Synthesis and structural characterization of functionalized organolanthanide complexes [(η⁵-C₅H₄PPh₂)₂Ln(DIME)] (Ln = Eu, Yb) (DIME = diethylene glycol dimethyl ether)

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Received 20 February 1996, in revised form 18 March 1996

Abstract

The reaction of [(diphenylphosphino)cyclopentadienyl]thallium [Tl(C₅H₄PPh₂)] with metallic europium or ytterbium powder in THF (tetrahydrofuran) at 60°C in the presence of metallic mercury, followed by crystallization from a mixture of dimethoxyethane and diethylene glycol dimethyl ether (DIME) (10:1), afforded [(η⁵-C₅H₄PPh₂)₂Eu(DIME)] 1 and [(η⁵-C₅H₄PPh₂)₂Yb(DIME)] 2 respectively. Crystal and molecular structures of 1 and 2 have been determined by X-ray crystallography.

Keywords: Ytterbium; Europium; Thallium; Cyclopentadienyl; (Diphenylphosphino)cyclopentadienyl; Structure; Diethylene glycol dimethyl ether

1. Introduction

The ligand (diphenylphosphino)cyclopentadienyl is a combination of phosphine and cyclopentadienyl, both of which are ubiquitous in the organometallic chemistry of transition metals, so it has been extensively used in the syntheses of homo- and heterobimetallic complexes [1–9]. However, its application in organolanthanide chemistry is more limited. To date, less than ten complexes have been synthesized, and, to our knowledge, seven compounds have been structurally characterized: [{(η⁵-C₅H₄PPh₂)₂Er}₃(μ₃-O)(μ-OH)₂][Na(DME)₃] [10] (DME = dimethoxyethane), [(η⁵-C₅H₄PPh₂)₂Yb(μ-O₂CCH₃)]₂ [11], [(η⁵-C₅H₄PPh₂)₂Yb(DME)] [12], [(η⁵-C₅H₄PPh₂)₂Yb(μ-Cl)₂Na(DME)₂] [13], [(η⁵-C₅H₄PPh₂)₂YbCl]₂ [14], [Yb(THF)₂(η⁵-C₅H₄PPh₂)₂PtMe₂] · THF [15] and [Yb(THF)₂(η⁵-C₅H₄PPh₂)₂Ni(CO)₂] [16].

It has been reported that transmetallation reactions between (cyclopentadienyl)thallium, CpTl, and lanthanide elements in THF can be used to prepare cyclopentadienyl lanthanide complexes [17]. However, this reaction does not work for europium, even at elevated temperature. This is attributed to the fine balance of europium and CpTl redox potentials in THF. Nonetheless, the reaction of Eu with CpTl takes place in liquid ammonia and other N-donor solvents, such as pyridine and acetonitrile, to give divalent or trivalent europium complexes [18]. With europium being the least reducing among the lanthanide series [19], its reactivity in transmetallation should shed light on the reactivity of other lanthanide elements towards transmetallation.

We believe that changing the substituent on the cyclopentadienyl ring should change the redox potential and transmetallation will be observed. In this context, we have studied the interaction of [TlC₅H₄PPh₂] with metallic Eu and Yb in the presence of Hg in THF.

2. Results and discussion

The reactions of excess lanthanide elements (La, Nd, Sm, Eu, Er and Yb) with [TlC₅H₄PPh₂] in THF at 60°C have been investigated. They all give a complex reaction mixture which is difficult for isolation of pure lanthanide complex. However, in the cases of europium and ytterbium, addition of metallic mercury in the reaction led to a faster reaction and gave divalent complexes of Eu and Yb as the only isolated products.

