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Synthesis and characterization of pentaphenyldiytterbium $\text{Ph}_2\text{Yb}(\text{THF})(\mu\text{-Ph})_3\text{Yb}(\text{THF})_3$

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Abstract

The binuclear ytterbium complex $\text{Ph}_2\text{Yb}(\text{THF})(\mu\text{-Ph})_3\text{Yb}(\text{THF})_3$ (**1**) was obtained in reactions of naphthalenyttterbium $\text{C}_{10}\text{H}_8\text{Yb}(\text{THF})_2$ with diphenylmercury or triphenylbismuth in THF. An X-ray crystallographic study ($a = 11.099(2)$, $b = 19.876(4)$, $c = 19.723(4)$ Å, $\beta = 103.33(3)^\circ$, $Z = 2$, space group $P2_1$) showed that the molecule of **1** has two Yb atoms coupled by three bridging Ph groups, which are linked with the first Yb atom by an η^1 bond and with the second one by an unsymmetrical η^2 bond. In addition the first Yb atom has an η^1 bond with two terminal Ph groups and one coordinated THF molecule whereas the second Yb atom is linked with three THF molecules. The coordination of both Yb atoms is a distorted octahedron. In the crystal, there are two symmetrically independent molecules of **1** with a similar structure. The Yb–C (terminal Ph) bond length is 2.388–2.463 Å. The η^1 - and η^2 -Yb–C (bridging Ph) bond distance varies in the ranges 2.475–2.584, 2.547–2.751 and 2.877–3.250 Å. The magnetic moment μ_{eff} (per Yb^{III} atom) is $4.0 \pm 0.05 \mu_{\text{B}}$. Reactions of **1** with water, HCl, Br_2 , MeI and CO_2 give benzene, bromobenzene, toluene and PhCOOH , respectively.

Introduction

Attempts to determine the structure of σ -aryl complexes of rare earth elements of the R_2Ln or R_3Ln type have been undertaken repeatedly [1,2]. Only one such compound, $(2\text{-Me}_2\text{NCH}_2\text{C}_6\text{H}_4)_3\text{Lu}$, with chelating aminotolyl ligands, has been characterized by X-ray crystallography [3]. In other cases, the structures of the compounds were suggested on the basis of indirect data [2,4–7].

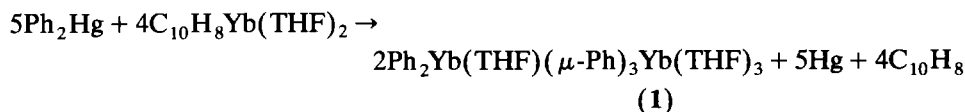
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A suitable route to R_xLn compounds is transmetallation reactions of organomercurials R_2Hg and metallic lanthanoids [4–7]. This method led to pentafluorophenyl, tetrafluorophenyl, ethynyl [4,5], cymantrenyl, benzenechromiumtricarbonyl and carboranyl [6,7] derivatives of lanthanoids. Diphenylmercury does not react with metallic ytterbium according to the data of ref. 5. According to other data [7], such reaction occurs but very slowly, yielding diphenylytterbium in low yield. Ytterbium activated by methylenediiodide interacts with Ph_2Hg violently and yields 75% of Ph_2Yb during 24 h at room temperature [2]. In both cases, conclusions on diphenylytterbium formation and its yield were made on the products of hydrolysis of the reaction mixture.

We have shown by the reaction with $(Ph_3Ge)_2Hg$ [8] that naphthaleneytterbium [9] is more active in transmetallation reactions than free ytterbium. In this paper we report on the reactions of naphthaleneytterbium with diphenylmercury and triphenylbismuth, which gave an opportunity to isolate and carry out the first X-ray investigation of the phenyl complex of a rare earth metal, $Ph_2Yb(THF)(\mu-Ph)_3Yb(THF)_3$ (**1**) [10].

