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Synthesis of lanthanide chlorides supported by β-diketiminate ligands and molecular structures of $L^1SmCl_2(THF)_2$ and $L^2SmCl_2(THF)_2$ [L^1 = PhNC(Me)CHC(Me)NPh; L^2 = *p*-ClPhNC(Me)CHC(Me)NPh(2,6-Pr^i_2)]

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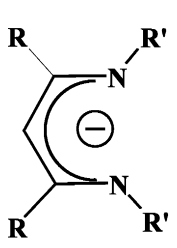
Abstract

The β-diketiminate lithium salts [L^1Li, L^1 = PhNC(Me)CHC(Me)NPh; L^2Li, L^2 = *p*-ClPhNC(Me)CHC(Me)NPh(2,6-Pr^i_2)] reacted with anhydrous $LnCl_3$ (Ln = Sm, Yb) in 1:1 molar ratio in THF to afford the new heteroleptic lanthanide complexes $L^1LnCl_2(THF)_2$ (Ln = Sm (**1**), Yb (**2**)) and $L^2LnCl_2(THF)_2$ (Ln = Sm (**3**), Yb (**4**)) in high yields. The substituents on the arene ring have significant effect on the solubility of these complexes. Crystal structure analysis revealed that complexes **1** and **3** are both monomeric, in which the samarium atom is coordinated by two nitrogen atoms of the β-diketiminate ligand, two chloro and two THF ligands in distorted octahedral environment.

Keywords: Synthesis; Crystal structures; Lanthanide; Samarium; Ytterbium; β-Diketiminate ligand

1. Introduction

In recent years, the use of β-diketiminates (**A**) as supporting ligand systems in both main and transition metal coordination chemistry has attracted considerable attention [1–25]. These ligands have several attractive features. For example, β-diketiminate ligands and cyclopentadienyl anions are isoelectronic, and both of them are monoanions in their deprotonated forms; the steric and electronic properties of β-diketiminate ligands can be readily altered through an appropriate choice of amine and β-diketone used in their synthesis [26]. In particular, some of these β-diketiminate complexes have found potential applications as homogeneous polymerization catalysts [9–16]. However, the utilization of β-diketiminate ligands for the preparation of lanthanide complexes remains relatively poorly explored [27–31].



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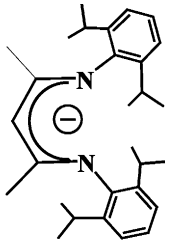

B

Recently, we became interested in the synthesis and reactivity of β-diketiminate lanthanide complexes and

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found that replacing one methylcyclopentadienyl by a $(DIPPh)_2$nacnac group ($(DIPPh)_2$nacnac = *N*,*N*-diisopropylphenyl-2,4-pentanediimine anion, (**B**)) in $(CH_3C_5H_4)[(DIPPh)_2nacnac]YbNPh_2$ results in a dramatic decrease of catalytic activity for the polymerization of methyl methacrylate. We attribute this drop to the steric congestion around the metal center brought about by the bulky $(DIPPh)_2$nacnac ligand [32]. In order to further study the effect of β-diketiminate ligands on the structure and reactivity of lanthanide complexes, we used two less bulky β-diketiminate ligands L^1H [L^1 = PhNC(Me)CHC(Me)NPh] and L^2H [$L^2 = p$-ClPhNC(Me)CHC(Me)NPh(2,6-Pr^i_2)] as reactants and synthesized four β-diketiminate lanthanide dichlorides, which serve as important precursors for further transformation. Herein, we report the synthesis and characterization of these complexes and the crystal structures of $L^1SmCl_2(THF)_2$ (**1**) and $L^2SmCl_2(THF)_2$ (**3**).

2. Results and discussion

2.1. Synthesis

One equiv. of L^1Li was added to a slurry of $LnCl_3$ (Ln = Sm, Yb) in toluene–THF mixed solvent at room temperature. The color of the solution gradually changed to yellow (for Sm) or red (for Yb), and yellow or red precipitate appeared at the same time. After work up, the yellow (for complex **1**) or red (for complex **2**) precipitate was collected by centrifugation. The compositions of complexes **1** and **2** were established as $L^1LnCl_2(THF)_2$ (Ln = Sm (**1**), Yb (**2**)) by elemental analyses (C, H, N and Ln) (Scheme 1). The IR spectra of complexes **1** and **2** exhibited strong absorptions near 1554 and 1532 cm^{-1}, which were consistent with partial C=N double bond character [33].