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The crystal structure determination of complexes 1 and 2 revealed that, in the crystalline state, both compounds consist of discrete neutral monomeric molecules with similar structural configurations. Figs. 1 and 2 are the perspective views of molecular structures of 1 and 2 respectively. The two molecules have almost identical atom-continuity. The central ion (Eu^{2+} in 1 and Yb^{2+} in 2) has a formal coordination number (CN) of nine, complemented by two cyclopentadienyl rings (assuming Cp is equivalent to three σ-donor ligands) and three oxygen atoms of a diethylene glycol dimethyl ether (DIME) molecule. The oxygen atoms and central ion are almost coplanar. In 1, the least-square plane defined by O(1), O(2), O(3), and Eu(1) gave the maximum deviation of 0.16 Å. Therefore, the structural geometry around the central ion can be described as a distorted tetrahedron formed by O(1), O(3) and the two centroids of the cyclopentadienyl rings. Atom O(2) coordinated to the central ion and located in the plane defined by O(1), O(3) and Eu^{2+} (in 1) or Yb^{2+} (in 2) with the bond of O(2)–Eu[Yb] bisecting the angle of O(1)–Eu(1)–O(3) [O(1)–Yb(1)–O(3)]. This kind of structural geometry around a lanthanide ion with CN of nine was also observed in the cation of [((η^5-C$_5$H$_4$PPh$_2$)$_2$Er)$_3$(μ$_3$-O)(μ-OH)$_2$]$^-$ [10]. The orientation of phosphorus atoms in 1 and 2 are syn-oriented almost directly above or
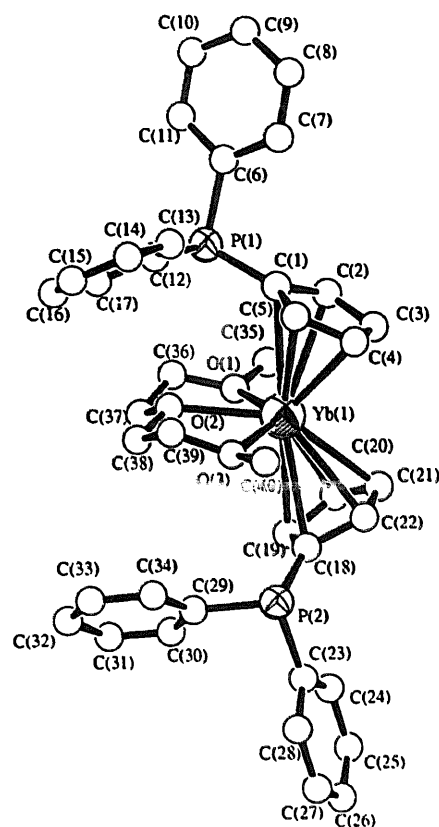
Fig. 2. The molecular structure of [(η^5-C$_5$H$_4$PPh$_2$)$_2$Yb(DIME)] 2.
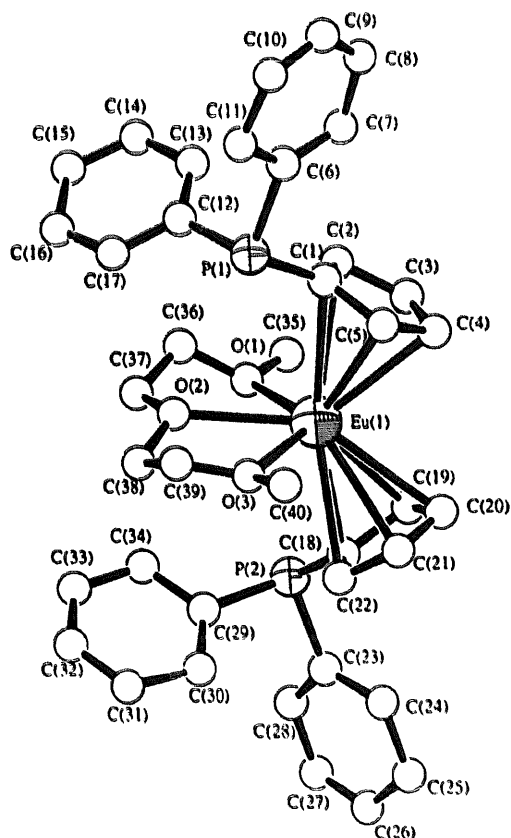
below the trapezoid formed by O(1), O(2), O(3) and Eu(1) (in 1) or Yb(1) (in 2). This is not observed in the structures of other organolanthanide complexes containing (diphenylphosphino)cyclopentadienyl ligands.

