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Ytterbium(+ 2) indenyl complexes: synthesis and crystal structures of $(C_9H_7)_2Yb{\cdot}DME$, *rac*-$(CH_2)_2(1\text{-}C_9H_6)_2Yb(THF)_2$, and *rac*-$(CH_2)_2[1\text{-}(4,7\text{-}(CH_3)_2C_9H_4)]Yb(THF)_2$

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Abstract

Metallation of 2,2-bis(1′-indene)propane by sodium hydride followed by the reaction of the sodium derivative with $YbCl_3$ yields $(C_9H_7)_2Yb{\cdot}DME$ (**1**), an ytterbium(+ 2) complex with unlinked indenyl ligands. The reaction between $(CH_2)_2(1\text{-Ind}')_2Li_2$ and $YbCl_3$ in diethyl ether with subsequent reduction of the product by sodium metal in THF gives *rac*-$(CH_2)_2(1\text{-Ind}')_2Yb(THF)_2$, where $\text{Ind}' = C_9H_6$ (**2**) and 4,7-$(CH_3)_2C_9H_4$ (**3**). The structures of the complexes were determined by X-ray structural analysis. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Crystal structure; Indenyl; *ansa*-Metallocenes; Ytterbium

1. Introduction

In recent years, indene-derived ligands have found wide application in organometallic chemistry. For example, Group IV *ansa*-indenyl complexes with various bridging groups and substituents were used to prepare effective catalysts for the stereospecific polymerisation of α-olefins [1]. Among rare-earth indenyl complexes, metal(+ 3) tris–complexes of compositions $Ind_3M{\cdot}L$ ($L = (R)$-(+)-methyl-*p*-tolylsulfoxide and triphenylphosphinoxide, $M = La$ [2]; $L = NC_5H_5$, $M = Ce$ [3]; $L = THF$, $M = Pr$ [4], Nd [5], Gd [5]) and $[Na(THF)_6][Ind_3M(\mu\text{-}Cl)MInd_3]$ ($M = Pr$, Nd, Sm [6–8]) are best studied. Also reported is the hydride $[(2,4,7\text{-}Me_3Ind)_2Y(\mu\text{-}H)]_2{\cdot}1/2C_6H_6$ [9].

Lanthanide(+ 2) indenyl complexes are represented by the bis-complexes of samarium ($Ind_2Sm(THF)_x$ [10]) and ytterbium ($Ind_2Yb(THF)_2$ [11]). $Ind_2Sm(THF)_x$ and the well-known complex $(C_5Me_5)_2Sm(THF)_2$ [12] react differently with various substrates largely because the coordination sphere of the indenyl complex is sterically less crowded [13]. It is reasonable to expect that binding indenyl ligands by short carbon or silicon bridges will reduce the screening effect, that is, make the lanthanide atom more easily accessible.

In this publication, we report the synthesis and structures of bis(η^5-indenyl)(1,2-dimethoxyethane)ytterbium(+ 2), [μ-ethylenebis(η^5-indene-1-yl)]bis(tetrahydrofuran)ytterbium(+ 2), and [μ-ethylenebis(η^5-4,7-dimethylindene-1-yl]bis(tetrahydrofuran)ytterbium(+ 2).

2. Experimental

All manipulations involved in the pretreatment of the starting reagents and synthesis and physicochemical studies of reaction products were performed in flowing argon or vacuum with the use of Schlenk techniques. Solvents were refluxed over $LiAlH_4$ and then distilled. $(CH_3)_2C(1\text{-}C_9H_7)_2$, $(CH_2)_2(1\text{-}C_9H_7)_2$, $(CH_2)_2[1\text{-}(4,7)\text{-}(CH_3)C_9H_5)]_2$, and $YbCl_3$ were synthesised by published procedures [14–17].

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2.1. $(C_9H_7)_2Yb{\cdot}DME$ (**1**)

To a solution of $(CH_3)_2C(1\text{-}C_9H_7)_2$ (3.50 g, 12.85 mmol) in 250 ml of 1,2-dimethoxyethane, 1.25 g (52.08 mmol) of NaH was added. The resulting suspension was stirred until gas evolution stopped. The solution was decanted and added to 3.62 g (12.96 mmol) of $YbCl_3$ to obtain a suspension in a blue solution. After stirring this suspension for 12 h, the solvent was removed in vacuum, and the product was extracted with 150 ml of benzene. Recrystallisation from diethyl ether afforded 2.14 g of **1** as red–brown crystals. Found (%): C, 53.60; H, 5.07; Yb, 35.2. Anal. Calc. for $C_{22}H_{24}O_2Yb$ (%): C, 53.55; H, 4.90; Yb, 35.07.