Results and discussion

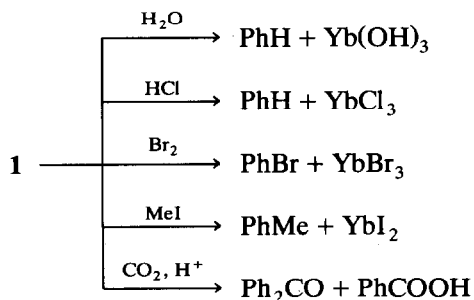
We have found that reaction of $C_{10}H_8Yb(THF)_2$ with diphenylmercury in THF is completed during 2 h at room temperature. Naphthalene, mercury and pentaphenyldiytterbium (**1**) are the main products of the reaction:



Product **1** is precipitated with hexane from THF solution as a pyrophoric fawn powder in 80% yield. Recrystallization of the product in the THF/ether mixture gives red paramagnetic crystals, unstable in air, with m.p. 104–106°C (dec.). The relation of five Ph ligands per two metal atoms in the molecule of **1** means that it contains both Yb^{II} and Yb^{III} atoms. This is confirmed by the μ_{eff} value, which is equal to $3.1 \pm 0.05 \mu_B$ for all Yb atoms or $4.0 \pm 0.05 \mu_B$ for Yb^{III} . The last value is typical for Yb^{III} derivatives [1].

The complex **1** was also obtained in the reaction of $C_{10}H_8Yb(THF)_2$ with Ph_3Bi under similar conditions, but this reaction is slower and gives a lower yield of **1** than the reaction with Ph_2Hg .

Hydrolysis and treatment of **1** with HCl give benzene in a quantitative yield. Bromobenzene is formed in the reaction with Br_2 . Interaction of **1** with methyl-iodide at room temperature leads to toluene formation.



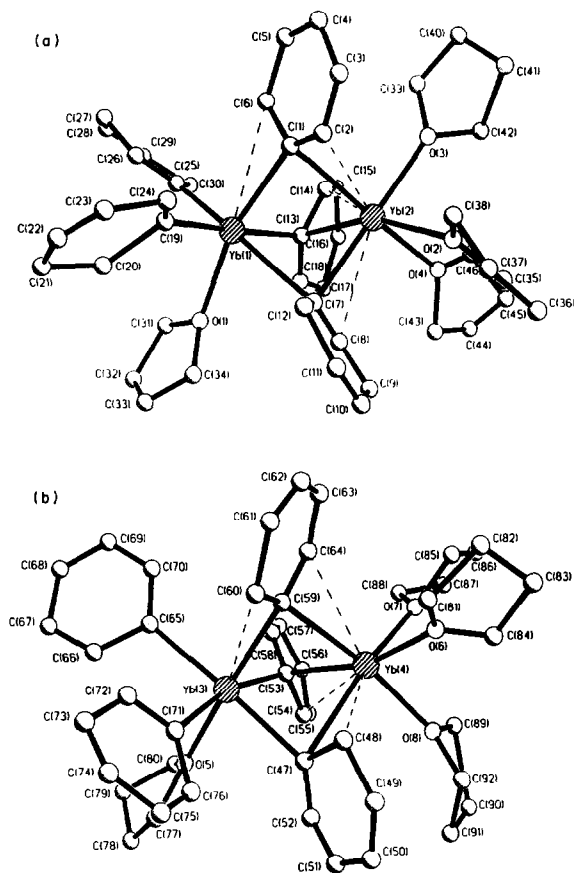


Fig. 1. Ball-and-stick drawing of **1** (two symmetrically independent molecules **1a** and **1b**). Only one position of the disordered THF molecules is shown. Bond distances and angles are listed in Tables 3 and 4, respectively.

Benzophenone and benzoic acid were isolated in approximately equal amounts after the hydrolysis of the products formed in the ready reaction of **1** with carbon dioxide. It should be noted that treatment of PhLi with CO₂ followed by hydrolysis gives the same products [11].

An attempt to use C₁₀H₈Yb(THF)₂ for the preparation of ethyl derivatives of Yb failed. Interaction of the complex with Et₂Hg proceeds easily; more than 97% of mercury is present after 5 h at room temperature. However, the process in this case is accompanied by splitting of C–H and C–C bonds of ethyl ligands, as shown by the presence of methane, ethane, ethylene, propane and other hydrocarbons among the products of hydrolysis of the reaction mixture.

