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Ytterbium complexes supported by β-diketiminate ligands: cyclopentadienyl, indenyl, and aryloxide derivatives

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Abstract

Reactions of β-diketiminate ytterbium dichloride, $LYbCl_2(THF)_2$ (L = *N*,*N*-2,6-diisopropylphenyl-2,4-pentanediimine anion), with C_5H_5Na, in 1:1 or 1:2 molar ratio in tetrahydrofuran (THF) at room temperature give the mixed-ligand ytterbium complexes $(C_5H_5)(L)YbCl$ (**1**) and $(C_5H_5)_2YbL$ (**2**), respectively. $LYbCl_2(THF)_2$ reacts with C_9H_7K or ArONa ($ArO = 2,6\text{-}Bu^t_2\text{-}4\text{-}MeC_6H_2O$) in 1:1 molar ratio in THF to afford $(C_9H_7)(L)YbCl$ (**3**) and (ArO)(L)YbCl(THF) (**4**) in high yield, respectively. Crystal structure analysis revealed that complex **3** is an unsolvated monomer, in which the ytterbium atom is coordinated by one indenyl ring, two nitrogen atoms of the diketiminate ligand and one chlorine atom, while complex **4** is a THF solvated monomer with the central metal five-coordinated by two nitrogen atoms, two oxygen atoms, and one chlorine atom.

Keywords: Organolanthanide complex; Ytterbium; β-Diketiminate ligand; Synthesis; Crystal structures

1. Introduction

In recent years, nitrogen-containing bidentate chelating ligands have attracted considerable interest in organometallic chemistry. Amidinates [1–5], guanidinates [6,7], and β-diketiminates [8–24] are well established as ancillary ligands for transition and main group metals. These ligand systems can coordinate metal center in different bonding modes, and their steric and electronic properties can be readily tuned by an appropriate choice of starting materials used in their synthesis. The coordination chemistry of lanthanide elements and Group 3 metals supported by amidinates has been studied [25–27]. In contrast, the utilization of guanidinates [28–30] and β-diketiminate ligands [31–34] for the preparation of lanthanide complexes remains relatively poorly explored.

Recently, we became interested in studying the synthesis and reactivity of organolanthanide complexes supported by the β-diketiminate ligand, and found that the bulky β-diketiminate group $(DIPPh)_2$nacnac (L) (L = *N*,*N*-2,6-diisopropylphenyl-2,4-pentanediimine anion) is an ideal ligand for synthesis of the mixed-ligand lanthanide derivatives [35]. In order to further explore the reactivity of β-diketiminate lanthanide chloride, we have studied the reaction of $LYbCl_2(THF)_2$ with some anionic ligands, including cyclopentadienyl, indenyl, and aryloxide. In this paper, we report the synthesis and characterization of these ytterbium complexes, and the molecular structures of $(C_9H_7)(L)YbCl$ (**3**) and (ArO)(L)YbCl(THF) (**4**) (ArO = 2,6-di-tert-butyl-4-methylphenoxo) are also described.

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2. Experimental

Reactions were performed under pure argon with exclusion of air and moisture by Schlenk techniques. Solvents were dried and freed of oxygen by refluxing over Na or sodium benzophenone ketyl and distilled under argon prior to use. Anhydrous $YbCl_3$ [36] and LLi [8] were prepared according to the literature methods. C_5H_5Na and ArONa were obtained by the reaction of C_5H_6 or ArOH with sodium in THF. C_9H_7K was prepared by the reaction of C_9H_8 with potassium in THF.

Melting points were determined in sealed argon-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 pellets.