The average bond lengths of Eu(1)–C 2.89 Å, (range from 2.83 to 2.94 Å) and Eu–O 2.62 Å, (range from 2.60 to 2.63 Å) in 1 are longer than those of Yb–C 2.774 Å, (range from 2.736 to 2.830 Å) and Yb(1)–O 2.496 Å, (range from 2.470 to 2.521 Å) in 2, which is in accordance with the 'lanthanide contraction'. However, the angle of Ccent–Eu(1)–C'cent, 124.8°, is smaller than that of Ccent–Yb(1)–C'cent, 132.0°, which is presumably due to the greater steric crowding around Yb^{2+} than Eu^{2+} because of the relatively smaller size of Yb^{2+}. The average bond distances of Yb(1)–C and Yb(1)–O in 2 are slightly longer than the corresponding distances in complex [(η^5-C$_5$H$_4$PPh$_2$)$_2$Yb(DME)] 3, (2.710 Å and 2.404 Å) [12] due to a larger CN of nine in 1 compared with eight in 3. The average distance of Yb(1)–C in 2 is significantly longer than that in complex [(η^5-C$_5$H$_4$PPh$_2$)$_2$Yb(μ-O$_2$CCH$_3$)]$_2$ 4, 2.617 Å [11] with a difference of 0.157 Å, which should be the difference between Yb^{2+} and Yb^{3+} ionic radii with CN of nine. If this is the case, we can obtain the ionic radius of Yb^{2+} with CN of nine as 1.20 Å [20]. Subtraction of 1.20 Å from the average Yb(1)–C distance in 2 gives

Fig. 1. The molecular structure of [(η^5-C$_5$H$_4$PPh$_2$)$_2$Eu(DIME)] 1.

1.57 Å, as the effective 'ionic radius' of the cyclopentadienyl ion, which is almost the same as the result obtained by similar calculation on complex 1 (1.59 Å) and the value reported by Deacon et. al. (1.58 Å) [11]. Subtraction of the ionic radius (1.14 Å) for eight-coordinated Yb^{2+} from the Yb–C distance (2.71 Å) in 3 gives 1.57 Å [20].

The ^{31}P NMR spectra of complexes 1 and 2 in THF at room temperature are complicated. For 1, two peaks at −13.78 and −15.68 ppm were observed, which were almost unchanged while the experimental temperature was raised to 60°C. This is consistent with the solid state only if one assumes that the DIME ligand remains in the same conformation as in the solid state so that two P atoms are in a distinct environment. However, this is rather unlikely. Therefore, we suspect that some isomerisation processes of 1 may occur in solution. For 2, the spectrum is even more complicated. Eight peaks were observed in the range between 10.03 and −37.87 ppm. Those may be due to the complex adducts resulting from the exchange between the solvent THF and coordinated DIME.

3. Experimental details

The complexes described here are extremely air- and moisture-sensitive, so the synthesis and subsequent manipulations were all carried out under purified dinitrogen with rigorous exclusion of air and moisture by the use of vacuum line techniques. Solvents were dried as previously described [12]. Metallic ytterbium (Strem) was used as-received. Metallic europium (Aldrich) stored in oil was cut into small chips, washed with THF twice and dried in vacuum before used. [Tl($C_5H_4PPh_2$)] was prepared by the published procedure [12]. ^{31}P NMR spectra (proton decoupled) were recorded on a Jeol GSX 270 FT spectrometer with a glass capillary filled with phosphoric acid (85%) as external reference.

3.1. Synthesis of [(η^5-$C_5H_4PPh_2$)$_2$Eu(DIME)] 1

[Tl($C_5H_4PPh_2$)] (0.45 g, 1 mmol) was mixed with an excess of europium chips (0.45 g, 2.96 mmol) and a small amount of metallic mercury (ca. 0.1 g) in a Schlenk flask and THF (30 ml) was added with a syringe. The suspension was stirred at 60°C for 18 h. The mixture was then centrifuged to give a purple solution and dark thallium metal. Removal of the THF in vacuo left a purple residue, which was extracted with DME (10 ml). A red solution was obtained after centrifugation. DIME (1 ml) was added slowly to give a color change from red to yellow. The resulting solution was stirred for 10 min; the yellow block single crystals of 1 in ca. 30% yield were obtained by cooling the solution to −20°C overnight.

3.2. Preparation of [(η^5-$C_5H_4PPh_2$)$_2$Yb(DIME)] 2

A similar method was used as in the synthesis of complex 1; the orange block single crystals suitable for X-ray analysis were obtained in ca. 40% yield.

