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Syntheses of bis(t-butylcyclopentadienyl)lanthanoid chloride complexes and crystal structures of bis(t-butylcyclopentadienyl)chloro(bistetrahydrofuran)-praseodymium and bis(t-butylcyclopentadienyl)-chlorotetrahydrofuranytterbium

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Abstract

Reaction of lanthanoid trichloride with two equivalents of sodium t-butylcyclopentadienide in tetrahydrofuran affords bis(t-butylcyclopentadienyl)lanthanoid chloride complexes $(\text{t-BuCp})_2\text{LnCl}\cdot n\text{THF}$ (Ln = Pr, Nd, $n = 2$; Ln = Gd, Yb, $n = 1$). The compound $(\text{t-BuCp})_2\text{PrCl}\cdot 2\text{THF}$ (1) crystallizes from THF in monoclinic space group $P2_1/c$ with unit cell dimensions $a = 15.080(3)$, $b = 8.855(2)$, $c = 21.196(5)$ Å, $\beta = 110.34(2)°$, $V = 2653.9$ Å^3 and $D_{\text{calcd}} = 1.41$ g/cm^3 for $Z = 4$. The central metal Pr is coordinated to two t-BuCp ring centroids, one chlorine atom and two THF forming a distorted trigonal bipyramid. The crystal of $(\text{t-BuCp})_2\text{YbCl}\cdot\text{THF}$ (2) belongs to the monoclinic crystal system, space group $P2_1/n$ with $a = 7.726(1)$, $b = 12.554(2)$, $c = 23.200(6)$ Å, $\beta = 97.77(2)°$, $V = 2229.56$ Å^3, $D_{\text{calcd}} = 1.50$ g/cm^3 and $Z = 4$. The t-BuCp ring centroids, the chlorine atom and the oxygen atom of the THF describe a distorted tetrahedron around the central ion of ytterbium.

Introduction

The complexes $(C_5H_5)_2\text{LnCl}$ are important precursors for the synthesis of lanthanoid alkyl and hydride compounds. However, $(C_5H_5)_2\text{LnCl}$ complexes are not available for La–Nd because steric saturation cannot be easily achieved with these early metals.

Recently, the use of sterically bulky ligands or bridged dicyclopentadienyl has given $(C_5R_5)_2\text{LnCl}$ for early lanthanoids [1–10].

However, less attention has been given to the use of t-butylcyclopentadienyl as ligand for synthesis of (t-butylcyclopentadienyl)lanthanoid derivatives. Recently, Wayda has reported his primary results on the sytheses of mono-, bis- and tris(t-butylcyclopentadienyl)lanthanoid complex but the crystal structure was not determined [11].

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We report here the syntheses of (t-BuCp)$_2$LnCl · *n*THF (Ln = Pr, Nd, *n* = 2; Ln-Gd, Yb, *n* = 1) and the details of the crystal structures of compounds **1** and **2**.

Experimental

All manipulations were conducted under argon using Schlenk techniques. Hexane was washed with sulfuric acid and H_2O, dried over $CaCl_2$, and heated to reflux over Na. THF was distilled from sodium benzophenone ketyl.

Anhydrous $LnCl_3$ was made by a published procedure [12], as was t-BuCpH [13]. Na(t-BuCp) was obtained from the reaction of t-BuCpH and NaH in THF. Analyses of rare earth metals and Cl were accomplished using direct complexometric titration with disodium EDTA and the Volhard method, respectively. Carbon and H analyses were carried out by combustion. IR spectra were recorded on a Perkin–Elmer spectrometer as KBr pellets. ^{1}H NMR spectra were obtained on a JEOL-FX-100 spectrometer and referenced to the residual protons in THF-d_8 (δ 3.58).