2.1. *(C_5H_5)(L)YbCl (**1**)*

To a slurry of anhydrous $YbCl_3$ (1.75 g, 6.26 mmol) in about 40 ml THF was slowly added a solution of LLi (20.0 ml, 6.26 mmol) in toluene/hexane at room temperature. After $YbCl_3$ had disappeared completely, the THF solution of C_5H_5Na (9.8 ml, 6.26 mmol) was added slowly. The mixture was stirred at room temperature for another 48 h, and then the precipitate was removed from the reaction mixture by centrifugation. THF was completely removed in vacuum and toluene was added to extract the product. The precipitate was removed again by centrifugation. The orange red crystals were obtained from the concentrated toluene solution at −10 °C for 3 days (3.61 g, 83.5%). m.p.: 218–220 °C. Anal. Calc. for $C_{34}H_{46}ClN_2Yb$: C, 59.08; H, 6.71; N, 4.05; Yb, 25.03. Found: C, 58.87; H, 6.67; N, 3.98; Yb, 24.87%. IR (KBr, cm^{-1}): 3058(m), 2961(vs), 2928(s), 2865(s), 1622(vs), 1551(vs), 1521(vs), 1463(s), 1435(s), 1388(s), 1365(s), 1316(s), 1265(s), 1172(m), 1101(m), 1071(w), 1022(m), 931(m), 852(w), 835(m), 788(s), 782(s), 756(s), 689(w).

2.2. *(C_5H_5)$_2$YbL (**2**)*

The synthesis of complex **2** was carried out as described for complex **1**, but 2 equiv. of C_5H_5Na in THF (17.1 ml, 10.88 mmol) was used. The orange red crystals were obtained from the concentrated toluene solution at −10 °C (3.42 g, 87.3%). M.p.: 235–238 °C. Anal. Calc. for $C_{39}H_{51}N_2Yb$: C, 64.98; H, 7.13; N, 3.88; Yb, 24.00. Found: C, 64.66; H, 6.98; N, 3.68; Yb, 24.25%. IR (KBr, cm^{-1}): 3056(m), 2963(vs), 2925(s), 2867(s), 1623(vs), 1551(vs), 1524(vs), 1461(s), 1433(s), 1385(s), 1366(s), 1315(s), 1264(s), 1170(m), 1103(m), 1072(w), 1023(m), 935(m), 851(w), 832(m), 788(s), 783(s), 755(s), 689(w).

2.3. *(C_9H_7)(L)YbCl (**3**)*

The synthesis of complex **3** was carried out as described for complex **1**, but a THF solution of C_9H_7K (1.14 ml, 5.83 mmol) was used instead of the solution of C_5H_5Na. The black crystals were obtained from concentrated toluene solution at −20 °C for 4 days (3.34 g, 77.3%). m.p.: 245–247 °C. Anal. Calc. for $C_{38}H_{48}ClN_2Yb$: C, 61.57; H, 6.53; N, 3.78; Yb, 23.34. Found: C, 61.33; H, 6.24; N, 3.58; Yb, 23.51%. IR (KBr, cm^{-1}): 3062(w), 2962(vs), 2928(s), 2868(s), 1622(vs), 1552(vs), 1528(vs), 1461(s), 1437(s), 1388(s), 1363(s), 1325(s), 1276(s), 1178(s), 1096(m), 1068(w), 1019(m), 933(m), 786(m), 759(s), 692(m). The crystals suitable for X-ray crystal structures studies were obtained by recrystallization from toluene solution at room temperature for 3 days.

2.4. *(ArO)(L)YbCl(THF) (**4**)*

The synthesis of complex **4** was carried out as described for complex **1**, but a THF solution of ArONa (1.56 mmol) was used in place of the solution of C_5H_5Na. The red crystals were obtained from toluene solution (1.16 g, 81.2%). m.p.: 153–156 °C (dec). Anal. Calc. for $C_{48}H_{72}ClN_2O_2Yb$: C, 62.83; H, 7.91; N, 3.05; Yb, 18.86. Found: C, 62.52; H, 7.92; N, 3.07; Yb, 18.72%. IR (KBr, cm^{-1}): 3061(w), 2960(vs), 2927(s), 2867(s), 1635(s), 1621(vs), 1550(vs), 1531(vs), 1463(s), 1439(s), 1396(m), 1362(s), 1325(s), 1277(s), 1225(m), 1175(s), 1152(m), 1120(w), 1073(w), 932(m), 865(m), 789(s), 759(s), 685(w), 642(w). The crystals suitable for X-ray crystal structures studies were obtained by recrystallization from THF–toluene solution at −5 °C for a week.