3.3. X-ray analyses of complexes 1 and 2

Crystals of complexes 1 and 2 were selected and mounted in glass capillaries under deoxygenated paraffin oil. Crystal intensity data were collected at ambient temperature on Rigaku AFC7R for 1 and Enraf–Nonius for 2 diffractometers using Mo Kα radiation (λ = 0.71073 Å). Crystals of 1 were obtained with difficulty and diffracted relatively weakly. The data were corrected for Lorentz and polarization effects and absorp-

Table 1
Crystallographic data for 1 and 2 [a]

	1	2
Formula	$EuP_2C_{40}H_{42}O_3$	$YbP_2C_{40}H_{42}O_3$
Formula weight	784.68	805.76
Color; habit	Yellow, block	Orange, block
Crystal size (mm³)	0.12×0.22×0.28	0.22×0.28×0.28
Crystal system	Orthorhombic	Triclinic
Space group	$Pna2_1$ (No.33)	$P\bar{1}$ (No.2)
Unit cell dimensions		
a (Å)	17.258(3)	10.111(1)
b (Å)	21.924(4)	10.618(1)
c (Å)	9.864(2)	18.881(3)
α (deg)	90	86.49(2)
β (deg)	90	87.74(2)
γ (deg)	90	63.04(1)
Volume (Å³)	3732.3(9)	1803.2(4)
Z	4	2
D_{calc} (g cm⁻³)	1.396	1.484
μ(Mo Kα)(cm⁻¹)	17.97	27.18
F (000)	1596	812
2θ range (deg)	2–45	2–45
Scan range ω	0.89 + 0.35tanθ	0.75 + 0.35tanθ
Number of reflections collected	2793	5035
Number of independent reflections	2793	4708
Number of observed reflections ($I > 3\sigma(I)$)	1548	4028
R; R_w (observed data)	0.037, 0.035	0.034, 0.036
Goodness of fit	1.62	1.99
Largest Δ/σ	0.01	0.02
Number of parameters	199	215
Residual extrema in the final difference map (e Å⁻³)	0.50 to −0.30	0.88 to −0.68

[a] Data in common: radiation Mo Kα (λ = 0.71073 Å); temperature (K) 298; scan type ω–2θ; scan speed (deg min⁻¹) 16.0 (up to four scans); background measurement 25% at both ends; absorption correction ψ-scan method; refinement method full-matrix least squares; weighting scheme ω = 1/σ²(F_0^2).

tion by the ψ-scan method [21]. The structures were solved by the direct methods (SIR 88) [22] and refined by full-matrix least squares analysis (on F) with metal and phosphorus atoms varied anisotropically. Hydrogen atoms were placed at their idealized positions and included in structure factor calculations but not refined. For **1**, the alternative centrosymmetric space group

Table 2
Atomic coordinates and isotropic displacement coefficients B_{eq} for **1**