Single-crystal X-ray diffraction analysis showed that the molecule of **1** contains two Yb atoms related by three bridging Ph groups. One Yb atom is additionally bonded with two terminal Ph rings and one THF molecule. The second Yb atom is bonded with three THF molecules (Fig. 1). The coordination of both metal atoms is a distorted octahedron. In the crystal packing there are two symmetrically

Table 1

Summary of X-ray diffraction data

Empirical formula	C ₄₆ H ₅₇ O ₄ Yb ₂
Crystal system	Monoclinic
Space group	<i>P</i> 2 ₁
Unit cell dimensions	
<i>a</i> (Å)	11.099(2)
<i>b</i> (Å)	19.876(4)
<i>c</i> (Å)	19.723(4)
β (deg)	103.33(3)
<i>V</i> (Å ³)	4233.7(2)
<i>Z</i> (molecules/cell)	4
Diffractometer	Siemens P3/PC
Radiation (λ , Å)	Mo- <i>K</i> α (0.71069)
Temperature (K)	193
2 θ range (deg)	2.0 to 52.0
Scan type	2 θ - θ
Index ranges	-12 <i>h</i> 12, -7 <i>k</i> 24 0 <i>l</i> 24
Observed reflections	4917 (<i>F</i> > 3.0 σ (<i>F</i>))
Absorption correction	DIFABS
System used	Siemens SHELXTL PLUS
Solution	Direct methods
Weighting scheme	$w^{-1} = \sigma^2(F)$
Final <i>R</i> (obs. data) (%)	6.44
Final <i>R</i> _w (obs. data) (%)	7.54
Goodness of fit	8.07

independent molecules of **1** (**a** and **b**) of similar structure. Relevant data on the geometry are given in Tables 1–4.

The Yb(μ -C)₃Yb fragment has a distorted trigonal bipyramidal structure. Dihedral angles between Yb(1)–C(1)–Yb(2), Yb(1)–C(7)–Yb(2) and Yb(1)–C(13)–Yb(2) planes in **1a** are 123.6, 121.7 and 114.7°, respectively. The analogous angles in molecule **1b** are 112.4, 133.0 and 114.6°. The (μ -C)Yb(μ -C) angles in **1a** range from 80.2 to 86.2°. In the case of **1b** this range is wider (77.7–94.5°). The angles at bridging C(1), C(7), C(13) and C(47), C(53), C(59) atoms are equal (79(1), 81(1), 79(1) and 78(1), 81(1), 80(1)°).

The Yb–C distance in bridging Yb–C–Yb (average value 2.60 Å) exceeds the length of bridging Yb–C(Me) bonds in the dimer (Cp₂YbMe)₂ (2.54 and 2.49 Å) [12] and in the trinuclear complex (Me₅C₅)₄Yb₃(C \equiv CPh)₄ (2.40, 2.52 Å) [13]. As with this complex, compound **1** contains Yb^{II} and Yb^{III} atoms. Differences in the oxidation state of the metal atoms in the trinuclear complex affect the lengths of bridging bonds: μ -C–Yb^{III} bonds are 0.1 Å longer than μ -C–Yb^{II} bonds. In the molecule of **1**, which can be regarded as composed of associated Ph₂Yb and Ph₃Yb molecules, (Ph₂Yb(THF)–Ph₃Yb(THF)₃ or Ph₃Yb(THF)–Ph₂Yb(THF)₃), similar differences in bond lengths are lacking. Thus, Yb(2)–C interatomic distances of two Yb(1)–C–Yb(2) bridges in **1a** are longer than Yb(1)–C distances (Yb(1)–C(1) 2.58(3), Yb(2)–C(1) 2.67(3) Å and Yb(1)–C(13) 2.58(4), Yb(2)–C(13) 2.64(5) Å). In the third bridge there is an opposite relation: the Yb(2)–C(7) bond (2.55(5) Å) is

Table 2

Atom coordinates ($\times 10^4$) and temperature factors ($\text{\AA}^2 \times 10^4$).