2.2. *rac*-$(CH_2)_2(1\text{-}C_9H_6)_2Yb(THF)_2$ (**2**)

To a stirred and cooled (−10°C) solution of $(CH_2)_2(1\text{-}C_9H_7)_2$ (1.03 g, 3.99 mmol) in a mixture of diethyl ether (70 ml) and toluene (30 ml), 12 ml of 0.75 M ^{n}BuLi in hexane was added dropwise. A suspension formed, which was warmed to room temperature and stirred for 2 h. The resulting precipitate was filtered off and dried in vacuum. This afforded 0.74 g (2.74 mmol) of $(CH_2)_2(1\text{-}C_9H_6)_2Li_2$, which was added to a suspension of $YbCl_3$ (0.80 g, 2.86 mmol) in 100 ml of diethyl ether. The resulting mixture of a bluish green solution and a white precipitate was stirred for 24 h. Next, the solution was filtered and evaporated, and the residual solid was dissolved in 50 ml of THF and, after addition of highly dispersed sodium (0.2 g, 8.70 mmol), stirred for a day. The resultant dark red solution was filtered and evaporated until crystallisation commenced. Red crystals of **2** (0.34 g) were thus obtained. Yield: 15%. Found (%): C, 58.69; H, 5.65; Yb, 30.2. Anal. Calc. for $C_{28}H_{32}O_2Yb$ (%): C, 58.63; H, 5.62; Yb, 30.17.

2.3. *rac*-$(CH_2)_2[1\text{-}(4,7\text{-}(CH_3)_2C_9H_4)]_2Yb(THF)_2$ (**3**)

Complex **3** was synthesised similarly to complex **2**. However, the synthesis had poor reproducibility, and we were unable to obtain satisfactory analytical data.

2.4. X-ray structure determination

The details of unit cell determination, data collection, and structure refinement are given in Table 1. Experimental data were processed using the PROFIT procedure [18]. An absorption correction was not applied. The structure was solved by heavy-atom methods and refined using the anisotropic full-matrix least squares. Because scattering from the single crystal of **3** was weak, only one fifth of 4000 measured reflections were 'observable'. Therefore, only the Yb and O atoms were refined anisotropically. H atoms were located from geometric considerations and not refined. The structure

Table 1
Crystal data and structure refinement for $(C_9H_7)_2Yb{\cdot}DME$ (**1**), *rac*-$(CH_2)_2(C_9H_6)_2Yb(THF)_2$ (**2**) and *rac*-$(CH_2)_2[1\text{-}(4,7\text{-}(CH_3)_2C_9H_4)]_2Yb(THF)_2$ (**3**)

Compound	**1**	**2**	**3**
Crystal system	Orthorhombic	Triclinic	Orthorhombic
Space group	$C222_1$	$P\bar{1}$	*Pcab*
Unit cell dimensions			
a (Å)	11.091(2)	8.361(2)	15.817(3)
b (Å)	13.561(3)	16.442(3)	21.631(4)
c (Å)	13.091(3)	19.121(4)	16.910(3)
α (°)		67.58(3)	
β (°)		83.54(3)	
γ (°)		89.58(3)	
V (Å^3)	1969.0(7)	2412.7(9)	5785.5(18)
$D_{calc.}$ (Mg m^{-3})	1.665	1.568	1.437
Wavelength (Å)	0.71073	0.71073	0.71073
Temperature (K)	293(2)	293(2)	293(2)
Z	4	4	8
Diffractometer	Syntex P$\bar{1}$	Syntex P$\bar{1}$	CAD-4
Collection method	$\theta/2\theta$	$\theta/2\theta$	$\theta/2\theta$
Radiation type	Mo–K$_\alpha$	Mo–K$_\alpha$	Mo–K$_\alpha$
Radiation monochromator	β-Filter	Graphite	β-Filter
Absorption coefficient (mm^{-1})	4.760	3.897	3.257
Crystal size (mm)	$0.50\times0.40\times0.35$	$0.35\times0.15\times0.13$	$0.27\times0.25\times0.15$
Theta range for data collection (°)	2.37–24.97	2.08–21.54	2.00–22.36
Reflections collected	720	2683	4277
Independent reflections	720	2682	4277
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Final R indices [$I>2\sigma(I)$]	$R_1=0.0561$, $wR_2=0.1319$	$R_1=0.0507$, $wR_2=0.1271$	$R_1=0.0470$, $wR_2=0.1162$

