 6.58. Found: C, 55.78; H, 7.82; N, 8.93; Y, 6.56%. IR (KBr pellet): 3074 (s) cm⁻¹, 2973 (s), 2926 (s), 2872 (s), 1630 (s), 1600 (s), 1585 (s), 1542 (s), 1500 (s), 1484 (s), 1359 (m), 1163 (m), 1090 (m), 1012 (m), 952 (m), 859 (m).

4.6. X-ray crystallography of complexes **3**, **5** and **6**

Crystals of complexes **3**, **5** and **6** suitable for X-ray diffraction study were sealed in a thin-walled glass capillary filled under argon. Diffraction data were collected on a Rigaku Mercury CCD area detector in ω scan mode using Mo Kα radiation (λ = 0.71070 Å). The diffracted intensities were corrected for Lorentz polarization effects and empirical absorption corrections. Details of the intensity data collection and crystal data are given in Table 4.

The structures were solved by direct methods and refined by full-matrix least-squares procedures based on |F|². All the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were all generated geometrically, assigned appropriate isotropic thermal parameters, and allowed to ride on their parent carbon atoms. All the H atoms were held stationary and included in the structure factor calculation in the final stage of full-matrix least-squares refinement. The structures were solved and refined using SHELXL-97 program.

4.7. General procedure for the synthesis of amides from reaction of amines with aldehydes catalyzed by complex **5**

A 30 mL of Schlenk flask under dried argon was charged with the solution of complex **5** (0.01 mmol, 0.50 mL THF). Amine was added (1.00 mmol), after stirring for 0.5 h, aldehyde was then added (3.00 mmol). The resulting mixture was stirred at 25 °C for 3 h, filtered through a small plug of silica gel to remove the catalyst. The crude product was purified by column chromatography. All products were identified by comparison with those of authentic samples.

Table 4

Details of the crystallographic data and refinements for complexes **3**, **5** and **6**.

	3	5	6
Empirical formula	C ₄₃ H ₆₅ Cl ₃ N ₉ NdO	C ₆₃ H ₁₀₈ Cl ₃ Li ₃ N ₉ NdO ₉	C ₆₃ H ₁₀₈ Cl ₃ Li ₃ N ₉ O ₉ Y
Fw	974.63	1406.99	1351.66
Temperature (K)	223(2)	223(2)	223(2)
λ (Å)	0.71075	0.71075	0.71075
Crystal system	Triclinic	Monoclinic	Monoclinic
Space group	<i>P</i> $\bar{1}$	<i>P</i> 21/ <i>c</i>	<i>P</i> 21/ <i>c</i>
<i>a</i> (Å)	10.375(3)	19.072(3)	19.029(3)
<i>b</i> (Å)	13.052(4)	12.2723(18)	12.0563(19)
<i>c</i> (Å)	18.016(5)	35.336(5)	35.222(6)
α (°)	80.124(7)	90	90
β (°)	89.943(8)	94.420(2)	94.175(3)
γ (°)	86.812(8)	90	90
<i>V</i> (Å ³)	2399.5(13)	8246(2)	8059
<i>Z</i>	2	4	4
<i>D</i> _{calc} (g cm ⁻³)	1.349	1.133	1.114
μ (mm ⁻¹)	1.290	0.776	0.874
<i>F</i> (0 0 0)	1010	2964	2880
θ Range (°)	3.01–25.50	3.01–25.50	3.00–25.50
No. of rflns	16 520	31 830	46 446
No. of unique rflns	8746	15 146	14 767
<i>R</i> _{int}	0.0689	0.0325	0.0715
Variables	521	728	729
<i>R</i> [<i>I</i> > 2σ(<i>I</i>)]	1.099	0.0864	0.1308
w <i>R</i> ₂	0.1737	0.2391	0.3192
GOF	1.099	1.169	1.141

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Appendix A. Supplementary data

CCDC 751062, 751063 and 751064 contain the supplementary crystallographic data for complexes **3**, **5** and **6**. These data can be obtained free of charge from The Cambridge Crystallographic Data Center via http://www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2009.12.010.

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