Atom	x	y	z	U^a
Yb(1)	2929(1)	4444(2)	5400(1)	37(1)*
Yb(2)	993(1)	5684(2)	4728(1)	38(1)*
Yb(3)	3051(1)	-369(2)	10071(1)	39(1)*
Yb(4)	962(1)	-1552(2)	9488(1)	42(1)*
O(1)	4893(22)	4805(12)	6068(12)	42(6)
O(2)	-564(29)	6331(17)	5225(16)	67(8)
O(3)	-629(26)	5722(17)	3701(15)	69(8)
O(4)	1477(29)	6834(16)	4452(16)	23(7)
O(5)	4969(24)	-665(14)	10732(13)	53(7)
O(6)	-499(25)	-1526(17)	8356(14)	59(7)
O(7)	-659(22)	-2051(12)	9920(12)	47(6)
O(8)	1179(32)	-2769(17)	9214(17)	27(8)
C(1)	624(33)	4359(17)	4761(14)	48(12)*
C(2)	-338(25)	4492(27)	4991(19)	66(17)*
C(3)	-1466(43)	4186(20)	4666(22)	60(17)*
C(4)	-1581(36)	3749(17)	4154(16)	53(13)*
C(5)	-471(37)	3602(27)	3936(24)	73(19)*
C(6)	663(42)	3900(22)	4232(16)	64(16)*
C(7)	2272(42)	5523(24)	5967(27)	90(20)*
C(8)	2704(37)	6106(27)	6063(17)	67(17)*
C(9)	2686(42)	6605(29)	6517(29)	89(24)*
C(10)	2249(53)	6433(32)	7029(32)	135(28)*
C(11)	1497(60)	5822(33)	7077(29)	97(27)*
C(12)	1552(48)	5369(29)	6569(24)	78(22)*
C(13)	3127(43)	5317(20)	4462(15)	24(17)*
C(14)	2568(24)	5369(20)	3799(15)	57(14)*
C(15)	3012(35)	5447(32)	3225(21)	109(25)*
C(16)	4028(58)	5997(42)	3382(25)	182(41)*
C(17)	4768(42)	6191(26)	4120(21)	89(18)*
C(18)	4202(23)	5709(18)	4632(14)	39(10)*
C(19)	2640(55)	3677(31)	6280(26)	94(26)*
C(20)	4034(71)	3316(26)	6664(30)	122(32)*
C(21)	3881(127)	2851(34)	7176(39)	226(75)*
C(22)	2820(88)	2754(34)	7361(38)	161(42)*
C(23)	1337(98)	3058(43)	7084(34)	180(45)*
C(24)	1639(91)	3507(32)	6487(29)	172(48)*
C(25)	3938(41)	3725(25)	4691(28)	89(20)*
C(26)	3682(53)	3051(35)	4978(38)	131(30)*
C(27)	3983(54)	2724(65)	4542(54)	347(79)*
C(28)	4693(48)	2722(34)	4104(36)	149(30)*
C(29)	5027(37)	3308(37)	3859(27)	139(29)*
C(30)	4526(42)	3874(27)	4168(21)	92(20)*
C(31)	6072(45)	4603(35)	5904(25)	97(16)
C(32)	7032(40)	4624(28)	6583(21)	71(12)
C(33)	6306(44)	4717(31)	7139(25)	83(15)
C(34)	5146(34)	4988(19)	6790(18)	40(9)
C(35)A	-1891(48)	6200(26)	4878(26)	18(11)
C(35)B	-529(70)	7052(39)	5323(41)	49(18)
C(36)A	-2443(99)	6189(53)	5616(55)	83(28)
C(36)B	-1406(126)	7270(69)	5867(74)	119(40)
C(37)	-1459(66)	6420(38)	6107(38)	104(22)
C(38)A	-337(70)	6827(39)	5601(41)	45(12)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ^a
C(38)B	-1622(89)	5992(47)	5601(47)	52(24)