Table 2
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for **1** [a]

Atom	*x*	*y*	*z*	U_{eq}
Yb	10000	7656(1)	2500	71(1)
O	9150(12)	6184(12)	3248(11)	83(4)
C(1)	8464(39)	7739(34)	863(18)	141(16)
C(2)	7785(22)	7973(27)	1688(24)	119(11)
C(3)	6786(49)	7470(36)	2190(71)	237(42)
C(4)	6614(45)	6644(55)	1724(59)	195(25)
C(5)	7170(58)	6279(38)	986(31)	164(19)
C(6)	8108(43)	6651(49)	286(27)	226(30)
C(7)	9291(47)	8422(58)	708(56)	304(59)
C(8)	9066(44)	9096(30)	1405(71)	227(42)
C(9)	8191(36)	8880(34)	2076(35)	176(21)
C(10)	9764(11)	5275(13)	3063(18)	73(5)
C(11)	8493(26)	6207(28)	4220(21)	111(9)

[a] U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

solution and refinement were performed using the SHELXTL-81 [19] and SHELXL-93 [20] packages, respectively. Atomic coordinates for **1**, **2**, and **3** are presented in Tables 2–4, respectively.

3. Results and discussion

Assuming that the reactions

$$(CH_3)_2C(1\text{-}C_9H_7)_2 + 2NaH \rightarrow Na_2[(1\text{-}C_9H_6)_2C(CH_3)_2] + 2H_2 \quad (1)$$

$$Na_2[(1\text{-}C_9H_6)_2C(CH_3)_2] + YbCl_3 \rightarrow (CH_3)_2C(1\text{-}C_9H_6)_2YbCl + 2NaCl \quad (2)$$

are possible, we attempted the synthesis of a $(CH_3)_2C$-bound complex. In fact, we obtained the ytterbium (+2) indenyl complex $Ind_2Yb{\cdot}DME$ (**1**) with unbonded cyclopentadienyl ligands.

In the structure of complex **1** (Fig. 1, Table 5), $Ind_2Yb{\cdot}DME$ molecules are arranged along the 2-fold axis, parallel to the *y* axis. As in the samarium analog $Ind_2Sm(THF)_3$ [10], indenyl ligands in **1** are oriented rather unusually: their benzene rings are directed toward the oxygen atoms of DME. The Yb–Cp distance in **1** (2.43(2) Å) is equal to those in the most of the structurally characterised ytterbium(+2) bis-cyclopentadienyl complexes: $(C_5H_4(CH_2)_3C_5H_4)Yb(THF)_2$ [21]; $(C_5H_5)_2Yb(THF)_2$ [22]; $(C_5H_5)_2Yb(DME)$ [23,24]; $({}^tBu\text{-}C_5H_4)_2Yb(THF)_2$ [25]; $(1,3\text{-}(SiMe_3)_2C_5H_3)_2Yb(THF)$ [26]; $(C_5Me_4Et)_2Yb(THF)$ [27]; $(C_5H_4PPh_2)_2Yb(DME)$ [28]; $(1,2\text{-}{}^tBu_2C_5H_3)_2Yb(DME)$ [29]; and $(1,3\text{-}{}^tBu_2C_5H_3)_2Yb(OEt_2)$ [29]. The Cp–Yb–Cp′ angle in **1** (129.4(8)°) is intermediate between those in unsubstituted and long-bridge ytterbocenes(+2) ($(C_5H_5)_2Yb(DME)$, 124° [23]; $(C_5H_4(CH_2)_3C_5H_4)Yb(THF)_2$, 127°

Table 3
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters (Å$^2 \times 10^3$) for **2** [a]