2.5. *X-ray structures determination*

Suitable single crystals of complexes **3** and **4** were each sealed in thin-walled glass capillary, and intensity data were collected at ambient temperature on a Siemens P4 diffractometer in ω-scan mode using Mo Kα radiation ($\lambda = 0.71073$ Å). Details of the intensity data collection and crystal data are given in Table 1.

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.

Table 1
Crystallographic data for complexes **3** and **4**

Compound	**3**	**4**
Formula	$C_{38}H_{48}ClN_2Yb$	$C_{48}H_{72}ClN_2O_2Yb$
Fw	741.27	917.57
T (K)	290(2)	290(2)
Radiation	Mo Kα	Mo Kα
Crystal system	triclinic	triclinic
Space group	$P\bar{1}$	$P\bar{1}$
Unit cell		
a (Å)	10.479(1)	10.379(1)
b (Å)	12.392(1)	11.193(1)
c (Å)	14.273(1)	21.418(2)
α (°)	97.301(7)	84.06(1)
β (°)	98.179(7)	85.47(1)
γ (°)	106.933(8)	68.86(1)
V (Å^3)	1727.0(2)	2305.9(4)
Z	2	2
D_{calcd} (g cm^{-3})	1.425	1.322
μ (Mo Kα) (cm^{-1})	28.12	21.23
F(0 0 0)	754	954
Crystal size (mm)	0.58 × 0.42 × 0.20	0.52 × 0.42 × 0.30
$2\theta_{max}$ (°)	50.0	50.0
No. of reflections collected	6625	8898
No. of unique reflections	6049	8117
No of observed reflections [$I > 2.0\sigma(I)$]	4778	7328
GOF	0.949	1.014
R	0.0393	0.0221
R_w	0.0855	0.0548
Largest difference peak and hole (e Å^{-3})	2.130, −0.664	0.585, −0.664

3. Results and discussion

3.1. Synthesis

We previously reported that $LYbCl_2(THF)_2$ can be obtained in high yield by the metathesis reaction of LLi with $YbCl_3$ in 1:1 molar ratio [35]. Thus, freshly prepared $LYbCl_2(THF)_2$ in situ was used as a precursor to synthesize the β-diketiminato ytterbium derivatives. Anhydrous $YbCl_3$ reacted completely with LLi in 1:1 molar ratio in THF. A THF solution of C_5H_5Na was added in 1:1 or 1:2 molar ratio. After workup, orange red crystals were obtained in high yield, which were identified to be $(C_5H_5)(L)YbCl$ (**1**) and $(C_5H_5)_2YbL$ (**2**) by elemental analyses, respectively. We proposed that complex **1** exists as a monomer according to the structures of the analogous complex $(CH_3C_5H_4)(L)YbCl$ which was identified by structure determination [35]. Complex **2** may be a solvent-free monomer, just like $(C_5H_5)_3Yb$, one β-diketiminato instead of one $C_5H_5^-$ to coordinate to the ytterbium atom. Attempts to determine the definitive structures of **1** and **2** were unsuccessful due to twinning of the crystals.