(t-BuCp)$_2$PrCl · 2THF (1)

To a suspension of $PrCl_3$ (0.99 g, 3.88 mmol) in 10 ml THF was slowly added a solution of Na(t-BuCp) (1.13 g, 7.84 mmol) in THF (40 ml). The mixture was stirred at 60 °C for 72 h. After centrifugation the solvent was concentrated and certain amount of hexane was added until the solution became a slightly turbid. The resulting solution was cooled to −30 °C, to give 0.82 g of the green crystals of **1** (37.5%). Anal. Found: C, 55.10; H, 7.25; Cl, 6.30; Pr, 25.83. $C_{26}H_{44}ClO_2Pr$ calcd: C, 55.47; H, 7.52; Cl, 6.30; Pr, 25.03%. IR (KBr pellet, cm^{-1}) 2950s, 2890m, 1600s, 1460m, 1360s, 1200m, 1150w, 1050s, 890m, 750s, 670s.

(t-BuCp)$_2$NdCl · 2THF

To a suspension of $NdCl_3$ (1.70 g, 6.7 mmol) in THF (10 ml) was added a solution of Na(t-BuCp) (1.95 g, 13.5 mmol) in THF (50 ml). After stirring 72 h at 60 °C, NaCl was removed by centrifugation. The purple solution was concentrated, an appropriate amount of hexane was added to the concentrate. The mixture was cooled at −30 °C for crystallization to give 1.21 g of the purple crystals of the product (32.2%). Anal. Found: C, 54.60; H, 7.20; Cl, 6.38; Nd, 25.8. $C_{26}H_{44}ClNdO_2$ calcd: Nd, C, 55.12; H, 7.40; Cl, 6.26; Nd, 25.47%. IR (KBr pellet, cm^{-1}): 2950s, 2890m, 1597s, 1461m, 1360s, 1200m, 1150w, 1055s, 890m, 750m, 670s.

(t-BuCp)$_2$GdCl · THF

To 10 ml a suspension of $GdCl_3$ (1.23 g, 4.71 mmol) in THF (10 ml) was added a solution of Na(t-BuCp) (1.36 g, 9.41 mmol) in THF (30 ml). After stirring 48 h at 60 °C, NaCl was separated from the reaction mixture by centrifugation. Evacuation of solvent produced a thick viscous sludge of light yellow product. The residue was washed twice with 10 ml portions of hexane, and dried to give 1.48 g white powder (62%). Anal. Found: C, 51.23; H, 6.70; Cl, 6.86; Gd, 30.67. $C_{22}H_{36}ClGdO$ calcd: Gd, 31.00; C, 52.10; H, 6.76; Cl, 6.99%. IR (KBr pellet, cm^{-1}): 2950s, 2890m, 1690s, 1359s, 1203m, 1152w, 1055m, 890m, 755m, 675m.

Table 1

Crystal data

	1	2
Compound	$(t\text{-BuCp})_2PrCl \cdot 2THF$	$(t\text{-BuCp})_2YbCl \cdot THF$
Molecular weight	563.1	523
Crystal size, mm	0.20×0.30×0.35	0.24×0.33×0.28
Cell constants		
a, Å	15.080(3)	7.726(1)
b, Å	8.855(2)	12.554(2)
c, Å	21.196(5)	23.200(6)
β, deg	110.34(2)	97.77(2)
V, Å^3	2653.9	2229.48
D_{calcd}, g/cm^3	1.41	1.50
Z	4	4
Temperature, °C	−60	25
Scan technique	ω	ω
2θ range, deg	3–40	3–56
Reflections	2882	4197
Reflections for $I \geqslant 3\sigma(I)$	1674	3156
Space group	$P2_1/c$	$P2_1/n$
$F(000)$	1160	972
$R = \Sigma \lVert F_o \rvert - \lvert F_c \rVert / \Sigma \lvert F_o \rvert$	0.058	0.044
$R_w = (\Sigma w(\lvert F_o \rvert - \lvert F_c \rvert)^2 / \Sigma w F_o^2)^{1/2}$	0.058	0.045

(t-BuCp)$_2$YbCl · THF (2)