Atom	*x*	*y*	*z*	U_{eq}
Yb(1)	1955(1)	1801(1)	2498(1)	85(1)
C(1)	−1060(3)	1730(2)	2180(3)	137(14)
C(2)	−1270(6)	1950(6)	2740(3)	290(5)
C(3)	−790(5)	2510(4)	2920(3)	200(2)
C(4)	−90(3)	3100(3)	1990(2)	114(9)
C(5)	450(4)	3960(3)	1520(4)	156(16)
C(6)	910(6)	4090(4)	760(4)	280(5)
C(7)	400(6)	3580(6)	410(3)	220(3)
C(8)	30(6)	2780(3)	880(4)	170(2)
C(9)	−360(4)	2550(3)	1610(3)	121(12)
C(10)	−1390(4)	950(3)	2020(3)	210(2)
C(11)	−710(5)	180(2)	2410(2)	166(13)
C(12)	950(4)	200(2)	2540(3)	137(12)
C(13)	2260(7)	440(19)	2037(19)	144(14)
C(14)	3600(7)	540(3)	2250(4)	210(3)
C(15)	3300(5)	180(3)	3010(4)	173(19)
C(16)	4210(6)	0(3)	3670(5)	260(4)
C(17)	3330(14)	−350(4)	4320(5)	280(5)
C(18)	1730(11)	−440(5)	4580(5)	290(5)
C(19)	1070(7)	−290(3)	3960(4)	189(19)
C(20)	1650(4)	51(19)	3240(2)	118(10)
O(1)	2990(2)	2143(11)	3449(10)	110(5)
C(21)	2260(3)	2340(3)	4020(3)	180(14)
C(22)	3320(6)	2290(4)	4580(2)	230(2)
C(23)	4780(6)	2250(3)	4240(3)	210(2)
C(24)	4620(4)	2200(3)	3530(2)	199(17)
O(2)	4125(17)	2715(10)	1626(10)	103(5)
C(25)	5010(3)	2607(19)	986(15)	130(10)
C(26)	5710(4)	3440(18)	462(13)	131(11)
C(27)	5690(4)	3972(18)	930(2)	155(12)
C(28)	4830(4)	3510(2)	1661(18)	172(14)
Yb(2)	4302(1)	−3208(1)	2497(1)	89(1)
C(1′)	5180(4)	−4823(18)	2610(3)	124(10)
C(2′)	4160(8)	−4520(2)	2001(18)	172(15)
C(3′)	2530(4)	−4600(3)	2490(5)	200(3)
C(4′)	2770(7)	−4870(2)	3180(3)	190(3)
C(5′)	1350(6)	−5090(4)	3780(6)	300(5)
C(6′)	2280(10)	−5330(4)	4500(3)	230(4)
C(7′)	3830(11)	−5430(4)	4420(4)	250(4)
C(8′)	4740(10)	−5210(4)	3790(7)	320(6)
C(9′)	4090(5)	−4970(3)	3310(3)	170(2)
C(10′)	6900(5)	−4910(2)	2440(3)	220(2)
C(11′)	7850(3)	−4140(3)	1960(3)	207(18)
C(12′)	7450(3)	−3260(2)	2200(3)	142(15)
C(13′)	7400(6)	−3290(4)	2880(3)	250(4)
C(14′)	6970(4)	−2440(3)	2750(2)	127(14)
C(15′)	6690(3)	−1880(2)	2010(2)	104(10)
C(16′)	6280(5)	−1070(4)	1580(5)	240(3)
C(17′)	6310(7)	−800(4)	850(3)	210(3)
C(18′)	6660(7)	−1340(5)	440(3)	250(4)
C(19′)	7020(4)	−2160(3)	840(3)	138(12)
C(20′)	7140(3)	−2540(2)	1610(2)	98(9)
O(1′)	2791(18)	−2842(10)	3464(9)	110(5)
C(21′)	3310(4)	−2610(3)	4050(2)	196(16)
C(22′)	1900(5)	−2750(3)	4588(18)	209(19)
C(23′)	600(4)	−2680(3)	4150(3)	204(19)
C(24′)	1140(4)	−2730(4)	3520(2)	240(2)
O(2′)	2606(18)	−2276(12)	1623(10)	109(5)
C(25′)	2180(4)	−2370(3)	950(2)	160(13)
C(26′)	1440(5)	−1540(3)	500(2)	170(14)
C(27′)	1250(4)	−1007(19)	950(2)	135(10)
C(28′)	1950(5)	−1500(3)	1610(2)	171(14)

[a] U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 4
Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **3** [a]

