Di-*tert*-butylcyclopentadienyl complexes of europium and ytterbium: synthesis and crystal structures of $(1,3\text{-}Bu^t_2C_5H_3)_2Eu \cdot THF$, $(1,3\text{-}Bu^t_2C_5H_3)(C_5Me_5)Yb \cdot THF$, and $(1,3\text{-}Bu^t_2C_5H_3)_2Yb(\mu_2\text{-}Cl)_2Li(THF)_2$

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The $(1,3\text{-}Bu^t_2C_5H_3)_2Eu \cdot THF$ complex was prepared by the reaction of $1,3\text{-}Bu^t_2C_5H_3Na$ with EuI_2 in tetrahydrofuran. The mixed-ligand $(1,3\text{-}Bu^t_2C_5H_3)(C_5Me_5)Yb \cdot THF$ complex was obtained by the reaction of YbI_2 with C_5Me_5Na and $1,3\text{-}Bu^t_2C_5H_3Na$. The reaction of $1,3\text{-}Bu^t_2C_5H_3Li$ with $YbCl_3$ afforded the $(1,3\text{-}Bu^t_2C_5H_3)_2Yb(\mu_2\text{-}Cl)_2Li(THF)_2$ *ate*-complex. The structures of the title compounds were established by X-ray diffraction analysis.

Key words: europium(+2), ytterbium, metallocenes, crystal structure.

In recent years, many studies have been devoted to cyclopentadienyl lanthanide complexes.[1] Primary attention has been given to lanthanidocenes with cyclopentadienyl rings containing various substituents, whose sizes and number allow one to vary both the geometry of the ligand environment about the metal atom and the properties of the resulting compounds over wide ranges.[2]

The aim of this work is to synthesize metallocene complexes of europium(+2), ytterbium(+2), and ytterbium(+3) using the bulky di-*tert*-butylcyclopentadienyl and pentamethylcyclopentadienyl ligands.

Results and Discussion

Divalent lanthanide iodides, which are the starting compounds in the synthesis of the majority of metallocene complexes of the corresponding elements in the oxidation state +2, are generally prepared by the reactions of the corresponding metal with 1,2-diiodoethane.[3] However, the synthesis of SmI_2 and YbI_2 was performed with the use of elemental iodine as an oxidizing agent.[4,5] We also used an analogous procedure for preparing EuI_2. Iodide solvate crystallized from a solution contains a variable amount of THF. Hence, this reagent is more convenient to use as a solution after determination of the metal content in this solution (if required, the solvating solvent can be completely removed *in vacuo* on heating).

The reaction of a solution of EuI_2 in THF with the sodium salt of the di-*tert*-butylcyclopentadienyl anion, which was carried out according to a standard procedure, proceeded without complications to give a monosolvate of composition $(1,3\text{-}Bu^t_2C_5H_3)_2Eu \cdot THF$ (**1**) in ~70% yield.

The structure of complex **1**, which was established by X-ray diffraction analysis, is typical of these compounds (Fig. 1, Table 1). The average Eu—C distance (2.811(9) Å) corresponds to the distance in the known europocenes(+2) (2.79(1) Å in $(C_5Me_5)_2Eu$,[6] 2.80 Å in $(C_5Me_5)_2Eu \cdot OEt_2$,[7] and 2.828(6) Å in $\{(C_5H_3(SiMe_3)_2)_2Eu\}_\infty$[8]), but this distance is smaller than that in the complex containing the bulkier diphenylphosphine substituents and the tridentate solvating ligand, *viz.*, in $(C_5H_4PPh_2)_2Eu \cdot DIME$, where DIME is dimethyl ether of diethylene glycol, (2.89(2) Å).[9] The Cp(1)—Eu—Cp(2) angle in complex **1** (131.9°) is in the range of the values found for europium complexes (140.3° in $(C_5Me_5)_2Eu$,[6] 124.8° in $(C_5H_4PPh_2)_2Eu \cdot DIME$,[9] and 147° and 122° in two independent molecules of $\{(C_5H_3(SiMe_3)_2)_2Eu\}_\infty$[8]). The Eu—O distance (2.545(7) Å) in complex **1** is smaller than those in $(C_5H_4PPh_2)_2Eu \cdot DIME$ (2.60(1), 2.63(1), and 2.63(1) Å).[9] The oxygen atom deviates from the Cp(1)—Eu—Cp(2) plane by 0.09 Å.