To suspension of $YbCl_3$ (0.91 g, 3.2 mmol) in THF (10 ml) was added a solution of Na(t-BuCp) (0.92 g, 6.4 mmol) in THF (30 ml). After the Na(t-BuCp) solution had been added, the suspension turned dark-red. The reactant was then stirred at 60 °C for 48 h. After centrifugation, the solution was concentrated and cooled at −30 °C to give 0.45 g of the orange crystals of the product (27%). Anal. Found: Cl, 7.6; Yb, 32.3. $C_{22}H_{36}ClOYb$ calcd.: Yb, 33.1; Cl, 6.8%. IR (KBr pellet, cm^{-1}): 2950s, 2890m, 1598s, 1460m, 1359s, 1200m, 1155w, 1050m, 890m, 768s, 675s. ^1H NMR (THF-d_8) δ 5.90 (m, 8H), 0.9 (s, 18H) ppm.

X-Ray crystallography

A suitable single crystal was sealed under Ar in a thin-walled glass capillary. Intensity data were collected on a Nicolet *R3M/E* diffractometer with Mo-K_α radiation in the ω-scan mode. Final lattice parameters were obtained by a least-squares refinement of the 2θ values of 25 reflections. The space group $P2_1/c$ for compound **1** and $P2_1/n$ for compound **2** were identified from the systematic absences. The crystal data and the data collected parameters are listed in Table 1. The intensities were corrected for Lorentz and polarization, but not for absorption effects for compound **1** and for Lorentz, polarization and absorption effects for compound **2**.

The positions of metal atoms were revealed by a Patterson map and all non-hydrogen atoms were derived on successive Fourier synthesis. All positional parameters and temperature factors for non-hydrogen atoms were refined anisotropically.

Calculations were made on an Eclipse S/140 minicomputer with the SHELXTL program system. The atomic coordinates for compounds **1** and **2** are listed in Tables 2 and 3, respectively.

Table 2

Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters for **1** (Å$\times 10^3$, e.s.d.'s in parentheses)

	x	*y*	*z*	*U*
Pr	2217(1)	5484(1)	7154(1)	26(1)
Cl	3537(4)	3346(6)	7215(3)	39(2)
Oa	1182(10)	3360(17)	6382(7)	34(6)
Ca(1)	154(13)	3398(29)	6054(10)	41(9)
Ca(2)	−57(17)	1764(29)	5838(12)	43(11)
Ca(3)	718(13)	1396(27)	5560(10)	46(9)
Ca(4)	1607(15)	2185(22)	6080(11)	46(9)
Ob	3754(10)	6799(18)	7943(7)	39(6)
Cb(1)	3783(18)	8368(32)	8145(13)	52(12)
Cb(2)	4398(18)	8343(30)	8900(11)	64(11)
Cb(3)	5069(16)	7001(31)	8935(11)	65(11)
Cb(4)	4689(15)	6150(26)	8287(11)	41(9)
C(11)	1874(14)	4251(25)	8310(10)	31(9)
C(12)	970(14)	4275(27)	7750(10)	53(9)
C(13)	712(17)	5807(28)	7595(11)	42(11)
C(14)	1413(18)	6746(24)	8009(10)	69(11)
C(15)	2107(15)	5816(23)	8455(10)	34(9)
C(16)	2383(13)	2921(23)	8743(10)	37(8)
C(17)	1879(17)	2717(30)	9285(12)	62(12)
C(18)	2219(19)	1459(25)	8304(13)	64(12)
C(19)	3349(16)	3160(27)	9082(11)	52(10)
C(21)	2461(13)	7234(21)	6091(9)	20(8)
C(22)	2361(15)	8275(24)	6548(10)	30(9)
C(23)	1436(17)	8219(25)	6569(11)	53(11)
C(24)	943(16)	7074(26)	6122(11)	43(10)
C(25)	1553(15)	6449(25)	5804(10)	38(9)
C(26)	3267(15)	7049(25)	5829(11)	44(10)
C(27)	3328(16)	8525(26)	5431(11)	54(10)
C(28)	4230(15)	6976(26)	6445(11)	49(10)
C(29)	3230(16)	5693(29)	5388(11)	54(10)

Supplementary material. Tables of thermal parameters, bond distances and angles, best plane results and observed and calculated structure factors (49 pages) are available from the authors.