Atom	x	y	z	U_{eq}
Yb	1902(1)	1208(1)	584(1)	55(1)
O(1)	2914(18)	1917(11)	42(13)	76(8)
O(2)	2227(15)	447(12)	−399(19)	90(10)
C(1)	1610(2)	903(17)	2090(2)	68(11)
C(2)	2212(18)	1378(14)	2104(18)	51(10)
C(3)	3000(3)	1230(2)	1800(2)	86(10)
C(4)	2920(2)	624(16)	1650(2)	69(11)
C(5)	3620(3)	190(2)	1480(3)	103(15)
C(6)	3370(3)	−370(3)	1320(3)	130(19)
C(7)	2610(3)	−620(2)	1460(3)	111(16)
C(8)	1910(3)	−274(18)	1670(3)	97(12)
C(9)	2080(2)	397(15)	1850(2)	62(10)
C(10)	340(2)	1544(18)	1140(2)	67(10)
C(11)	240(2)	1086(17)	580(3)	87(11)
C(12)	420(2)	1210(2)	−201(19)	70(10)
C(13)	670(3)	1879(17)	−200(2)	77(11)
C(14)	850(2)	2312(17)	−770(2)	73(12)
C(15)	990(2)	2890(17)	−570(3)	87(11)
C(16)	940(2)	3079(17)	220(2)	74(11)
C(17)	750(3)	2692(17)	830(2)	84(13)
C(18)	576(19)	2043(14)	670(2)	55(9)
C(19)	730(3)	930(2)	2350(2)	111(17)
C(20)	150(3)	1450(2)	1990(3)	107(15)
C(21)	4460(3)	470(2)	1190(3)	119(16)
C(22)	1100(4)	−520(2)	1820(3)	150(2)
C(23)	860(3)	2050(2)	−1590(3)	129(17)
C(24)	730(3)	2900(2)	1640(3)	131(18)
C(25)	3260(3)	2440(2)	440(3)	109(15)
C(26)	3340(3)	2924(18)	−150(3)	93(14)
C(27)	3400(3)	2610(2)	−930(3)	107(16)
C(28)	3230(3)	1940(2)	−710(3)	107(15)
C(29)	1710(6)	10(4)	−690(5)	240(4)
C(30)	2090(4)	−430(2)	−1180(3)	131(18)
C(31)	2970(4)	−320(2)	−1000(3)	134(18)
C(32)	3030(3)	310(2)	−700(3)	118(15)

[a] U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

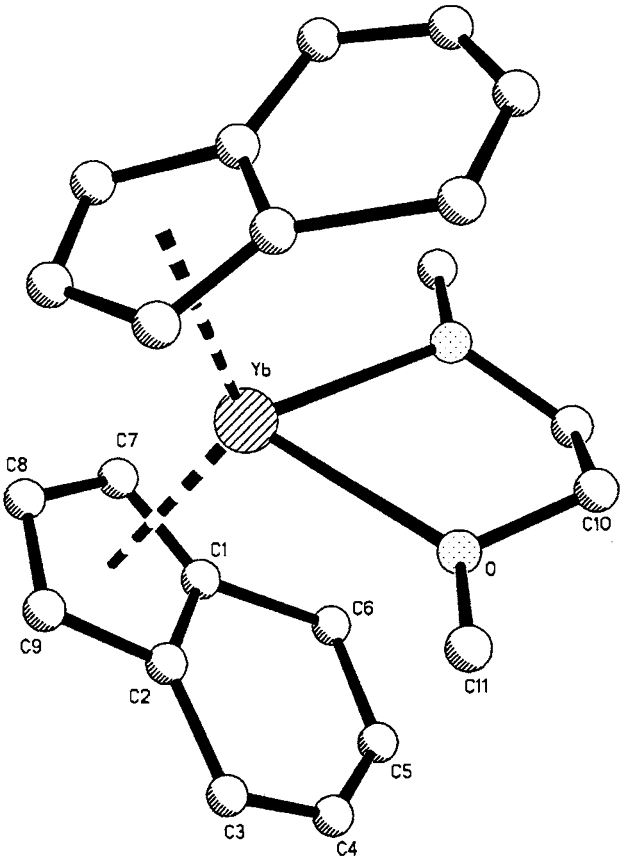
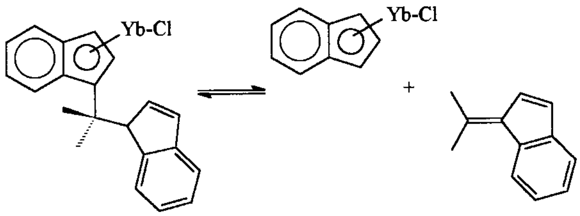


Fig. 1. Molecular structure of the complex $(C_9H_7)_2Yb{\cdot}DME$ (**1**).