Complexes **1** and **2** are slightly soluble in THF. Due to their sparing solubility in THF, they can be precipitated from the reaction mixture in high yield (81–85%). This behavior is quite different from that of $[(DIPPh)_2nacnac]YbCl_2(THF)_2$, which is very soluble in THF [32]. It is really unexpected that replacing the diisopropylphenyl groups by the phenyl groups on the β-diketiminate ligand results in such a dramatic decrease of its solubility in organic solvents.

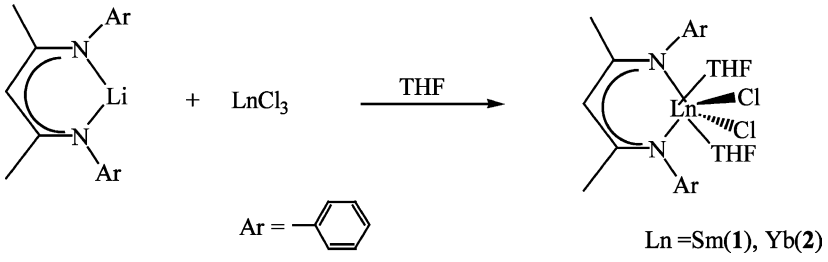
In order to increase the solubility of lanthanide β-diketiminate complex, a more bulky β-diketiminate ligand, L^2H, was synthesized. The reaction of its lithium salt with anhydrous $LnCl_3$ (Ln = Sm, Yb) in THF at room temperature gave, after 48 h, a bright yellow or red solution of L^2SmCl_2 or L^2YbCl_2, respectively. After workup, the yellow (for complex **3**) or red (for complex **4**) microcrystals were obtained in high yields which were identified to be $L^2LnCl_2(THF)_2$ (Ln = Sm (**3**), Yb (**4**)) by elemental analyses (C, H, N and Ln) and IR spectra, as shown in Scheme 2.

As expected, complexes **3** and **4** are quite soluble in THF, and moderately soluble in toluene. These results indicate that selected suitable substituents on the arene ring of β-diketiminate ligand can efficiently improve the solubility of β-diketiminate lanthanide complex. Complexes **1**–**4** are slightly sensitive to air and moisture, and the crystals can be exposed in air for a few hours without apparent decomposition.

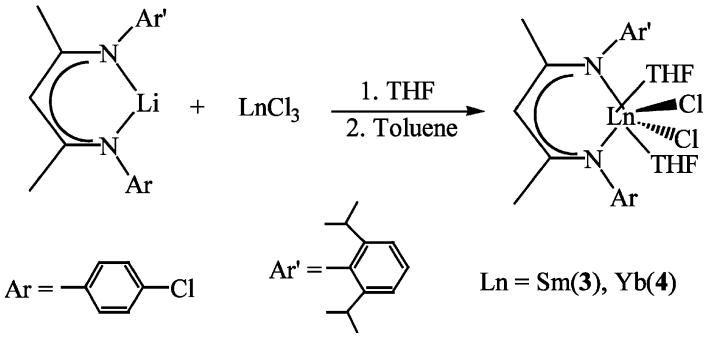
2.2. Crystal structure of $L^1SmCl_2(THF)_2$ (1) and $L^2SmCl_2(THF)_2$ (3)

Since the structures of $(L)LnCl_2(THF)_2$ derivatives (where L is a β-diketiminate ligand) have seldom been reported [29], the structures for complexes **1** and **3** were determined. Both complexes **1** and **3** have monomeric structure in the solid state. The two THF molecules in complex **3** are disordered due to strong thermal motion. The molecular structures of complexes **1** and **3** are shown in Figs. 1 and 2, and their selected bond lengths and angles are listed in Tables 1 and 2, respectively. The coordination geometries around samarium atoms in complexes **1** and **3** are similar, and each samarium atom is six-coordinate with two nitrogen atoms of the β-diketiminate ligand, two chloro, and two THF ligands in a distorted octahedron. The molecular structures establish cis disposition of the THF ligands, and trans disposition of the chlorine atoms.