Results and discussion

Synthesis

$LnCl_3$ (Ln = Pr, Nd, Gd, Yb) reacts with 2 equiv. of Na(t-BuCp) in THF to give $(t\text{-BuCp})_2LnCl \cdot n\text{THF}$ according to eq. 1:

$$LnCl_3 + 2Na(t\text{-BuCp}) \xrightarrow{\text{THF}} (t\text{-BuCp})_2LnCl \cdot n\text{THF} + 2NaCl \qquad (1)$$

Ln = Pr, Nd, $n = 2$; Ln = Gd, Yb, $n = 1$

These complexes were characterized by IR, elemental analyses and NMR (for Yb). All these complexes can be crystallized from THF/hexane to give single crystals. The structures of compounds **1** and **2** are described below.

Table 3

Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters for **2** (Å$\times 10^3$, e.s.d.'s in parentheses)

	x	y	z	U
Yb	−213(1)	−1157(1)	1721(1)	42(1)
Cl	−3195(3)	−1140(2)	2067(1)	54(1)
O	1079(8)	−902(5)	2679(3)	58(2)
C(11)	−692(12)	−3009(8)	1126(4)	55(3)
C(12)	−690(14)	−3233(8)	1720(4)	59(4)
C(13)	916(14)	−3041(8)	2029(5)	64(4)
C(14)	2002(14)	−2645(9)	1641(5)	70(4)
C(15)	999(13)	−2646(8)	1075(5)	64(4)
C(16)	−2146(14)	−3249(9)	635(4)	64(4)
C(17)	−2219(24)	−4426(13)	536(8)	138(8)
C(18)	−1899(22)	−2676(16)	82(6)	126(8)
C(19)	−3925(17)	−2900(16)	782(7)	119(8)
C(21)	−1002(11)	763(7)	1221(4)	46(3)
C(22)	325(13)	915(8)	1687(4)	57(3)
C(23)	1859(13)	404(9)	1569(5)	74(4)
C(24)	1489(13)	−76(10)	1023(6)	80(5)
C(25)	−273(13)	106(8)	814(4)	55(3)
C(26)	−2717(12)	1346(8)	1100(5)	59(4)
C(27)	−4126(14)	638(12)	762(6)	85(5)
C(28)	−2447(16)	2339(10)	767(7)	93(6)
C(29)	−3397(16)	1651(11)	1679(5)	82(5)
C(31)	277(15)	−325(10)	3111(4)	71(4)
C(32)	1439(15)	−502(10)	3681(4)	79(5)
C(33)	3039(17)	−963(12)	3532(5)	99(6)
C(34)	2199(14)	−1041(11)	2898(5)	84(5)

The elemental analyses show that the bis(tetrahydrofuranate) of $(t\text{-BuCp})_2LnCl$ is isolated for the early lanthanide metal, Pr or Nd, but only mono(tetrahydrofuranate) of $(t\text{-BuCp})_2LnCl$ for the middle and late lanthanide metal (Gd and Yb). This is because steric saturation of the smaller metal can be readily achieved with fewer ligands. Attempts to prepare a similar complex with La failed. The results indicate that the t-BuCp ring is bulky enough to stabilize biscyclopentadienyl derivatives of Pr or Nd, but it is too small for the largest metal La.

The complexes are thermally stable but very sensitive to air and moisture. They are soluble in ethers and moderately soluble in aromatic solvents.

Crystal structure

The crystal structures of compounds **1** and **2** are illustrated in Figs. 1 and 2, respectively. The important bond lengths and angles are listed in Tables 4 and 5, respectively.