[21]; $(C_5H_5)_2Yb(THF)_2$, 127.9° [22]) and those in ytterbium(+2) bis-cyclopentadienyl complexes with substituted rings (133–144° [24–32]). The Yb–O distance in **1** (2.41(2) Å) is equal to that in $(C_5H_4PPh_2)_2Yb(DME)$ [28] but shorter than those in $(C_5H_5)_2Yb(DME)$ (2.45, 2.50 Å [23]; 2.47 Å [24]) and $(1,2\text{-}^tBu_2C_5H_3)_2Yb(DME)$ (2.53 Å [29]). The observed O–Yb–O′ angle (68.5(7)°) is similar to those in most DME-solvated ytterbocenes(+2).

Thus, we observed not only the reduction of the ytterbium atom but also the breaking of the carbon bridge. We propose two mechanisms of this transformation, which are however disputable.

It is possible that the disodium salt of the ligand is the reductant. In this case, a substituted indenylytterbium(+2) chloride initially forms. Monometallic *ansa*-complexes of alkali metals are reported [14,33] to participate in the equilibrium which, in our case, can be written as Eq. (3).



(3)

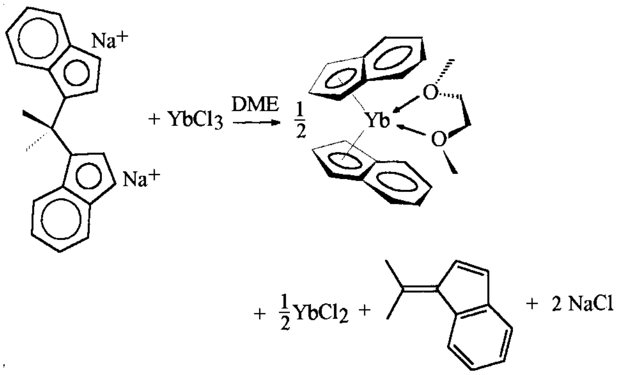
The subsequent disproportionation of unstable indenylytterbium(+2) chloride

Table 5
Selected bond lengths (Å) and bond angles (°) for $(C_9H_7)_2Yb{\cdot}DME$ (**1**)

Bond distances			
Yb–Cp	2.43(2)	Yb–C(7)	2.68(5)
Yb–O	2.41(2)	Yb–C(8)	2.63(4)
Yb–C(1)	2.74(2)	Yb–C(9)	2.66(3)
Yb–C(2)	2.71(2)		
Bond angles			
Cp–Yb–Cp′	129.4(8)	Cp–Yb–O	110.4(9)
Cp/Cp′	55.2(9)	Cp–Yb–O′	111.0(9)
O–Yb–O′	68.5(7)		

$$2IndYbCl \rightarrow Ind_2Yb + YbCl_2 \quad (4)$$

yields **1**, a more stable complex, and can therefore shift equilibrium of Eq. (3) to the right. The observed yield of **1** (68%) seems quite reasonable if ascribed to the overall reaction (5).



(5)

Alternatively, the reduction can occur by action of the monosodium salt of the ligand, which can result from the incomplete metallation of (CH$_3$)$_2$C(1-C$_9$H$_7$)$_2$. In this case, the calculated yield of **1** is 102%. This value is obviously unreal. However, it is not impossible that the reaction proceeds simultaneously by both mechanisms, that is, involves both mono- and disodium salts.

The factors that caused this unexpected reaction remain to be elucidated. We can only suppose that it was possible because of (1) the use of dry, unsolvated ytterbium(III) chloride, whose dissolution in DME, both a strongly basic and chelating agent, is accompanied by noticeable heat evolution and (2) the use of the sodium salt of the ligand in place of the commonly used lithium salt.

The reaction between the dilithium salt of ethylenebis(1-indene) or that of ethylenebis[1-(4,7-dimethylindene)] with YbCl$_3$ in diethyl ether leads to the *ate*-complex (CH$_2$)$_2$(1-Ind′)$_2$YbCl$_2$Li(Et$_2$O)$_2$, which, upon reduction by sodium metal in THF, gives *rac*-(CH$_2$)$_2$(1-Ind′)$_2$Yb(THF)$_2$ (Ind′ = C$_9$H$_6$ (**2**) or 4,7-(CH$_3$)$_2$C$_9$H$_4$ (**3**)).