Atom	x	y	z	B_{eq} (Å^2)
Eu(1)	0.23870(3)	0.00120(5)	−0.0070	3.36(1)
P(1)	0.0394(2)	0.0934(2)	−0.1059(5)	4.0(1)
P(2)	0.4596(2)	−0.0709(2)	−0.0784(5)	3.8(1)
O(1)	0.3378(7)	0.0706(6)	−0.133(1)	6.5(3)
O(2)	0.2249(6)	0.0114(5)	−0.271(1)	5.5(3)
O(3)	0.1422(7)	−0.0718(6)	−0.134(1)	5.6(3)
C(1)	0.1101(8)	0.0877(7)	0.024(2)	4.0(4)
C(2)	0.1809(9)	0.1228(7)	0.052(2)	5.0(4)
C(3)	0.209(1)	0.0988(8)	0.180(2)	5.2(4)
C(4)	0.163(1)	0.0534(9)	0.226(2)	6.9(5)
C(5)	0.104(1)	0.0448(8)	0.132(2)	5.3(4)
C(6)	−0.0278(8)	0.1516(7)	−0.033(2)	4.3(4)
C(7)	−0.013(1)	0.1862(8)	0.074(2)	5.5(4)
C(8)	−0.071(1)	0.2280(9)	0.127(2)	7.1(5)
C(9)	−0.141(1)	0.2299(9)	0.061(2)	6.8(5)
C(10)	−0.155(1)	0.1935(10)	−0.045(2)	7.2(6)
C(11)	−0.102(1)	0.1527(9)	−0.100(2)	6.2(5)
C(12)	0.0852(8)	0.1406(6)	−0.237(2)	3.0(3)
C(13)	0.1150(9)	0.1982(7)	−0.214(2)	4.2(4)
C(14)	0.150(1)	0.2333(8)	−0.315(2)	5.3(4)
C(15)	0.153(1)	0.2097(9)	−0.443(2)	5.7(5)
C(16)	0.123(1)	0.1506(9)	−0.472(2)	6.6(5)
C(17)	0.0911(10)	0.1177(8)	−0.369(2)	5.2(4)
C(18)	0.3741(8)	−0.0786(6)	0.026(2)	3.4(3)
C(19)	0.3652(9)	−0.0446(7)	0.154(2)	3.8(4)
C(20)	0.300(1)	−0.0682(9)	0.215(2)	6.2(5)
C(21)	0.2644(10)	−0.1101(8)	0.132(2)	5.2(4)
C(22)	0.3088(8)	−0.1171(6)	0.020(2)	4.1(4)
C(23)	0.5204(7)	−0.1343(6)	−0.021(2)	3.6(3)
C(24)	0.5037(9)	−0.1721(7)	0.083(2)	4.3(4)
C(25)	0.555(1)	−0.2200(9)	0.127(2)	7.0(5)
C(26)	0.622(1)	−0.2268(8)	0.044(2)	5.7(5)
C(27)	0.642(1)	−0.1883(9)	−0.052(2)	5.9(5)
C(28)	0.592(1)	−0.1432(8)	−0.088(2)	5.2(4)
C(29)	0.4299(8)	−0.1036(7)	−0.240(2)	3.7(3)
C(30)	0.4042(9)	−0.1620(7)	−0.257(2)	4.3(4)
C(31)	0.378(1)	−0.1836(9)	−0.388(2)	5.9(5)
C(32)	0.3855(10)	−0.1417(9)	−0.491(3)	6.8(5)
C(33)	0.4115(10)	−0.0866(8)	−0.481(3)	6.5(5)
C(34)	0.4320(10)	−0.0656(8)	−0.353(2)	5.0(4)
C(35)	0.388(1)	0.105(1)	−0.065(2)	8.6(7)
C(36)	0.313(1)	0.0927(9)	−0.259(2)	7.0(5)
C(37)	0.281(1)	0.0453(9)	−0.342(2)	7.0(5)
C(38)	0.197(1)	−0.0396(9)	−0.340(2)	7.5(6)
C(39)	0.128(1)	−0.061(1)	−0.269(3)	9.3(7)
C(40)	0.081(1)	−0.100(1)	−0.069(3)	10.3(8)

$$B_{eq} = \frac{8}{3}\pi^2\left(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2\right.$$
$$+ 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta$$
$$\left. + 2U_{23}bb^*cc^*\cos\alpha\right).$$

Table 3
Atomic coordinates and isotropic displacement coefficients B_{eq} for **2**