A comparison of the geometry of complex **1** with that of the $(1,3\text{-}Bu^t_2C_5H_3)_2Yb \cdot Et_2O$ complex[5] demonstrates that the M—Cp and M—O distances increase (from 2.42(2) to 2.55(1) Å and from 2.430(14) to 2.545(7) Å, respectively) as the radius of lanthanide(+2) increases on going from the Yb atom to the Eu atom,

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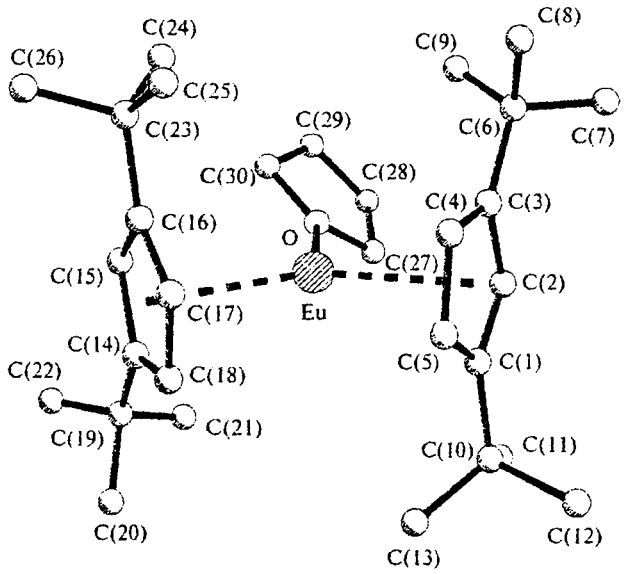

Fig. 1. Molecular structure of the $(1,3\text{-}Bu^t_2C_5H_3)_2Eu \cdot THF$ complex (**1**).

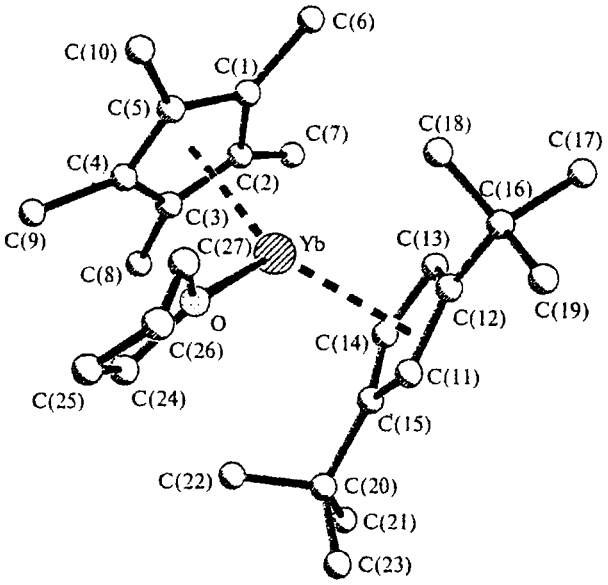

Fig. 2. Molecular structure of the $(1,3\text{-}Bu^t_2C_5H_3)(C_5Me_5)Yb \cdot THF$ (**2**) complex.