As shown in Fig. 1, compound **1** is a monomer. To our knowledge, this is the first example of a monomer structure of $(C_5R_5)_2LnCl$ for Ln = La–Nd. The formally nine-coordinate Pr center is surrounded by two t-BuCp, one Cl and two oxygens. The coordinated polyhedron is a distorted trigonal bipyramid in which Oa, Ob are at both apical positions and Cl, Pr and two centroids of the cyclopentadienyl rings are at the equatorial positions. In the plane, the angles of ring centroid–Pr–ring

Table 4

Selected bond distances (Å) and angles (°) for **1** (e.s.d.'s in parentheses)

Pr–Cl	2.718(6)	Pr–Oa	2.622(13)
Pr–Ob	2.614(13)	Pr–C(11)	2.888(23)
Pr–C(12)	2.816(24)	Pr–C(13)	2.753(28)
Pr–C(14)	2.743(27)	Pr–C(15)	2.835(23)
Pr–C(21)	2.862(21)	Pr–C(22)	2.829(22)
Pr–C(23)	2.788(22)	Pr–C(24)	2.745(20)
Pr–C(25)	2.815(2))	AvPr–C(ring 1)	2.807(23)
AvPr–C(ring 2)	2.808(21)	C(11)–C(12)	1.462(24)
C(12)–C(13)	1.419(34)	C(13)–C(14)	1.391(30)
C(14)–C(15)	1.407(28)	C(21)–C(22)	1.383(30)
C(22)–C(23)	1.412(35)	C(23)–C(24)	1.411(30)
C(24)–C(25)	1.427(37)	C(21)–C(25)	1.466(27)
C(21)–C(26)	1.511(34)	C(11)–C(16)	1.526(27)
C(11)–C(15)	1.437(30)	Pr–Cent(1) [a]	2.533(2)
Pr–Cent(2)	2.536(2)		
Cl–Pr–Oa	78.3(3)	Cl–Pr–Ob	78.9(4)
Oa–Pr–Ob	157.2(5)	C(12)–C(13)–C(14)	109.7(18)
C(11)–C(12)–C(13)	107.8(18)	C(11)–C(15)–C(14)	110.5(16)
C(13)–C(14)–C(15)	107.4(19)	C(12)–C(11)–C(16)	129.1(19)
C(12)–C(11)C(15)	104.4(17)	C(21)–C(22)–C(23)	110.4(18)
C(15)–C(11)–C(16)	125.5(15)	C(24)–C(25)–C(21)	106.5(18)
C(22)–C(23)–C(24)	107.4(22)	C(23)–C(24)–C(25)	108.6(21)
C(22)–C(21)–C(25)	107.0(19)	C(25)–C(21)–C(26)	123.5(18)
C(22)–C(21)–C(26)	128.8(18)	C(11)–C(16)–C(18)	109.8(16)
C(21)–C(26)–C(29)	116.8(19)	C(11)–C(16)–C(19)	113.7(18)
C(21)–C(26)–C(28)	109.0(18)	Cent(1) [a]–Pr–Cl	117.9(2)
Cent(2)–Pr–Cl	121.0(2)	Cent(1)–Pr–Oa	95.5(5)
Cent(2)–Pr–Oa	93.7(4)	Cent(1)–Pr–Ob	95.2(5)
Cent(2)–Pr–Ob	97.9(4)		

[a] Cent(1) is the centroid of cyclopentadienyl ring defined by C(11)–C(15).

centroid, 121.1(1)°, ring centroid(1)–Pr–Cl, 117.9(2)° and ring centroid(2)–Pr–Cl, 121.0(2)°, are very close to the expected 120° for a trigonal bipyramid. However, the angle Oa–Pr–Ob of 157.2(5)° is far smaller than the theoretical angle of 180° for this geometry. The angle Oa–Pr–Cl, 78.3(3)° is almost equal to the angle Ob–Pr–Cl of 78.9(4)° and the distances of Pr–Oa, Pr–Ob are 2.614(13) and 2.622(13) Å, respectively. So the molecule has an approximate mirror plane formed by Cl, Pr and two centroids of the cyclopentadienyl rings.