In complex **1**, the β-diketiminate ligand is asymmetrically coordinated to the samarium atom, such that the Sm(1)–N(2) distance (2.37(1) Å) is about 0.08 Å longer than the Sm(1)–N(1) distance (2.29(1) Å). This is similar



Scheme 1.



Scheme 2.

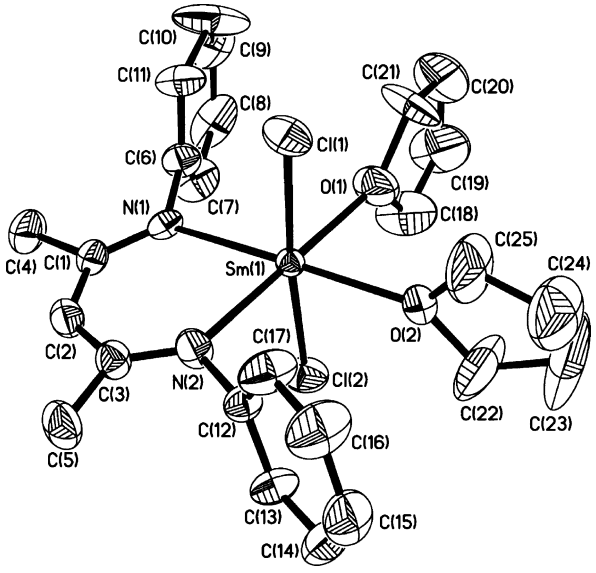


Fig. 1. Molecular structure of $L^1SmCl_2(THF)_2$.

Table 1
Selected bond lengths (Å) and angles (°) in complex **1**

Bond lengths			
Sm(1)–O(1)	2.34(1)	Sm(1)–N(1)	2.29(1)
Sm(1)–O(2)	2.29(1)	Sm(1)–N(2)	2.37(1)
Sm(1)–Cl(2)	2.538(2)	Sm(1)–Cl(1)	2.541(2)
N(1)–C(1)	1.30(1)	N(2)–C(3)	1.27(2)
C(1)–C(2)	1.40(1)	C(2)–C(3)	1.39(1)
Bond angles			
O(2)–Sm(1)–N(1)	178.3(4)	O(2)–Sm(1)–O(1)	82.5(5)
N(1)–Sm(1)–O(1)	99.3(3)	O(2)–Sm(1)–N(2)	98.8(3)
N(1)–Sm(1)–N(2)	79.5(4)	O(1)–Sm(1)–N(2)	177.5(5)
O(2)–Sm(1)–Cl(2)	87.6(3)	N(1)–Sm(1)–Cl(2)	92.4(3)
O(1)–Sm(1)–Cl(2)	86.9(3)	N(2)–Sm(1)–Cl(2)	91.0(4)
O(2)–Sm(1)–Cl(1)	88.7(4)	N(1)–Sm(1)–Cl(1)	91.4(3)
O(1)–Sm(1)–Cl(1)	89.4(3)	N(2)–Sm(1)–Cl(1)	92.8(4)
Cl(2)–Sm(1)–Cl(1)	175.1(2)	C(3)–ZC(2)–C(1)	130.6(9)

to that found in $[(DIPPh)_2nacnac]ScCl_2(THF)$ [29], but different from that in $L^1GdBr_2(THF)_2$, in which two nitrogen atoms are symmetrically coordinated to the central metal [27]. The average Sm–N bond length is 2.33(1) Å, which is apparently shorter than the average Sm–N bond length in L_3^1 Sm (2.466(8) Å) [27]. This can be attributed to steric congestion around samarium atom in the latter. Subtraction of the effective ionic radius (0.958 Å) [34] for six-coordinate Sm^{3+} from the