Table 1. Principal distances (d) and bond angles (ω) in the $(1,3\text{-}Bu^t_2C_5H_3)_2Eu \cdot THF$ complex (**1**)

Distance	d/Å	Angle	ω/deg
Eu—C_{aver}	2.811(9)	Cp(1)—Eu—Cp(2)	131.9(7)
Eu—O	2.545(7)	Cp(1)/Cp(2)	52.3(9)
Eu—Cp	2.55(1)		

Table 2. Principal distances (d) and bond angles (ω) in the $(1,3\text{-}Bu^t_2C_5H_3)(C_5Me_5)Yb \cdot THF$ complex (**2**)

Distance	d/Å	Angle	ω/deg
Yb—C_{aver}	2.657(14)	Cp(1)—Yb—Cp(2)	135.5(14)
Yb—O	2.447(13)	Cp(1)/Cp(2)	45.8(13)
Yb—Cp(1)	2.373(13)		
Yb—Cp(2)	2.386(14)		

which leads to a decrease in the shielding of the metal atom, while the Cp(1)—M—Cp(2) angles remain virtually unchanged (133.0(12) and 131.9(7)°, respectively). However, the accessibility of the metal atom is still insufficient for coordination of the second solvent molecule, as is generally observed for the metallocene complexes of lanthanides with nonsubstituted rings.

With the aim of preparing a complex with lower steric crowding of the coordination sphere of the Yb atom but with the M—Cp bond length intermediate between those of $(1,3\text{-}Bu^t_2C_5H_3)_2Yb$ (Yb—Cp = 2.42(2) Å in the $(1,3\text{-}Bu^t_2C_5H_3)_2Yb \cdot Et_2O$ complex[5]) and of $(C_5Me_5)_2Yb$ (Yb—Cp = 2.37(1) Å in the $(C_5Me_5)_2Yb \cdot THF$ complex[21]), we performed a synthesis of the $(1,3\text{-}Bu^t_2C_5H_3)(C_5Me_5)Yb \cdot THF$ complex (**2**), by the reaction of ytterbium(+2) iodide with sodium salts of the corresponding ligands taken in a ratio of 1 : 1 : 1.

In this case, rapid crystallization afforded separately red and green crystals of the bis(di-*tert*-butylcyclopentadienyl) and bis(pentamethylcyclopentadienyl) ytterbium complexes, respectively. Taking into account this fact as well as the fact that the bond in lanthanidocenes is to a large degree ionic, it can be suggested that the following equilibrium exists in the solution:

$$2(1,3\text{-}Bu^t_2C_5H_3)(C_5Me_5)Yb \cdot THF \rightleftharpoons$$

$$\rightleftharpoons (1,3\text{-}Bu^t_2C_5H_3)_2Yb \cdot THF + (C_5Me_5)_2Yb \cdot THF.$$

However, slow crystallization gave black crystals of mixed-ligand complex **2** in high yield.

In the crystal structure of this compound (Fig. 2, Table 2), the ytterbium atom deviates from the Cp(1)—O—Cp(2) plane by 0.03 Å. The angles between this plane and the planes of the Cp(1) and Cp(2) cyclopentadienyl rings are 90.4° and 91.0°, respectively. The Yb—Cp and Yb—O distances and the Cp(1)—Yb—Cp(2) angles are in the ranges typical of ytterbocenes(+2). A comparison of the structural parameters of complex **2** (Yb—Cp, 2.38(2) Å; Yb—O, 2.447(13) Å; Cp(1)—Yb—Cp(2), 135.5(14)°) and $(1,3\text{-}Bu^t_2C_5H_3)_2Yb \cdot Et_2O$[5] (Yb—Cp, 2.42(2) Å; Yb—O, 2.430(14) Å; Cp(1)—Yb—Cp(2), 133.0(12)°) demonstrates that the replacement of one $Bu^t_2C_5H_3$ ring by C_5Me_5 (in accordance with the initial proposal) leads to a change in the principal structural