C(39)	-719(75)	5568(45)	3132(42)	47(21)
C(40)A	-2257(63)	5341(35)	2775(33)	35(15)
C(40)B	-2204(90)	5759(59)	2588(50)	91(27)
C(41)A	-2843(66)	5532(36)	3066(36)	34(17)
C(41)B	-2672(77)	6051(43)	3003(43)	62(20)
C(42)	-2063(81)	5838(51)	3884(47)	216(34)
C(43)	2510(43)	7322(22)	4873(23)	32(9)
C(44)	2694(48)	7864(22)	4405(22)	43(9)
C(45)	2851(51)	7614(29)	3908(28)	103(15)
C(46)	1545(54)	7014(30)	3754(30)	131(17)
C(47)	3325(31)	-1349(17)	9314(21)	137(23)*
C(48)	2448(43)	-1140(18)	8413(17)	57(17)*
C(49)	2956(44)	-1504(26)	7930(25)	74(20)*
C(50)	3957(48)	-1901(27)	8065(22)	76(21)*
C(51)	4504(33)	-1959(27)	8735(23)	68(20)*
C(52)	4189(33)	-1729(17)	9333(22)	64(16)*
C(53)	2159(28)	-1276(16)	10724(17)	35(11)*
C(54)	2791(35)	-1925(18)	10979(21)	60(15)*
C(55)	2726(49)	-2241(31)	11507(35)	103(28)*
C(56)	1980(54)	-2074(37)	11863(26)	96(29)*
C(57)	1467(45)	-1450(27)	11826(27)	89(22)*
C(58)	1618(46)	-1105(26)	11223(20)	62(29)*
C(59)	836(27)	-205(15)	9507(27)	65(18)*
C(60)	854(41)	334(21)	8996(24)	65(18)*
C(61)	-303(55)	716(25)	8673(27)	80(23)*
C(62)	-1265(49)	614(19)	8816(22)	64(18)*
C(63)	-1393(30)	125(23)	9319(26)	72(19)*
C(64)	-292(29)	-292(17)	9666(17)	43(12)*
C(65)	2939(36)	522(14)	10923(16)	39(13)*
C(66)	4047(33)	845(19)	11285(21)	54(15)*
C(67)	4206(44)	1298(19)	11835(20)	61(17)*
C(68)	3153(46)	1483(23)	12082(22)	70(18)*
C(69)	2125(48)	1188(20)	11770(23)	69(19)*
C(70)	1952(31)	760(19)	11176(18)	43(13)*
C(71)	4094(29)	274(20)	9353(17)	41(13)*
C(72)	4129(43)	1003(17)	9336(17)	56(15)*
C(73)	4714(37)	1438(23)	8985(23)	69(17)*
C(74)	5204(38)	1137(19)	8462(18)	57(15)*
C(75)	5282(32)	446(19)	8417(23)	51(15)*
C(76)	4829(35)	58(20)	8847(19)	52(14)*
C(77)	6154(34)	-544(25)	10582(19)	66(11)
C(78)	7104(39)	-708(23)	11204(21)	66(12)
C(79)	6556(46)	-583(36)	11783(27)	105(17)
C(80)	5279(46)	-861(27)	11481(24)	80(15)
C(81)	-809(53)	-1016(30)	7889(31)	101(19)
C(82)	-2201(70)	-1119(37)	7720(42)	167(28)
C(83)	-2311(82)	-1818(46)	7530(47)	209(38)
C(84)	-1022(49)	-2058(29)	7997(28)	94(16)
C(85)	-1972(57)	-1931(36)	9685(36)	129(23)
C(86)	-2425(48)	-2515(27)	10035(26)	89(15)
C(87)	-1579(57)	-2735(34)	10653(32)	98(19)
C(88)	-646(15)	-2287(85)	10612(78)	379(73)
C(89)	927(69)	-3247(37)	9843(40)	99(28)