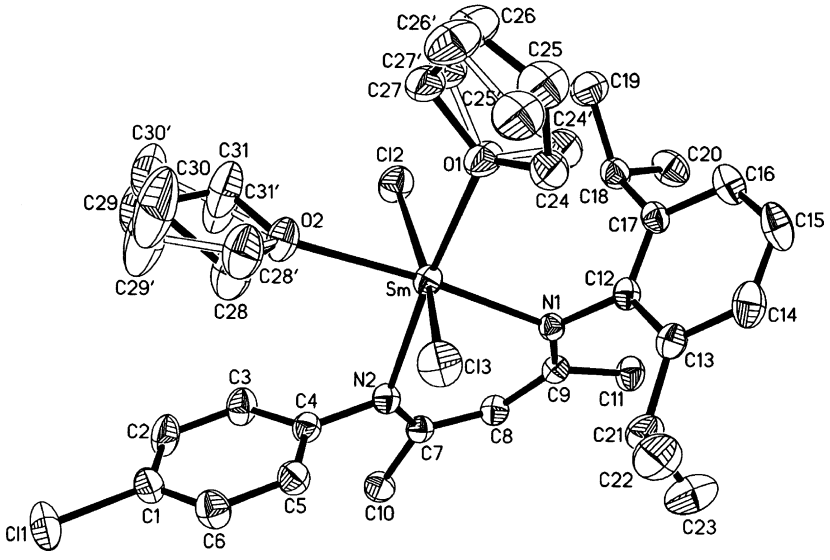


Fig. 2. Molecular structure of $L^2SmCl_2(THF)_2$. Each disordered THF ligand is shown in two possible orientations.

Table 2
Selected bond lengths (Å) and angles (°) in complex **3**

Bond lengths			
Sm(1)–O(1)	2.463(2)	Sm(1)–N(1)	2.398(2)
Sm(1)–O(2)	2.477(2)	Sm(1)–N(2)	2.387(3)
Sm(1)–Cl(2)	2.628(1)	Sm(1)–Cl(3)	2.638(1)
N(1)–C(9)	1.332(4)	N(2)–C(7)	1.331(4)
C(7)–C(8)	1.402(4)	C(8)–C(9)	1.392(4)
Bond angles			
O(2)–Sm(1)–N(1)	171.92(8)	O(2)–Sm(1)–O(1)	87.34(9)
N(1)–Sm(1)–O(1)	100.12(8)	O(2)–Sm(1)–N(2)	95.36(9)
N(1)–Sm(1)–N(2)	77.54(9)	O(1)–Sm(1)–N(2)	173.54(8)
O(2)–Sm(1)–Cl(2)	82.72(6)	N(1)–Sm(1)–Cl(2)	94.03(6)
O(1)–Sm(1)–Cl(2)	90.58(6)	N(2)–Sm(1)–Cl(2)	95.58(7)
O(2)–Sm(1)–Cl(3)	85.30(6)	N(1)–Sm(1)–Cl(3)	98.81(6)
O(1)–Sm(1)–Cl(3)	81.30(7)	N(2)–Sm(1)–Cl(3)	93.06(7)
Cl(2)–Sm(1)–Cl(3)	165.82(3)	C(7)–C(8)–C(9)	131.4(3)

average Sm–N distance gives 1.371 Å, which is slightly shorter than those in $L^1GdBr_2(THF)_2$ (1.428 Å) [27] and [(DIPPh)$_2$nacnac]$ScCl_2(THF)$ (1.456 Å) [29], but comparable with that in (MeC_5H_4)[(DIPPh)$_2$nacnac]YbCl (1.375 Å) [32]. Two Sm–Cl bond lengths are 2.538(2) and 2.541(2) Å, which are slightly shorter than the corresponding distances in [(DIPPh)$_2$nacnac]$ScCl_2(THF)$ [29] even when the difference in ionic radii is considered.

The bond distances of C(1)–C(2), C(2)–C(3), N(1)–C(1) and N(2)–C(3) lie intermediate between the corresponding single- and double-bond distances (see Table 1), which suggest significant delocalization within the π-system of β-diketiminate backbone. The Sm–C(1, 2, 3) distances are quite long, suggesting a negligible π contribution to the β-diketiminate-Sm bonding in this compound. The N(1)–Sm(1)–N(2) angle is 79.5(4)°, which is more acute than the corresponding angles in complexes [(DIPPh)$_2$nacnac]$ScCl_2(THF)$, [29] and {[(DIPPh)$_2$nacnac]YbCl(μ-Cl)$_3$Yb[(DIPPh)$_2$-nacnac](THF)}·1/2MePh [32].