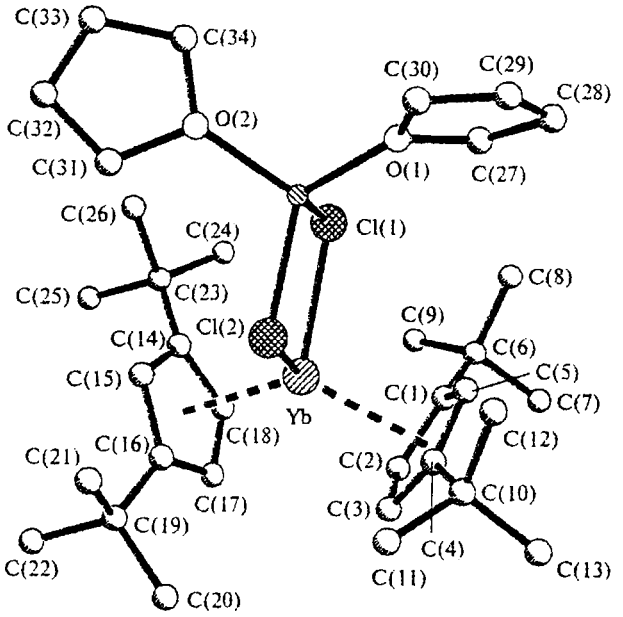

Fig. 3. Molecular structure of the $(1,3\text{-}Bu^t_2C_5H_3)_2Yb(\mu_2\text{-}Cl)_2Li(THF)_2$ (**3**) complex.

Table 3. Principal distances (d) and bond angles (ω) in the $(1,3\text{-}Bu^t_2C_5H_3)_2Yb(\mu_2\text{-}Cl)_2Li(THF)_2$ complex (**3**)

Distance	d/Å	Angle	ω/deg
Yb—C_{aver}	2.646(12)	Cl(1)—Yb—Cl(2)	82.95(12)
Yb—Cl(1)	2.608(3)	Cp(1)—Yb—Cp(2)	126.6(4)
Yb—Cl(2)	2.594(3)	Cp(1)/Cp(2)	61.6(12)
Yb—Cp(1)	2.354(12)	O(1)—Li—O(2)	108.4(13)
Yb—Cp(2)	2.360(12)	Cl(1)—Li—Cl(2)	94.7(9)
Li—O(1)	1.90(3)		
Li—O(2)	1.95(2)		
Li—Cl(1)	2.40(3)		
Li—Cl(2)	2.28(3)		

characteristics of the molecule of bulky ytterbocene. However, these changes do not correspond to the average values of the bond lengths observed in di-*tert*-butyl- and pentamethylcyclopentadienyl complexes.

In the synthesis of the trivalent ytterbium complex with the use of the lithium salt of the di-*tert*-butylcyclopentadienyl anion, the *ate*-complex (**3**) was formed (Fig. 3, Table 3). The crystal structure of the latter is typical of compounds of this type. All carbon atoms of the Cp rings are in a single plane to within 0.012 (Cp(1)) and 0.007 (Cp(2)) Å. The Yb, Cl(1), Cl(2), and Li atoms are also in a single plane (±0.005 Å). The angles between this plane and the planes of the cyclopentadienyl rings are 30.0 (Cp(1)) and 31.7° (Cp(2)). The average Yb—Cl distance (2.60 Å) falls within the upper limits of this parameter for the known ytterbium(+3) *ate*-complexes with lithium chloride (2.57 Å in *rac*-$(CH_3)_2C(C_5H_3\text{-}3\text{-}Si(CH_3)_3)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$,[10] 2.59 Å in $(Ph_2MeSiC_5H_4)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$, and 2.59 and 2.60 Å in $(C_5Me_5)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$[11]). The Cl(1)—Yb—Cl(2) angle (82.95(12)°) is smaller that those in the *rac*-$(CH_3)_2C(C_5H_3\text{-}3\text{-}Si(CH_3)_3)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$ (85.9°),[10] $(C_5Me_5)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$ (86.0°), and $(Ph_2MeSiC_5H_4)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$ complexes (87.1°).[11] The average Yb—C distance (2.646(12) Å) in complex **3** is among the largest values known for the structurally characterized ytterbocenes(+3), along with, probably, the $(C_5Me_5)_2YbCl \cdot THF$ complex (2.65 Å),[15] (2.59 Å in $[(Bu^tC_5H_4)_2Yb(THF)_2][BPh_4]$,[12] 2.59 Å in *rac*-$(CH_3)_2C(C_5H_3\text{-}3\text{-}Si(CH_3)_3)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$ [10] 2.60 Å in $(Me_4C_2(C_5H_4)_2YbCl_2)^-(Mg_2Cl_3 \cdot 6THF)^+ \cdot 2THF$,[13] 2.61 Å in $(C_5Me_5)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$, 2.63 Å in $(Ph_2MeSiC_5H_4)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$,[11] and 2.63 Å in $(Bu^tC_5H_4)_2YbCl \cdot THF$[14]). The angular characteristics of the metallocene fragment (Cp(1)/Cp(2) 61.6(12)°) are intermediate between those found in the *ate*-complexes of *ansa*-ytterbocenes(+3) (74.0° in