When 1 equiv. of C_9H_7K in THF was added to the THF solution of $LYbCl_2$, the color of the solution gradually changed from red to black. The black crystals obtained were identified to be $(C_9H_7)(L)YbCl$ (**3**) by elemental analyses, IR spectrum and X-ray crystal structures determination (see below). Usually, the synthesis of unsubstituted indenyl lanthanide chlorides was difficult due to disproportionation. To our knowledge, there are few structurally characterized unsubstituted indenyl lanthanide chlorides in the literature [37,38]. Complex **3** is the first example of an indenyl-containing mixed-ligand lanthanide chloride. When $LYbCl_2$ was treated with 1 equiv. of ArONa in THF, the desired mixed-ligand aryloxo ytterbium complex (ArO)(L)-YbCl(THF) (**4**) was isolated as red crystals in high yield. All reactions studied are shown in Scheme 1. These results reveal that the $(DIPPh)_2$nacnac anion is an ideal ligand for the synthesis of mixed-ligand lanthanide derivatives.

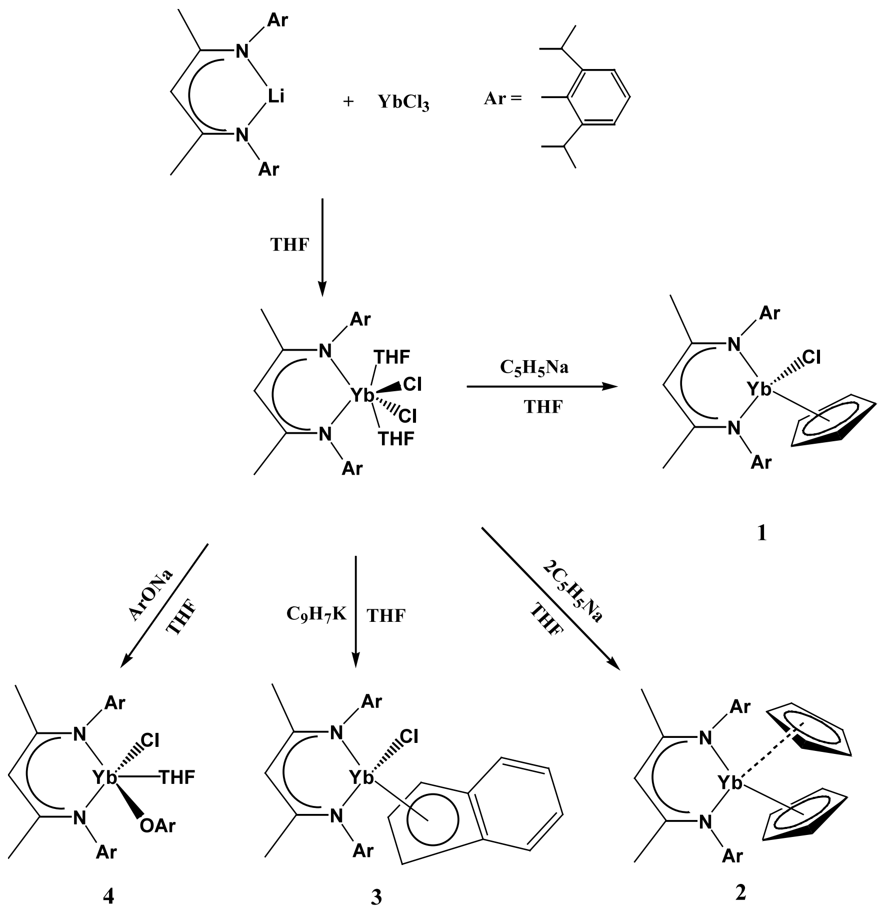
In the IR spectra, there are strong absorptions near 1550 and 1530 cm^{-1}, which are consistent with partial C=N double bond character [39]. These complexes are moderately sensitive to air and moisture, and have good thermal stability, they are well soluble in THF and DME, moderately in aromatic solvents, and insoluble in aliphatic hydrocarbons, such as hexane.

3.2. Molecular structure of $(C_9H_7)(L)YbCl$ (**3**)

The molecular structure of complex **3** is shown in Fig. 1, and the selected bond lengths and angles are listed in Table 2. The coordination geometry about the ytterbium center approximates a three-legged piano stool with the two nitrogen atoms of the chelating β-diketiminate anion, and the chlorine atom constituting the "legs" of the molecule, and the formal coordination number of the central metal is 6.