Figure 2 shows that the two t-BuCp, the one Cl and the one oxygen of the THF, form a distorted tetrahedron around ytterbium. The ring centroid–Yb–ring centroid angle is 121.3(3)°, while in the crystal structure of $(C_5Me_5)_2YbCl(THF)$ [14] and $(C_5Me_5)_2YbCl$[1,1-bis(dimethylphosphino)methane] [15], they are 136.1(1)° and 134.9°, respectively. The smaller angle in compound **2** is probably due to the least steric interaction among the two t-BuCp, Cl and O. The ring centroid–Yb–O angle of 104.3(3)° and O–Yb–Cl angle of 89.3(3)° are in the range commonly observed for bis(pentamethylcyclopentadienyl)lanthanoid [16].

The average metal–C(ring) distance in compound **2**, 2.628(11) Å, is comparable with the analogous distances in $(C_5Me_5)_2YbCl \cdot THF$, 2.65(1) Å and in compound **1**, 2.808(23) Å, after corrections of 0.15 Å for the difference in ionic radii of Pr^{3+} and

Table 5

Selected bond distances (Å) and angles (°) for **2** (e.s.d.'s in parentheses)

Yb–Cl	2.539(3)	Yb–O	2.333(6)
Yb–C(11)	2.704(10)	Yb–C(12)	2.632(10)
Yb–C(13)	2.588(10)	Yb–C(14)	2.557(11)
Yb–C(15)	2.645(11)	Yb–C(1) [a]	2.625(10)
Yb–C(21)	2.708(9)	Yb–C(22)	2.637(10)
Yb–C(23)	2.585(11)	Yb–C(24)	2.602(13)
Yb–C(25)	2.630(10)	Yb–C(2) [a]	2.632(11)
C(11)–C(12)	1.404(14)	C(11)–C(15)	1.404(14)
C(11)–C(16)	1.518(13)	C(12)–C(13)	1.368(14)
C(13)–C(14)	1.403(16)	C(14)–C(15)	1.431(15)
C(21)–C(25)	1.426(14)	C(21)–C(22)	1.398(14)
C(21)–C(26)	1.507(13)	C(22)–C(23)	1.407(15)
C(23)–C(24)	1.399(18)	C(24)–C(25)	1.401(13)
Cent(1) [a]–Yb	2.338(1)	Cent(2)–Yb	2.342(1)
Cl–Yb–O	89.3(2)	Cl–Yb–Cent(1) [a]	108.7(1)
Cl–Yb–Cent(2)	114.2(1)	O–Yb–Cent(1)	104.3(3)
O–Yb–Cent(2)	101.9(3)	Cent(1)–Yb–Cent(2)	121.3(3)
Yb–C(11)–C(16)	125.1(7)	Yb–C(21)–C(26)	130.8(6)

[a] C(1)–Yb is the mean distance of five Yb–C(ring) bond lengths; Cent(1) is the centroid of cyclopentadienyl ring defined by C(11)–C(15).