Table 4. Crystallographic characteristics of complexes **1**, **2**, and **3** and details of X-ray diffraction studies

Parameter	**1**	**2**	**3**
Lattice		Monoclinic	
Space group	$P2_1/n$	$P2_1/c$	$P2_1/n$
a/Å	11.440(2)	9.026(2)	10.641(2)
b/Å	17.682(4)	15.014(3)	20.986(4)
c/Å	15.285(3)	20.020(4)	16.943(3)
β/deg	92.99(3)	101.67(3)	94.41(3)
V/Å^3	3087.7(11)	2657.0(10)	3772.4(12)
d_{calc}/g cm^{-3}	1.245	1.394	1.320
Z	4	4	6
μ/mm^{-1}	2.048	3.533	2.646
Crystal dimensions/mm	0.24×0.20×0.15	0.29×0.25×0.12	0.35×0.21×0.20
Scanning range θ/deg	1.76—22.54	2.30—25.05	2.15—24.04
Number of reflections	2542	2179	2720
R [$I > 2\sigma(I)$]			
R_1	0.0381	0.0476	0.0453
wR_2	0.0963	0.1220	0.1117

Table 5. Atomic coordinates ($\times 10^4$) and temperature factors ($\times 10^3/Å^2$) in the molecule of complex **1**

Atom	*x*	*y*	*z*	U_{eq}
Eu	6258(1)	7331(1)	6657(1)	58(1)
O	6388(6)	7832(4)	8220(4)	90(2)
C(1)	4305(8)	6367(5)	6363(6)	59(2)
C(2)	3844(7)	6967(7)	6800(6)	65(3)
C(3)	3844(8)	7628(7)	6273(8)	75(3)
C(4)	4337(9)	7408(7)	5482(7)	80(3)
C(5)	4607(8)	6636(7)	5539(6)	69(3)
C(6)	3340(10)	8383(7)	6519(12)	110(5)
C(7)	2163(23)	8317(12)	6693(31)	357(25)
C(8)	3114(46)	8840(16)	5777(21)	385(28)
C(9)	4024(19)	8854(13)	7013(25)	349(23)
C(10)	4368(9)	5550(6)	6667(7)	79(3)
C(11)	4295(19)	5485(8)	7614(10)	178(8)
C(12)	3351(12)	5120(8)	6194(10)	134(5)
C(13)	5449(13)	5157(7)	6387(10)	130(5)
C(14)	8628(8)	6884(8)	6500(7)	73(3)
C(15)	8646(8)	7682(8)	6495(6)	70(3)
C(16)	8065(7)	7941(6)	5706(6)	58(2)
C(17)	7693(8)	7290(7)	5254(6)	66(2)
C(18)	8016(9)	6641(7)	5724(8)	79(3)
C(19)	9194(10)	6408(9)	7204(10)	113(5)
C(20)	9399(41)	5683(21)	6905(24)	447(29)
C(21)	8553(21)	6276(18)	7915(14)	281(17)
C(22)	10239(20)	6623(22)	7481(26)	469(33)
C(23)	7964(9)	8754(7)	5435(7)	78(3)
C(24)	7581(24)	9249(9)	6087(14)	259(14)
C(25)	7213(17)	8839(9)	4624(13)	192(9)
C(26)	9088(12)	9069(8)	5176(12)	155(7)
C(27)	5775(14)	7509(8)	8913(9)	124(5)
C(28)	5840(17)	8038(14)	9631(10)	172(7)
C(29)	6483(18)	8649(10)	9372(12)	157(7)
C(30)	7007(12)	8451(9)	8579(9)	119(4)