The Yb–C(13) and Yb–C(15) distances are 3.124(6) and 3.120(6) Å, respectively, which are somewhat longer than those corresponding bond distances of 3.086(4) and 3.049(5) Å in $(CH_3C_5H_4)(L)YbCl$ [35], but still comparable with those of 2.986(6) and 3.180(9) Å for bridging η^2-C_5H_5 bonding in $(C_5Me_5)_2Sm(\mu\text{-}C_5H_5)Sm(C_5Me_5)_2$ [40], and 2.814(4)–3.148(6) Å for chelating η^6-, η^1-Ph-Yb bonding in $[Yb(Odpp)_3]$ (Odpp = 2,6-diphenylphenolate) [41], respectively. Therefore, the β-diketiminate ligand can be considered to coordinate to ytterbium in a η^4 fashion. The bond distances of C(13)–C(14), C(14)–C(15), N(1)–C(13) and N(2)–C(15) (see Table 2) fall in the range of corresponding distances of single and double bonds, which reflect substantial electron delocalization within the backbone of the β-diketiminate ligand.

The β-diketiminate ligand is symmetrically coordinated to the ytterbium atom with the variation of 0.008 Å in Yb–N bond lengths (2.245(4) and 2.237(4) Å, respectively). The average Yb–N bond length of 2.241(4) Å is comparable with that in



Scheme 1.

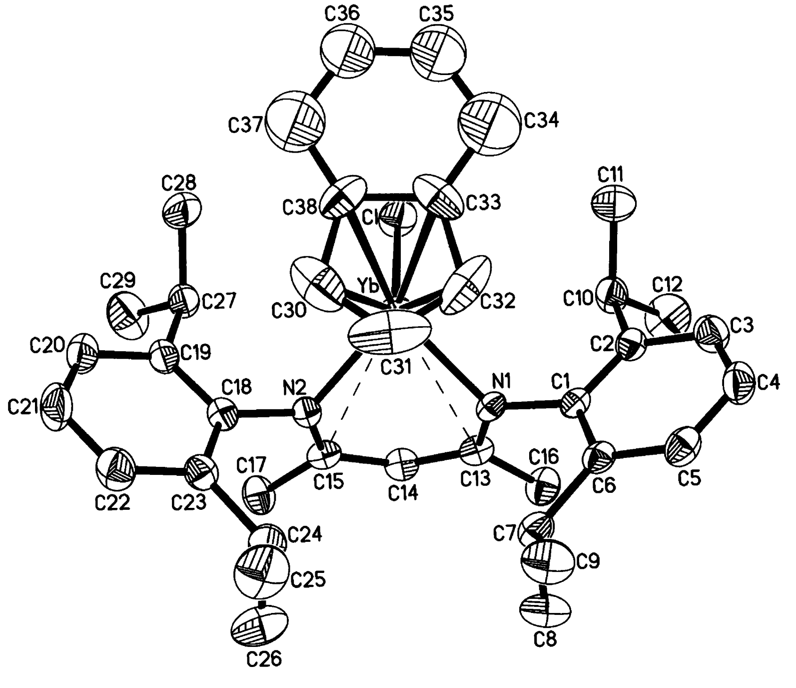


Fig. 1. Molecular structure of complex **3** (hydrogen atoms omitted for clarity).