Table 2 (continued)

Atom	x	y	z	U^a
C(90)	2036(55)	-3830(34)	9557(36)	92(23)
C(91)	2514(60)	-3556(33)	9078(33)	99(21)
C(92)	1624(65)	-2980(35)	8740(38)	82(26)

^a Atoms with an asterisk were refined anisotropically. Equivalent isotropic U is defined as one-third of the trace of the orthogonalized U_{ij} tensor.

shorter than the Yb(1)–C(7) bond (2.60(5) Å). In **1b**, one bridge is symmetrical (Yb(3,4)–C(53) 2.55(3) Å), but two others have short shoulders at the Yb(3) atom (2.52(4), 2.48(3) Å) and long shoulders at the Yb(4) atom (2.75(4), 2.68(3) Å).

Significant asymmetry of bridging Ph ring positions with respect to Yb atoms is observed in the molecule of **1**. The middle planes of C(1–6), C(7–12) and C(13–18) rings in **1a** and C(47–52), C(53–58) and C(59–65) rings in **1b** are oriented to the planes of the corresponding Yb–(μ -C)–Yb fragments so that the angles between these planes are 61°, 73° and 72°, and 70°, 79° and 53°. All three bridging Ph groups in **1a** are bent to the Yb(2) atom (to the Yb(4) in **1b**): the angle between the Yb(2)–C(1) bond and the C(1)–C(4) line (123.5°) is smaller but the Yb(1)–C(1)–C(4) angle (157.6°) is bigger than the 140° that should be present in the case of symmetric positioning of this ring. Corresponding angles for the C(7–12) ring are 122.9° and 156.3°; for the C(13–18) ring, 119.7° and 160.7°. In **1b** decreased angles on the side of the Yb(4) atom are 118.1°, 121.9°, 126.2° and relative increased angles on the side of Yb(3) atom are 151.0°, 156.5° and 152.8°. The Yb(2)–C(2,8,14) distances (2.90(5), 2.99(3), 2.88(3) Å) are significantly shorter than Yb(1)–C(6,12,18) separations (3.18(4), 3.54(4), 3.40(4) Å). Similar differences in the distances between Yb atoms and *ortho*-carbon atoms of bridging Ph groups are also observed in **1b** (Table 3). Note that the shortest Yb–C(*ortho*-) distances (2.90, 2.99, 2.88 Å) are similar to the average length of the Yb–C(η^5 -Cp) bonds in [(η^5 -MeC₅H₄)₂Yb(THF)] [14]. Bridging Yb(1)–C(1), Yb(1)–C(7) and Yb(1)–C(13) bonds are bent 18°, 22° and 20° away from the middle planes of the respective bridging Ph rings. The angles between the planes of these rings and the planes defined by Yb(2)–C(1)–C(2), Yb(2)–C(7)–C(8) and Yb(2)–C(13)–C(14) are 48°, 55° and 55° (46°, 52° and 55° for **1b**).

The marked geometrical features indicate that the bridging bonds in **1** can be regarded as η^1 : η^2 bridges: an η^1 bond of phenyl rings with Yb(1,3) and an η^2 bond with Yb(2,4). Note also the shortened distances of Yb(1)–C(6) (3.18 Å), Yb(3)–C(58) (3.39 Å) and Yb(3)–C(60) (3.16 Å), indicating a weak bonding of the C(1–6) ring with Yb(1) and the C(53–58) and C(59–64) rings with Yb(3). This means that η^2 bonds can also occur in these fragments.

It is possible that interaction between Yb atoms and *ortho*-carbons of bridging phenyl rings is realized via H atoms at these carbons. Unfortunately this question remains unsolved because the position of the protons in **1** were not located objectively.

The Yb(1) atom in **1a** (Yb(3) in **1b**), besides bridging Ph rings, is bonded with two terminal phenyl groups and a molecule of THF (Fig. 1). The Yb–C(Ph terminal) bond lengths of 2.39(6) and 2.44(5) Å for **1a** and 2.46(4) and 2.40(4) Å for **1b** are shorter than the bridging bonds and comparable with those found in the

Table 3

Bond lengths (Å)