Table 6. Atomic coordinates ($\times 10^4$) and temperature factors ($\times 10^3/Å^2$) in the molecule of complex **2**

Atom	*x*	*y*	*z*	U_{eq}
Yb	6225(1)	2219(1)	3737(1)	51(1)
C(1)	5250(22)	1413(12)	2542(7)	76(5)
C(2)	6135(18)	760(11)	2970(10)	78(5)
C(3)	5412(18)	550(9)	3483(7)	61(4)
C(4)	4076(18)	1014(11)	3389(8)	69(4)
C(5)	3970(18)	1537(11)	2834(9)	73(5)
C(6)	5523(41)	1754(19)	1891(10)	200(15)
C(7)	7498(25)	301(19)	2814(14)	164(11)
C(8)	5934(33)	-198(15)	3973(12)	172(12)
C(9)	2863(23)	867(14)	3816(11)	119(7)
C(10)	2630(27)	2110(13)	2531(14)	150(11)
C(11)	8424(13)	3297(10)	4399(6)	47(3)
C(12)	8501(15)	3330(9)	3723(7)	56(4)
C(13)	9004(16)	2484(10)	3544(7)	59(4)
C(14)	9156(15)	1932(10)	4127(8)	63(4)
C(15)	8851(17)	2428(10)	4652(8)	62(4)
C(16)	8119(21)	4094(11)	3228(9)	85(5)
C(17)	9126(31)	4161(21)	2714(15)	194(14)
C(18)	6576(26)	4033(14)	2851(13)	149(10)
C(19)	8249(42)	4949(15)	3584(13)	200(15)
C(20)	8932(22)	2126(13)	5382(8)	88(6)
C(21)	10175(56)	1586(40)	5632(15)	390(40)
C(22)	7663(54)	1628(27)	5447(10)	298(29)
C(23)	8998(29)	2852(18)	5895(10)	146(10)
O	4440(15)	3100(12)	4235(8)	114(5)
C(24)	3982(69)	2877(36)	4801(32)	304(26)
C(25)	2798(48)	3639(33)	4839(21)	227(17)
C(26)	3047(54)	4269(33)	4454(27)	260(20)
C(27)	3558(97)	3646(70)	4014(45)	475(59)

rac-$(CH_3)_2C(C_5H_3\text{-}3\text{-}Si(CH_3)_3)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$[10] and in the *ate*-complexes with nonbonded rings, *viz.*, in $(C_5Me_5)_2Yb(\mu\text{-}Cl)_2AlCl_2$, $(C_5Me_5)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$, and $(Ph_2MeSiC_5H_4)_2Yb(\mu\text{-}Cl)_2Li(OEt_2)_2$ (42.7—49.6°).[11]

Hence, the replacement of the C_5Me_5 ligands by the $Bu^t_2C_5H_3$ ligands in the series of the Eu(+2), Yb(+2), and Yb(+3) complexes leads to changes in the geometric characteristics of the resulting compounds. However, these changes are of no fundamental importance and the structural parameters are in the ranges observed in analogous compounds.