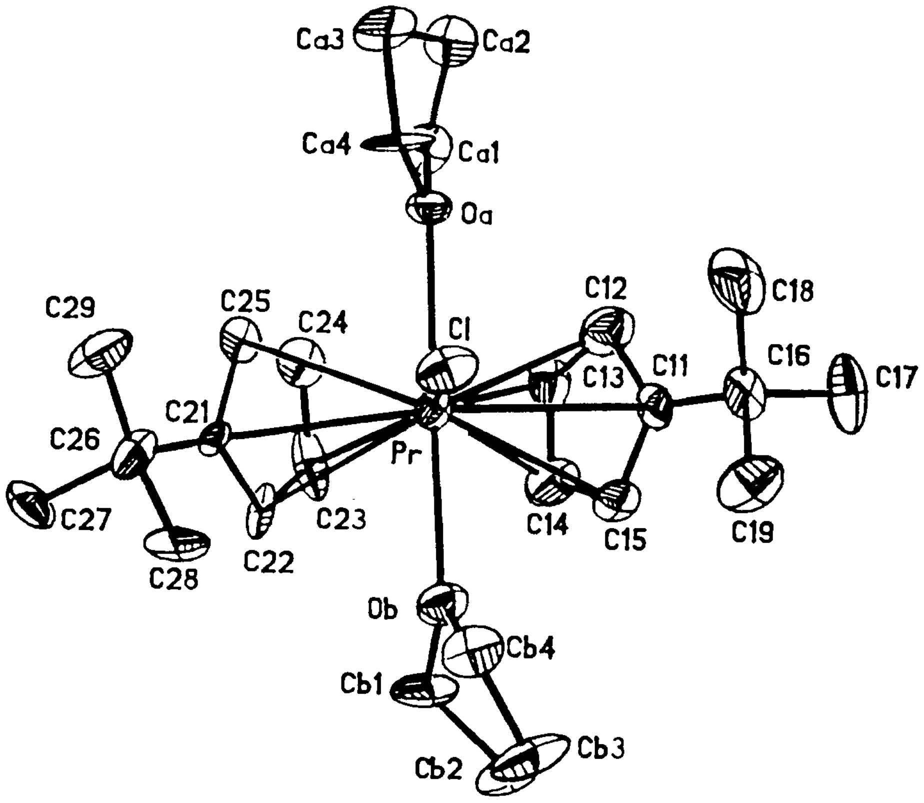

Fig. 1. Crystal structure of (t-BuCp)$_2$PrCl·2THF (1).

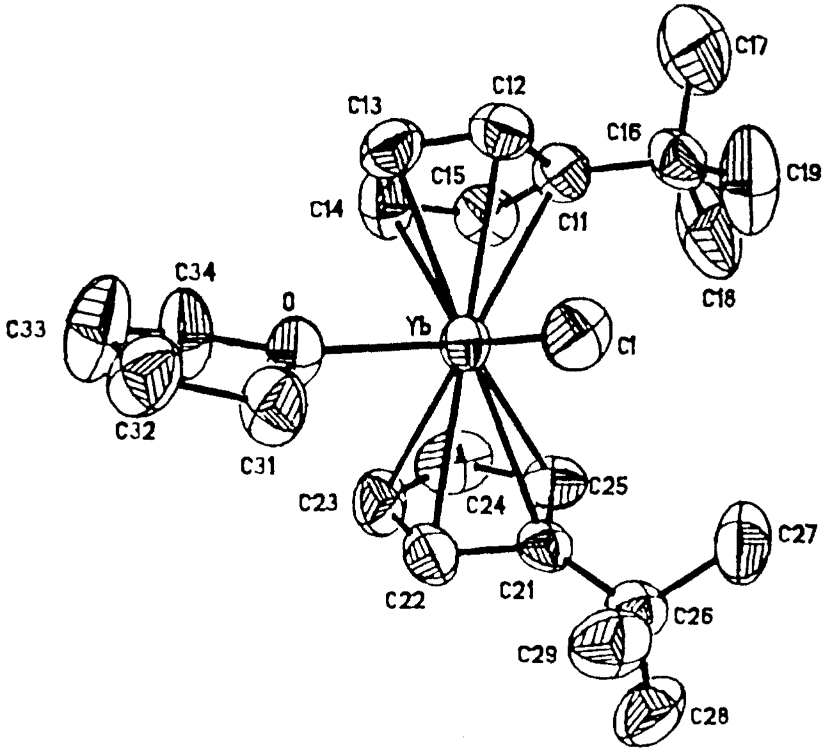

Fig. 2. Crystal structure of $(t\text{-BuCp})_2\text{YbCl}\cdot\text{THF}$ (**2**).

Yb^{3+}. The metal–O(THF) distance in **2**, 2.333(6) Å, is somewhat shorter than the distances in $(\text{C}_5\text{Me}_5)_2\text{YbCl}\cdot\text{THF}$, 2.362 Å and in **1**, 2.618(13) Å even though the difference in ionic radii of Pr^{3+} and Yb^{3+} was taken into account.

The t-BuCp groups in compound **1** are located at opposite sides of the molecule, directed away from each other and from the Cl, Oa and Ob, while the t-BuCp groups in compound **2** are arranged at the same side of the molecule. The ring carbon atoms for both compounds all lie within 0.016 Å of the calculated C(C(11)–C(15), C(21)–C(25)) mean plane.

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