In complex **3**, the β-diketiminate ligand is symmetrically coordinated to the samarium atom with Sm–N bond lengths of 2.398(2) and 2.387(3) Å, which is different from that in complex **1**. Moreover, the average Sm–N bond length of 2.392(3) Å in **3** is apparently longer than that in **1**, although both complexes have similar coordination geometry. However, this value is still comparable with those in $L^1GdBr_2(THF)_2$ [27] and [(DIPPh)$_2$nacnac]$ScCl_2(THF)$ [29] when the difference in ionic radii is considered. The long distances of Sm–C(7, 8, 9) reveal that the β-diketiminate ligand is coordinated to the samarium atom in an η^2 manner. There is the expected pattern of delocalization within the $SmNC_3N$ six-membered ring. The average Sm–Cl and Sm–O(THF) bond lengths in complex **3** are about 0.10 and 0.16 Å longer than those in complex **1**, respectively. It is reasonable to ascribe these differences in bond parameters to the increased steric congestion in the latter due to the bulky substituents on one arene ring. The N(1)–Sm(1)–N(2) angle is 77.54(9)°, which is comparable with those in complex **1**, $L^1GdBr(THF)_2$ and L^1_3 Ln (Ln = Sm, Gd) [27].

3. Experimental

Reactions were performed under pure Ar with exclusion of air and moisture by Schlenk techniques. Solvents were dried and freed of oxygen by refluxing over sodium or sodium benzophenone ketyl and distilled under Ar prior to use. Anhydrous $LnCl_3$ [35] and L^1Li [26] were prepared according to the literature methods.

Melting points were determined in sealed Ar-filled capillaries and are uncorrected. Metal analyses were carried out using complexometric titration. Carbon, hydrogen, and nitrogen analyses were performed by direct combustion on a Carlo Erba-1110 instrument; quoted data are the average of at least two independent determinations. The IR spectra were recorded on a Nicolet-550 FTIR spectrometer as KBr. ^1H NMR spectra were recorded on a Unity Varian-400 spectrometer.

3.1. Synthesis of 2-(2,6-diisopropylphenyl)aminopent-2-en-4-one (5)

A 20.0 mL amount of 2,6-diisopropylaniline (0.106 mol) was added to a solution of 2,4-pentanedione (16.4 mL, 0.159 mol) in $C_6H_5CH_3$ (100 mL) in a round-bottomed flask. The resulting mixture was heated to reflux for 4 h, and water was removed as a $C_6H_5CH_3$ azeotrope using a Dean and Stark apparatus. The reaction mixture was then evaporated to dryness. The resulting solid was recrystallized from C_6H_{14} to afford 2-(2,6-diisopropylphenyl)aminopent-2-en-4-one (**5**) (23.9 g, 87%). ^1H NMR (400 MHz, $CDCl_3$): 1.14 (d, 6H, CH_3CHCH_3), 1.20 (d, 6H, CH_3CHCH_3), 1.64 (s, 3H, CH_3), 2.13 (s, 3H, CH_3), 2.99–3.06 (m, 2H, CH_3CHCH_3), 5.21 (s, 1H, CH=C(CH_3)N), 7.18 (d, 2H, aromatic protons), 7.28–7.32 (m, 1H, aromatic proton), 12.06 (s, 1H, N*H*).

3.2. Synthesis of 2-(2,6-diisopropylphenyl)aminopent-2-en-4-(p-chlorophenyl)imine (L^2H) (6)

p-Chloroaniline hydrochloride (12.6 g, 0.077 mol), compound **5** (20 g, 0.077 mol), and 100 ml of absolute EtOH were added to a 250 ml round-bottomed flask. The reaction mixture was allowed to reflux for 4 h. The reaction solution was then evaporated to dryness. After stirring with 40 ml saturated Na_2CO_3, 2-(2,6-diisopropylphenyl)aminopent-2-en-4-(*p*-chlorophenyl)imine (L^2H) was extracted into ethyl ether. Evaporation of solvent and recrystallization from C_6H_{14} afforded L^2H