Experimental

All operations were carried out under an atmosphere of dry inert gas or *in vacuo*. Before use, the solvents were dried by refluxing and distilling over $LiAlH_4$. The $1,3\text{-}Bu^t_2C_5H_3Na$,[16] $1,3\text{-}Bu^t_2C_5H_3Li$,[17] $YbCl_3$,[18] and YbI_2 compounds[5] were prepared according to procedures reported previously and C_5Me_5Na was used as a 0.5 *M* solution in THF (Aldrich).

Europium diiodide was synthesized by the reaction of metallic europium (in pieces, 2.9 g, 18.9 mmol) and iodine (3.2 g, 12.6 mmol) in THF (150 mL) with stirring for 48 h. The resulting transparent solution exhibiting blue fluorescence was decanted from excess metal and the solvent was distilled off *in vacuo*. The resulting powder of the solvate $EuI_2 \cdot THF_x$ was dried *in vacuo* (10^{-2} Torr) with a gradual increase in the temperature from 20 °C to 180 °C. The pale-yellow powder was obtained in a yield of 4.8 g (94%). Found (%): Eu, 37.4; I, 62.6. EuI_2. Calculated (%): Eu, 37.5; I, 62.5.

Synthesis of bis(η^5-1,3-di-*tert*-butylcyclopentadienyl)europium(II) monotetrahydrofuranate (1). $1,3\text{-}Bu^t_2C_5H_3Na$ (1.3 g, 6.4 mmol) was added to a suspension of EuI_2 (1.3 g, 3.2 mmol) in THF (100 mL). The red solution that formed was stirred for 2 h. Then the solvent was distilled off *in vacuo* and the dry residue was extracted with toluene (100 mL). Evaporation of the resulting solution afforded red crystals of $(1,3\text{-}Bu^t_2C_5H_3)_2Eu \cdot THF$ in a yield of 1.3 g (71%). Found (%): Eu, 26.2. $C_{30}H_{50}EuO$. Calculated (%): Eu, 26.3.

Synthesis of (η^5-1,3-di-*tert*-butylcyclopentadienyl)(η^5-pentamethylcyclopentadienyl)ytterbium(II) monotetrahydrofuranate (2). A 0.5 *M* C_5Me_5Na solution in THF (7.0 mL, 3.5 mmol) was added with stirring to a yellow suspension of YbI_2 (1.5 g, 3.5 mmol) in THF (100 mL). Then $1,3\text{-}Bu^t_2C_5H_3Na$ (0.7 g, 3.5 mmol) was added to the resulting red solution. The reaction mixture was stirred for 1 h, the black solution that formed was concentrated to dryness *in vacuo*, and the solid residue was extracted with toluene (150 mL). Slow crystallization gave black crystals of $(1,3\text{-}Bu^t_2C_5H_3)(C_5Me_5)Yb \cdot THF$ in a yield of 1.6 g (83%). Found (%): Yb, 31.0. $C_{27}H_{44}OYb$. Calculated (%): Yb, 31.0.

Synthesis of lithium bis(η^5-1,3-di-*tert*-butylcyclopentadienyl)dichlorobis(tetrahydrofuran)ytterbate(III) (3). $1,3\text{-}Bu^t_2C_5H_3Li$ (2.2 g, 12.2 mmol) was added with stirring to

Table 7. Atomic coordinates ($\times 10^4$) and temperature factors ($\times 10^3/Å^2$) in the molecule of complex **3**