In the crystal structure of **2** (Fig. 2, Table 6), there are two crystallographically independent molecules with Yb–Cp distances (2.44(3) and 2.46(3) Å) shorter than that in **1** and Cp–Yb–Cp′ angles (117.3(9) and 118.3(9)°) smaller than those in **1** and any of the ytterbocenes(+2). The Yb–O distances in **2** (2.346(16)–2.404(15) Å) are close to the lowest limit of the Yb^{+2}–O^{THF} distances in the known ytterbocene(+2) mono- and bis(tetrahydrofuranates) (2.34–2.46 Å), and the O–Yb–O′ angles (87.1(6) and 87.6(6)°) are somewhat larger than those in other ytterbocene(+2) bis(terahydrofuranates): (C$_5$H$_5$)$_2$Yb(THF)$_2$ (81.6°) [22]; (C$_5$H$_4$(CH$_2$)$_3$C$_5$H$_4$)Yb(THF)$_2$ (82.4°) [21]; (^{t}BuC$_5$H$_4$)$_2$-Yb(THF)$_2$ (83.3°) [25]; and (SiMe$_3$C$_5$H$_4$)$_2$Yb(THF)$_2$ (85°) [34].

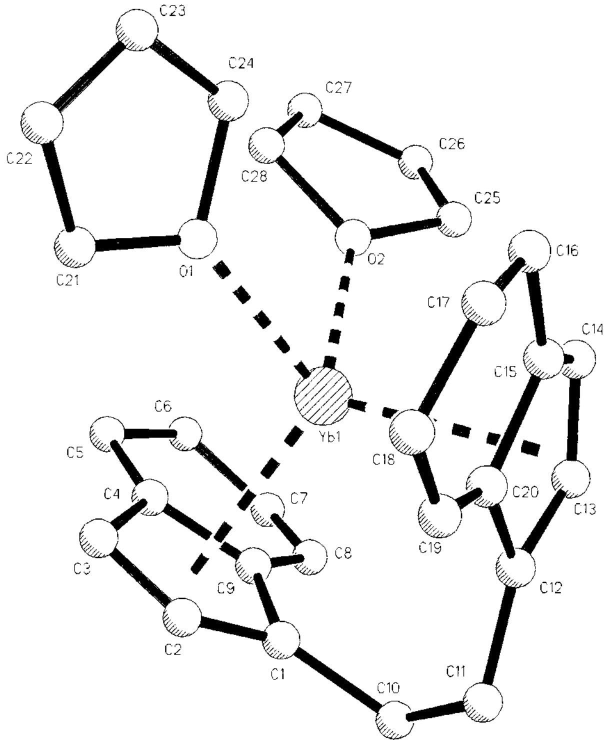


Fig. 2. Molecular structure of the complex *rac*-(CH$_2$)$_2$(C$_9$H$_6$)$_2$Yb(THF)$_2$ (**2**).

In the structure of **3** (Fig. 3, Table 7), the Yb–Cp and Yb–O distances and the Cp–Yb–Cp′ angle are identical to those in **2**, while the O–Yb–O′ angle

Table 6
Selected bond lengths (Å) and bond angles (°) for *rac*-(CH$_2$)$_2$(C$_9$H$_6$)$_2$Yb(THF)$_2$ (**2**)

Bond distances			
Yb(1)–Cp1	2.43(3)	Yb(2)–Cp1′	2.46(3)
Yb(1)–Cp2	2.45(3)	Yb(2)–Cp2′	2.46(3)
Yb(1)–O(1)	2.346(16)	Yb(2)–O(1′)	2.386(15)
Yb(1)–O(2)	2.404(15)	Yb(2)–O(2′)	2.388(15)
Yb(1)–C(14)	2.64(4)	Yb(2)–C(12′)	2.64(2)
Yb(1)–C(20)	2.68(3)	Yb(2)–C(1′)	2.69(3)
Yb(1)–C(4)	2.67(2)	Yb(2)–C(2′)	2.67(3)
Yb(1)–C(1)	2.67(2)	Yb(2)–C(9′)	2.71(5)
Yb(1)–C(13)	2.70(3)	Yb(2)–C(20′)	2.72(2)
Yb(1)–C(9)	2.69(3)	Yb(2)–C(3′)	2.74(3)
Yb(1)–C(12)	2.73(3)	Yb(2)–C(13′)	2.76(4)
Yb(1)–C(2)	2.71(4)	Yb(2)–C(14′)	2.75(2)
Yb(1)–C(3)	2.75(3)	Yb(2)–C(15′)	2.77(3)
Yb(1)–C(15)	2.75(4)	Yb(2)–C(4′)	2.79(4)
Bond angles			
O(1)–Yb(1)–O(2)	87.1(6)	O(1′)–Yb(2)–O(2′)	87.6(6)
Cp1-Yb(1)–Cp2	117.3(9)	Cp1′-Yb(2)–Cp2′	118.3(9)
Cp1/Cp2	61.1(9)	Cp1′/Cp2′	66.8(9)
Cp1–Yb–O(1)	111.3(9)	Cp1′–Yb–O(1′)	109.9(9)
Cp1–Yb–O(2)	115.1(9)	Cp1′–Yb–O(2′)	110.3(9)
Cp2–Yb–O(1)	113.9(9)	Cp2′–Yb–O(1′)	111.2(9)
Cp2–Yb–O(2)	108.4(9)	Cp2′–Yb–O(2′)	115.3(9)