as a white crystalline solid (9.7 g, 34%). ^{1}H NMR (400 MHz, $CDCl_3$): 1.12 (d, 6H, CH_3CHCH_3), 1.21 (d, 6H, CH_3CHCH_3), 1.68 (s, 3H, CH_3), 2.01 (s, 3H, CH_3), 2.97–3.04 (m, 2H, CH_3CHCH_3), 4.88 (s, 1H, CH=C(CH_3)N), 6.81 (d, 2H, aromatic protons), 7.13 (s, 3H, aromatic protons), 7.20 (d, 2H, aromatic proton), 12.54 (s, 1H, NH).

3.3. Synthesis of $L^1SmCl_2(THF)_2$ (1)

A solution of L^1Li (25 ml, 5.26 mmol) in $C_6H_5CH_3$/C_6H_{14} was slowly added to a suspension of $SmCl_3$ (1.47 g, 5.73 mmol) in 40 ml THF at room temperature (r.t.). The color of the solution gradually changed to yellow. The reaction mixture was stirred overnight at r.t., then centrifugation to remove the solution. Complex **1** was obtained as a yellow powder (2.85 g, 81%), m.p. 182–184 °C (dec.). *Anal*. Calc. for $C_{25}H_{33}Cl_2N_2O_2Sm$: C, 48.84; H, 5.41; N, 4.55; Sm, 24.46. Found: C, 48.27; H, 5.30; N, 4.58; Sm, 24.18%. IR (KBr, cm^{-1}): 3418(m), 2963(m), 2928(m), 2859(m), 1637(s), 1601(s), 1554(vs), 1532(vs), 1489(s), 1439(s), 1381(m), 1288(s), 1180(m), 1026(m), 752(m), 691(s). Crystals suitable for X-ray crystal structure studies were obtained by recrystallization from THF at −5 °C in 2 days.

3.4. Synthesis of $L^1YbCl_2(THF)_2$ (2)

The synthesis of compound **2** was carried out as described for **1**, but anhydrous $YbCl_3$ (1.28 g, 4.58 mmol) was used in place of $SmCl_3$. The red precipitate was obtained from THF solution at r.t. (2.48 g, 85%), m.p. 175–178 °C (dec.). *Anal*. Calc. for $C_{25}H_{33}Cl_2N_2O_2Yb$: C, 47.10; H, 5.18; N, 4.39; Yb, 27.16. Found: C, 46.43; H, 5.22; N, 4.26; Yb, 27.62%. IR (KBr, cm^{-1}): 3422(m), 2974(s), 2927(m), 2859(m), 1620(s), 1556(vs), 1535(vs), 1489(s), 1439(s), 1373(w), 1364(s), 1300(s), 1038(m), 760(m), 694(s).

3.5. Synthesis of $L^2SmCl_2(THF)_2$ (3)

A solution of *n*-BuLi in C_6H_{14} (4.3 ml, 7.65 mmol) was added to the THF (20 ml) solution of L^2H (2.80 g, 7.61 mmol) at 0 °C, the mixture was stirred at 0 °C for 1 h, then another 2 h at r.t. The solution was then slowly added to the suspension of $SmCl_3$ (1.93 g, 7.53 mmol) in 60 ml THF at r.t. The color of the solution gradually changed to yellow. The reaction mixture was stirred 48 h at r.t. The solvent was removed in vacuum and $C_6H_5CH_3$ was added to extract the product. The dissolved portion was removed by centrifugation. The yellow micro crystals were obtained from the concentrated $C_6H_5CH_3$ solution at r.t. (3.6 g, 65%), m.p. 187–189 °C (dec.). *Anal*. Calc. for $C_{31}H_{44}Cl_3N_2O_2Sm$: C, 50.77; H, 6.05; N, 3.82; Sm, 20.50. Found: C, 50.14; H, 6.00; N, 3.83; Sm, 20.75%. ^{1}H NMR (400 MHz, C_6D_6): 1.18 (d, 6H, $CH(CH_3)_2$), 1.24 (d, 6H, $CH(CH_3)_2$), 1.48 (m, 16H, THF), 1.65 (s, 3H, CH_3), 1.78 (s, 3H, CH_3), 3.22 (m, 2H, CH_3CHCH_3), 3.65 (m, 16H, THF), 4.88 (s, 1H, CH=C(CH_3)N), 6.69–7.22 (d, 7H, aromatic protons). IR (KBr, cm^{-1}): 3422(m), 2963(m), 2932(m), 2858(m), 1628(s), 1554(s), 1532(s), 1292(m), 1117(m), 1096(m), 1026(m), 926(w). Crystals suitable for X-ray crystal structure studies were obtained by recrystallization from $C_6H_5CH_3$ at −5 °C in a few days.