Atom	*x*	*y*	*z*	U_{eq}
Yb	1845(1)	5071(1)	2064(1)	55(1)
Cl(1)	417(3)	5139(2)	3246(2)	72(1)
Cl(2)	3510(4)	4678(2)	3155(2)	86(1)
C(1)	1205(11)	6281(5)	1744(8)	54(3)
C(2)	1708(14)	5999(5)	1079(7)	57(4)
C(3)	3016(14)	5875(6)	1219(9)	64(4)
C(4)	3365(14)	6094(6)	1986(9)	72(4)
C(5)	2285(14)	6320(6)	2332(8)	66(4)
C(6)	−69(13)	6581(5)	1773(9)	69(4)
C(7)	−50(16)	7208(7)	1304(10)	118(6)
C(8)	−350(15)	6746(7)	2622(11)	111(6)
C(9)	−1122(14)	6157(7)	1400(11)	105(6)
C(10)	4717(15)	6210(7)	2334(13)	105(6)
C(11)	5597(15)	5670(9)	2139(12)	125(6)
C(12)	4783(17)	6276(9)	3238(13)	136(7)
C(13)	5137(18)	6808(8)	1981(16)	174(10)
C(14)	301(13)	4105(6)	1575(8)	61(4)
C(15)	1504(17)	3823(6)	1703(9)	73(4)
C(16)	2363(14)	4056(6)	1184(9)	66(4)
C(17)	1661(14)	4504(5)	709(6)	56(3)
C(18)	440(13)	4546(5)	949(7)	54(3)
C(19)	3632(15)	3754(7)	1046(9)	81(4)
C(20)	4470(18)	4202(10)	631(15)	156(9)
C(21)	4338(18)	3524(9)	1787(11)	126(7)
C(22)	3355(18)	3168(10)	515(12)	157(9)
C(23)	−907(14)	3881(6)	1834(9)	78(4)
C(24)	−1948(13)	4412(7)	1839(9)	93(5)
C(25)	−1428(16)	3329(7)	1275(10)	108(5)
C(26)	−768(15)	3589(8)	2680(10)	102(5)
Li	2150(3)	4772(11)	4124(15)	97(9)
O(1)	2496(13)	5388(6)	4934(7)	121(4)
O(2)	2009(12)	3942(5)	4619(7)	112(4)
C(27)	2130(3)	6003(15)	4902(17)	270(2)
C(28)	2760(3)	6402(11)	5413(19)	204(13)
C(29)	3560(3)	5985(17)	5820(2)	226(16)
C(30)	3310(3)	5404(11)	5574(15)	230(17)
C(31)	2380(2)	3362(10)	4282(14)	167(10)
C(32)	2020(3)	2853(10)	4730(16)	204(14)
C(33)	1520(3)	3111(11)	5348(18)	224(16)
C(34)	1610(3)	3770(12)	5317(14)	176(10)

a white suspension of $YbCl_3$ (1.7 g, 6.1 mmol) in THF (100 mL). The reaction mixture was stirred for 1 h. Then the red solution that formed was concentrated to dryness *in vacuo* and the solid residue was extracted with toluene (150 mL). The solvent was removed and complex **3** was obtained in a yield of 4.0 g (87%). Found (%): Cl, 9.5; Li, 0.9; Yb, 23.1. $C_{34}H_{58}Cl_2LiO_2Yb$. Calculated (%): Cl, 9.46; Li, 0.93; Yb, 23.08.

X-ray diffraction studies of single crystals of compounds **1**, **2**, and **3** sealed in glass tubes were performed on an automated Syntex $P\bar{1}$ diffractometer (293(2) K, λ Mo-Kα radiation, β filter, θ/2θ scanning technique). The principal crystallographic characteristics of the complexes are given in Table 4. The structures were solved by the direct method with the use of the SHELXTL-81[19] and SHELXL-93[20] program packages and refined by the full-matrix least squares method with anisotropic thermal parameters for the heavy atoms. Absorption was ignored. The coordinates of the hydrogen atoms were calculated from geometric considerations and were not refined. The positional and thermal parameters of the atoms are given in Tables 5—7. The principal bond lengths and bond angles are listed in Tables 1—3.

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