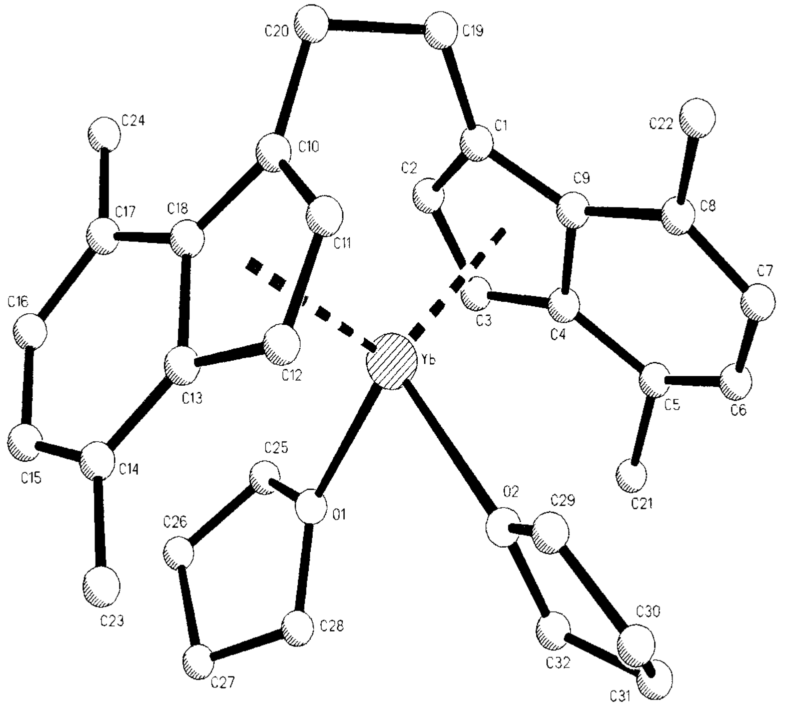


Fig. 3. Molecular structure of the complex *rac*-$(CH_2)_2[1\text{-}(4,7\text{-}(CH_3)_2C_9H_4)]_2Yb(THF)_2$ (**3**).

(91.8(9)°) is larger than those in **2** and any of the ytterbocene(+ 2) bis(tetrahydrofuranates).

Thus, our structural data suggest that the ytterbium atoms in ethylene-bound indenyl complexes **2** and **3** are screened by organic ligands to a lesser extent than those in the known ytterbocenes(+ 2).

4. Supplementary material

Listings of anisotropic temperature factors, hydrogen atom parameters, complete bond distances and angles of complexes **1**, **2** and **3** can be obtained from the authors on request. Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre under CCDC-116110 for **1**, CCDC-116111 for **2** and CCDC-116112 for **3**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: + 44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

Table 7
Selected bond lengths (Å) and bond angles (°) for *rac*-$(CH_2)_2[1\text{-}(4,7\text{-}(CH_3)_2C_9H_4)]_2Yb(THF)_2$ (**3**)

Bond distances			
Yb–Cp1	2.431	Yb–C(3)	2.70(4)
Yb–Cp2	2.437	Yb–C(12)	2.70(3)
Yb–O(1)	2.40(2)	Yb–C(4)	2.73(4)
Yb–O(2)	2.39(3)	Yb–C(10)	2.75(3)
Yb–C(11)	2.64(3)	Yb–C(13)	2.77(4)
Yb–C(2)	2.64(3)	Yb–C(18)	2.77(3)
Yb–C(1)	2.67(4)	Yb–C(9)	2.78(4)
Bond angles			
O(1)–Yb(1)–O(2)	91.8(9)	Cp1–Yb–O(1)	108.7
Cp1–Yb(1)–Cp2	118.8	Cp1–Yb–O(2)	112.8
Cp1/Cp2	66.8	Cp2–Yb–O(1)	112.6
		Cp2–Yb–O(2)	108.9

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