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

Bond lengths			
Yb–N(1)	2.245(4)	Yb–C(1)	3.198(5)
Yb–N(2)	2.237(4)	Yb–C(13)	3.124(6)
Yb–Cl	2.469(2)	Yb–C(14)	3.361(6)
Yb–C(30)	2.614(8)	Yb–C(15)	3.120(6)
Yb–C(31)	2.58(1)	Yb–C(18)	3.191(6)
Yb–C(32)	2.567(8)	N(1)–C(13)	1.335(7)
Yb–C(33)	2.653(6)	N(2)–C(15)	1.327(7)
Yb–C(38)	2.678(6)	C(13)–C(14)	1.395(8)
Yb–C(av)	2.617(8)	C(14)–C(15)	1.414(8)
Bond angles			
N(1)–Yb–N(2)	83.5(2)	N(1)–Yb–C(13)	21.9(2)
N(1)–Yb–Cl	103.5(1)	N(2)–Yb–C(13)	67.3(2)
N(2)–Yb–Cl	105.9(1)	C(13)–N(1)–Yb	119.3(4)
N(1)–Yb–C(15)	67.5(2)	C(15)–N(2)–Yb	120.0(4)
N(2)–Yb–C(15)	21.6(2)	C(13)–C(14)–C(15)	130.9(6)

$(CH_3C_5H_4)(L)YbCl$, but apparently shorter than that in $(CH_3C_5H_4)(L)YbNPh_2$ [35]. This may be caused by the increased steric congestion due to the coordination of a bulky diphenylamido group in the latter. The Yb–C(ring) distances range from 2.567(8) to 2.678(6) Å, giving the average Yb–C(ring) distance of 2.617(8) Å, which is comparable with those in $[(C_9H_7)_2PrCl(THF)]_2$ and $(C_9H_7)_3La(THF)$ [38] when the difference in ionic radii was considered. The N(1)–Yb–N(2) bond angle of 83.5(2)° is comparable with the corresponding values in $LScCl_2(THF)$ [33] and $(CH_3C_5H_4)(L)YbCl$ [35].

3.3. Molecular structure of (ArO)(L)YbCl(THF) (4)

The structure of complex **4** is shown in Fig. 2, and selected bond distances and angles are listed in Table 3.

Table 3
Selected bond lengths (Å) and angles (°) for complex **4**

Bond lengths			
Yb–N(1)	2.338(2)	Yb–C(15)	3.306(3)
Yb–N(2)	2.340(2)	N(1)–C(13)	1.340(3)
Yb–O(1)	2.024(2)	N(2)–C(15)	1.323(3)
Yb–O(2)	2.397(2)	C(13)–C(14)	1.390(4)
Yb–Cl	2.4959(8)	C(14)–C(15)	1.403(4)
Yb–C(13)	3.303(3)	O(1)–C(30)	1.347(3)
Yb–C(14)	3.592(3)		
Bond angles			
N(1)–Yb–N(2)	80.86(7)	O(1)–Yb–Cl	105.09(5)
O(1)–Yb–N(1)	141.59(7)	O(2)–Yb–Cl	83.73(6)
O(1)–Yb–N(2)	98.73(7)	N(1)–Yb–Cl	112.48(6)
O(1)–Yb–O(2)	85.24(7)	N(2)–Yb–Cl	102.89(6)
N(1)–Yb–O(2)	90.99(7)	Yb–O(1)–C(30)	166.7(2)
N(2)–Yb–O(2)	170.98(7)	C(13)–C(14)–C(15)	130.5(3)

Complex **4** is a THF-solvated monomer, the coordinated THF molecule is disordered due to strong thermal motion. The central ytterbium atom is five-coordinated with two nitrogen atoms from the β-diketiminate ligand, one oxygen atom from aryloxide, one oxygen atom from THF, and one chlorine atom in a distorted trigonal bipyramid geometry. One nitrogen atom of the β-diketiminate ligand (N(1)), the oxygen atom of aryloxide, and the chlorine atom form the equatorial vertices (the sum of these bond angles is 359.2°), another nitrogen atom (N(2)) and the THF ligand occupy the apical positions ($\angle N(2)–Yb–O(2) = 170.98(7)°$; see Table 3). The coordination geometry of this complex is similar to that of $(ArO)_2YbCl(THF)_2$ [42].