Atom	x	y	z	B_{eq} (Å^2)
Yb(1)	−0.92507(3)	−0.60028(3)	−0.26131(2)	2.913(7)
P(1)	−1.2278(2)	−0.7086(2)	−0.17830(10)	3.32(4)
P(2)	−0.7952(2)	−0.2824(2)	−0.30053(10)	3.30(4)
O(1)	−1.0069(5)	−0.3833(5)	−0.1924(3)	4.2(1)
O(2)	−1.1899(5)	−0.4321(4)	−0.2764(3)	3.7(1)
O(3)	−1.0554(5)	−0.6527(5)	−0.3579(3)	4.1(1)
C(1)	−1.0364(7)	−0.7442(7)	−0.1664(3)	3.2(1)
C(2)	−0.9700(7)	−0.6834(7)	−0.1238(4)	3.7(1)
C(3)	−0.8174(8)	−0.7488(8)	−0.1357(4)	4.3(2)
C(4)	−0.7851(8)	−0.8523(8)	−0.1851(4)	4.3(2)
C(5)	−0.9185(8)	−0.8509(7)	−0.2053(4)	3.8(1)
C(6)	−1.2501(7)	−0.8465(7)	−0.1206(4)	3.3(1)
C(7)	−1.1431(8)	−0.9404(8)	−0.0756(4)	4.5(2)
C(8)	−1.1695(8)	−1.0431(8)	−0.0355(4)	4.8(2)
C(9)	−1.3002(8)	−1.0515(8)	−0.0422(4)	4.2(2)
C(10)	−1.4053(8)	−0.9600(8)	−0.0879(4)	4.2(2)
C(11)	−1.3819(7)	−0.8563(7)	−0.1275(4)	3.5(1)
C(12)	−1.3351(7)	−0.5511(7)	−0.1262(4)	3.5(1)
C(13)	−1.3092(8)	−0.5407(8)	−0.0560(4)	4.6(2)
C(14)	−1.3947(9)	−0.4138(9)	−0.0221(5)	5.4(2)
C(15)	−1.5040(9)	−0.3047(9)	−0.0568(5)	5.7(2)
C(16)	−1.5364(9)	−0.3108(9)	−0.1255(5)	5.5(2)
C(17)	−1.4493(8)	−0.4360(8)	−0.1613(4)	4.3(2)
C(18)	−0.7713(7)	−0.4557(7)	−0.3251(4)	3.5(1)
C(19)	−0.6625(8)	−0.5794(8)	−0.2898(4)	4.3(2)
C(20)	−0.6536(9)	−0.6940(9)	−0.3276(4)	5.2(2)
C(21)	−0.7498(9)	−0.6475(9)	−0.3809(4)	5.2(2)
C(22)	−0.8252(8)	−0.4983(8)	−0.3817(4)	4.3(2)
C(23)	−0.6767(7)	−0.2440(7)	−0.3668(3)	3.2(1)
C(24)	−0.6114(7)	−0.3211(7)	−0.4258(4)	3.8(1)
C(25)	−0.5226(8)	−0.2856(8)	−0.4727(4)	4.1(2)
C(26)	−0.4994(8)	−0.1701(8)	−0.4599(4)	4.3(2)
C(27)	−0.5612(8)	−0.0938(8)	−0.4018(4)	4.5(2)
C(28)	−0.6498(8)	−0.1287(7)	−0.3547(4)	3.8(1)
C(29)	−0.9777(7)	−0.1552(7)	−0.3358(3)	2.9(1)
C(30)	−1.0143(8)	−0.1361(8)	−0.4065(4)	4.0(1)
C(31)	−1.1528(8)	−0.0390(8)	−0.4302(4)	4.7(2)
C(32)	−1.2569(8)	0.0441(8)	−0.3813(4)	4.0(1)
C(33)	−1.2210(8)	0.0299(8)	−0.3113(4)	4.0(1)
C(34)	−1.0822(7)	−0.0686(7)	−0.2876(4)	3.4(1)
C(35)	−0.9258(9)	−0.3763(9)	−0.1346(5)	5.4(2)
C(36)	−1.1643(8)	−0.3070(8)	−0.1825(4)	4.7(2)
C(37)	−1.2377(8)	−0.2942(8)	−0.2511(4)	4.7(2)
C(38)	−1.2514(8)	−0.4278(8)	−0.3436(4)	4.8(2)
C(39)	−1.2099(9)	−0.5732(8)	−0.3638(4)	4.9(2)
C(40)	−1.0034(9)	−0.7848(9)	−0.389(5)	5.9(2)

$$B_{eq} = \frac{8}{3}\pi^2\left(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2\right.$$
$$+ 2U_{12}aa^*bb^*\cos\gamma + 2U_{13}aa^*cc^*\cos\beta$$
$$\left. + 2U_{23}bb^*cc^*\cos\alpha\right).$$

Pnma (No. 62) does not give any chemically reasonable solution. All calculations were performed on a Silicon-Graphics computer using the package of TeXsan from MSC [23].

The pertinent crystallographic data for both **1** and **2** are listed in Table 1. Coordinates of non-hydrogen atoms and selected bond distances and angles of **1** and **2**

Table 4
Selected bond distances (Å) and angles (deg) in 1 [a]