3.6. Synthesis of $L^2YbCl_2(THF)_2$ (4)

The synthesis of compound **4** was carried out as described for **3**, but anhydrous $YbCl_3$ (1.83 g, 6.61 mmol) was used in place of $SmCl_3$. The product was collected in two crops by filtration (3.3 g, 67%), m.p. 179–181 °C (dec.). *Anal*. Calc. for $C_{31}H_{44}Cl_3N_2O_2Yb$: C, 49.24; H, 5.87; N, 3.71; Yb, 22.89. Found: C, 48.64; H, 5.91; N, 3.76; Yb, 22.64%. ^{1}H NMR (400 MHz, C_6D_6): 1.17 (d, 6H, $CH(CH_3)_2$), 1.24 (d, 6H, $CH(CH_3)_2$), 1.59 (m, 16H, THF), 1.65 (s, 3H, CH_3), 1.77 (s, 3H, CH_3), 3.21 (m, 2H, CH_3CHCH_3), 3.90 (m, 16H, THF), 4.88 (s, 1H, CH=C(CH_3)N), 6.53–7.41 (d, 7H, aromatic protons). IR (KBr, cm^{-1}): 3445(m), 2963(m), 2932(m), 2855(m), 1632(s), 1554(s), 1532(s), 1489(s), 1393(w), 1296(s), 1099(m), 1026(m), 926(w).

3.7. X-ray structure determination

Suitable single crystals of complexes **1** and **3** were each sealed in a thin-walled glass capillary, and intensity data were collected on a Siemens SMART diffractometer equipped with a CCD detector (for **1**) or Siemens P4 diffractometer in ω scan mode (for **3**) using Mo Kα radiation ($\lambda = 0.71073$ Å). Details of the intensity data collection and crystal data are given in Table 3.

The crystal structures of these complexes were solved by direct methods using the SHELXS-97 program and expanded by Fourier techniques. All the non-hydrogen atoms were refined anisotropically. Hydrogen atoms were all generated geometrically (C–H bond lengths fixed at 0.95 Å) with assigned appropriate isotropic thermal parameters.

4. Supplementary data

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 177470 and 177471 for complexes **1** and **3**, respectively. 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).

Table 3
Crystal data and experimental parameters

Compound	**1**	**3**
Formula	$C_{25}H_{33}Cl_2N_2O_2Sm$	$C_{31}H_{44}Cl_3N_2O_2Sm$
Molecular weight	614.78	733.38
Temperature (K)	298(2)	293(2)
Crystal system	orthorhombic	monoclinic
Crystal color and habit	yellow, prism	yellow, prism
Space group	*Pna*2(1)	*P*2(1)/*c*
Unit cell dimensions		
a (Å)	19.642(5)	9.130(1)
b (Å)	9.573(3)	25.037(4)
c (Å)	14.082(4)	14.840(2)
β (°)	90	97.33(1)
V (Å^3)	2648.0(12)	3364.5(8)
Z	4	4
D_{calc} (g cm^{-3})	1.542	1.448
μ (mm^{-1})	2.442	2.012
F(000)	1236	1492
Crystal size (mm)	0.25 × 0.20 × 0.15	0.48 × 0.44 × 0.34
θ_{max} (°)	27.52	25.01
Reflections collected	12 872	6695
Reflections with $I \geq 2.0\sigma(I)$	4274	4635
Independent reflections	5648	5919
Parameters refined	290	383
R	0.0469	0.0265
wR_2	0.0981	0.0393

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