The β-diketiminate ligand is also symmetrically coordinated to the ytterbium atom (2.338(2) and 2.340(2) Å, respectively). However, the average Yb–N distance

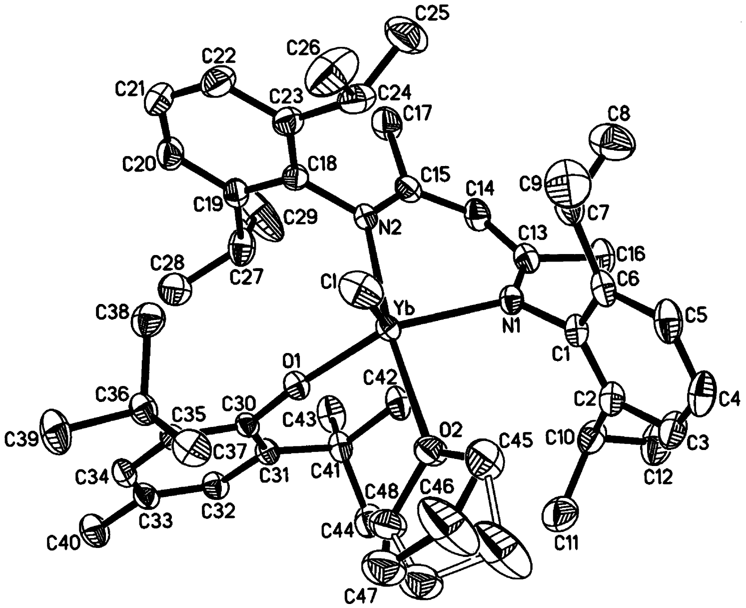


Fig. 2. Molecular structure of complex **4** (hydrogen atoms omitted for clarity). Each disordered THF ligand is shown in two possible orientations.

of 2.339(2) Å is much longer than that in complex **3** (2.241(4) Å), taking into account the difference in coordination number between these two complexes. This can be attributed to the steric congestion around ytterbium due to the bulky substituents on the arene ring of β-diketiminate ligand and aryloxide. The long distances of Yb–C(13, 14, 15) indicate that there is only purely σ-bonding, and the β-diketiminate ligand is coordinated to the ytterbium atom in an η^2 manner. There is expected electron delocalization within the $YbNC_3N$ six-membered ring, which is consistent with the IR results. The Yb–O(Ar) bond length is 2.024(2) Å. Subtraction of the estimated ionic radius (0.81 Å) for five-coordinate Yb^{3+} from this value gives 1.21 Å, which is comparable with that in $(ArO)YbCl_2(THF)_3$ (1.21 Å) [43], but apparently shorter than that in $(ArO)_2YbCl(THF)_2$ (1.27 Å) [42]. The Yb–Cl distance is 2.4959(8) Å. This value is comparable with those in $(ArO)_2YbCl(THF)_2$ (2.4770(9) Å) [42] and $(ArO)YbCl_2(THF)_3$ (2.555(2)) [43] when the difference in coordination number is considered. The C–O distance of the phenolate ligand is 1.347(3) Å, which is apparently shorter than the single bond length, reflecting substantial electron delocalization from oxygen into the aromatic rings.

The N(1)–Yb–N(2) bond angle is 80.86(7)°, which is comparable with the corresponding values in complexes **3**, $LScCl_2(THF)$ [33] and $(CH_3C_5H_4)(L)YbCl$ [35]. The Yb–O(1)–C(30) angle is 166.7(2)°, which is smaller than the corresponding values in $(ArO)_2YbCl(THF)_2$ (172.5(1)°) [42], and $(ArO)YbCl(THF)_3$ (179.7(3)°) [43]. This may be attributed to the difference in steric crowding around the metal among these compounds.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Center, CCDC No. 183737 for complex **3** and 183738 for complex **4**. 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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