Distances

Eu(1)–O(1)	2.60(1)	Eu(1)–O(2)	2.63(1)	Eu(1)–O(3)	2.63(1)
Eu(1)–C(1)	2.93(1)	Eu(1)–C(2)	2.91(2)	Eu(1)–C(3)	2.87(2)
Eu(1)–C(4)	2.88(2)	Eu(1)–C(5)	2.87(2)	Eu(1)–C(18)	2.94(1)
Eu(1)–C(19)	2.88(2)	Eu(1)–C(20)	2.87(2)	Eu(1)–C(21)	2.83(2)
Eu(1)–C(22)	2.87(1)	P(1)–C(1)	1.77(2)	P(1)–C(6)	1.87(2)
P(1)–C(12)	1.83(2)	P(2)–C(18)	1.81(2)	P(2)–C(23)	1.83(1)
P(2)–C(29)	1.82(2)	O(1)–C(35)	1.34(2)	O(1)–C(36)	1.40(2)
O(2)–C(37)	1.41(2)	O(2)–C(38)	1.39(2)	O(3)–C(39)	1.37(3)
O(3)–C(40)	1.39(3)	C(36)–C(37)	1.44(3)	C(38)–C(39)	1.46(3)
Eu(1)–Ccent	2.63	Eu(1)–C'cent	2.62		

Angles

O(1)–Eu(1)–O(2)	62.4(4)	O(1)–Eu(1)–O(3)	123.0(4)
O(2)–Eu(1)–O(3)	61.4(4)	C(1)–P(1)–C(6)	101.6(7)
C(1)–P(1)–C(12)	104.5(7)	C(6)–P(1)–C(12)	98.7(7)
C(18)–P(2)–C(23)	102.8(7)	C(18)–P(2)–C(29)	103.5(7)
C(23)–P(2)–C(29)	97.5(8)	O(1)–Eu(1)–Ccent	102.0
O(1)–Eu(1)–C'cent	105.1	O(2)–Eu(1)–Ccent	111.9
O(2)–Eu(1)–C'cent	123.2	O(3)–Eu(1)–Ccent	106.4
O(3)–Eu(1)–C'cent	97.4	Ccent–Eu(1)–C'cent	124.8

[a] Ccent and C'cent denote the centroids of the cyclopentadienyl rings C(1)–C(5) and C(18)–C(22) respectively.

Table 5
Selected bond distances (Å) and angles (deg) in 2

Distances

Yb(1)–O(1)	2.497(5)	Yb(1)–O(2)	2.470(4)	Yb(1)–O(3)	2.521(5)
Yb(1)–C(1)	2.807(7)	Yb(1)–C(2)	2.777(7)	Yb(1)–C(3)	2.736(7)
Yb(1)–C(4)	2.736(8)	Yb(1)–C(5)	2.776(7)	Yb(1)–C(18)	2.830(7)
Yb(1)–C(19)	2.792(7)	Yb(1)–C(20)	2.738(8)	Yb(1)–C(21)	2.740(8)
Yb(1)–C(22)	2.805(7)	P(1)–C(1)	1.817(6)	P(1)–C(6)	1.862(7)
P(1)–C(12)	1.847(7)	P(2)–C(18)	1.831(7)	P(2)–C(23)	1.852(7)
P(2)–C(29)	1.844(6)	O(1)–C(35)	1.417(9)	O(1)–C(36)	1.432(8)
O(2)–C(37)	1.424(9)	O(2)–C(38)	1.424(9)	O(3)–C(39)	1.405(8)
O(3)–C(40)	1.418(9)	C(36)–C(37)	1.49(1)	C(38)–C(39)	1.48(1)
Yb(1)–Ccent	2.49	Yb(1)–C'cent	2.51		

Angles

O(1)–Yb(1)–O(2)	66.4(1)	O(1)–Yb(1)–O(3)	130.0(2)
O(2)–Yb(1)–O(3)	64.5(2)	C(1)–P(1)–C(6)	104.5(3)
C(1)–P(1)–C(12)	103.6(3)	C(6)–P(1)–C(12)	100.3(3)
C(18)–P(2)–C(23)	102.6(3)	C(18)–P(2)–C(29)	104.9(3)
C(23)–P(2)–C(29)	98.4(3)	O(1)–Yb(1)–Ccent	103.8
O(1)–Yb(1)–C'cent	96.0	O(2)–Yb(1)–Ccent	108.2
O(2)–Yb(1)–C'cent	119.8	O(3)–Yb(1)–Ccent	100.0
O(3)–Yb(1)–C'cent	99.7	Ccent–Yb(1)–C'cent	132.0

are summarized in Tables 2, 3, 4 and 5. Complete lists of bond lengths and angles and tables of hydrogen atom coordinates have been deposited at the Cambridge Crystallographic Data Center.

Acknowledgements

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