Yb(1)...Yb(2)	3.336(5)	O(7)-C(85)	1.444(65)	C(41)A-C(42)	1.754(107)
Yb(1)-O(1)	2.382(27)	O(7)-C(88)	1.440(166)	C(41)B-C(42)	1.765(118)
Yb(1)-C(1)	2.584(33)	O(8)-C(89)	1.639(55)	C(43)-C(44)	1.464(120)
Yb(1)-C(6)	3.183(37)	O(8)-C(92)	1.227(60)	C(44)-C(45)	1.147(75)
Yb(1)-C(7)	2.600(50)	C(1)-C(2)	1.280(51)	C(45)-C(46)	1.848(290)
Yb(1)-C(13)	2.582(37)	C(1)-C(6)	1.394(50)	C(47)-C(48)	1.866(50)
Yb(1)-C(19)	2.388(60)	C(2)-C(3)	1.405(54)	C(47)-C(52)	1.213(50)
Yb(1)-C(25)	2.444(55)	C(3)-C(4)	1.316(53)	C(48)-C(49)	1.413(68)
Yb(2)-O(2)	2.526(35)	C(4)-C(5)	1.426(62)	C(49)-C(50)	1.338(72)
Yb(2)-O(3)	2.382(27)	C(5)-C(6)	1.392(60)	C(50)-C(51)	1.327(59)
Yb(2)-O(4)	2.439(32)	C(7)-C(8)	1.251(68)	C(51)-C(52)	1.382(66)
Yb(2)-C(1)	2.667(35)	C(7)-C(12)	1.607(79)	C(53)-C(54)	1.498(48)
Yb(2)-C(2)	2.901(48)	C(8)-C(9)	1.339(73)	C(53)-C(58)	1.310(60)
Yb(2)-C(7)	2.547(48)	C(9)-C(10)	1.264(91)	C(54)-C(55)	1.232(80)
Yb(2)-C(8)	2.990(34)	C(10)-C(11)	1.489(91)	C(55)-C(56)	1.247(93)
Yb(2)-C(13)	2.644(49)	C(11)-C(12)	1.359(79)	C(56)-C(57)	1.359(89)
Yb(2)-C(14)	2.877(32)	C(13)-C(14)	1.315(40)	C(57)-C(58)	1.417(72)
Yb(3)...Yb(4)	3.317(5)	C(13)-C(18)	1.399(53)	C(59)-C(60)	1.474(61)
Yb(3)-O(5)	2.302(24)	C(14)-C(15)	1.344(55)	C(59)-C(64)	1.371(52)
Yb(3)-C(47)	2.516(39)	C(15)-C(16)	1.550(89)	C(60)-C(61)	1.501(68)
Yb(3)-C(53)	2.546(34)	C(16)-C(17)	1.546(64)	C(61)-C(62)	1.183(85)
Yb(3)-C(59)	2.475(30)	C(17)-C(18)	1.620(59)	C(62)-C(63)	1.420(66)
Yb(3)-C(60)	3.164(41)	C(19)-C(20)	1.715(88)	C(63)-C(64)	1.504(48)
Yb(3)-C(65)	2.463(32)	C(19)-C(24)	1.313(118)	C(65)-C(66)	1.425(48)
Yb(3)-C(71)	2.395(38)	C(20)-C(21)	1.408(105)	C(65)-C(70)	1.389(55)
Yb(4)-O(6)	2.438(24)	C(21)-C(22)	1.325(172)	C(66)-C(67)	1.389(55)
Yb(4)-O(7)	2.378(26)	C(22)-C(23)	1.720(135)	C(67)-C(68)	1.414(73)
Yb(4)-O(8)	2.502(27)	C(23)-C(24)	1.575(109)	C(68)-C(69)	1.305(65)
Yb(4)-C(47)	2.751(37)	C(25)-C(26)	1.506(88)	C(69)-C(70)	1.424(56)
Yb(4)-C(48)	3.081(44)	C(25)-C(30)	1.374(74)	C(71)-C(72)	1.450(52)
Yb(4)-C(53)	2.552(31)	C(26)-C(27)	1.185(139)	C(71)-C(76)	1.489(56)
Yb(4)-C(59)	2.682(29)	C(27)-C(28)	1.296(119)	C(72)-C(73)	1.363(62)
Yb(4)-C(64)	2.925(34)	C(28)-C(29)	1.346(96)	C(73)-C(74)	1.407(63)
O(1)-C(31)	1.474(61)	C(29)-C(30)	1.451(84)	C(74)-C(75)	1.380(54)
O(1)-C(34)	1.434(41)	C(31)-C(32)	1.508(58)	C(75)-C(76)	1.327(60)
O(2)-C(35)A	1.497(58)	C(32)-C(33)	1.515(71)	C(77)-C(78)	1.459(51)
O(2)-C(35)B	1.445(85)	C(33)-C(34)	1.418(58)	C(78)-C(79)	1.434(74)
O(2)-C(38)A	1.225(79)	C(35)A-C(36)A	1.705(132)	C(79)-C(80)	1.510(71)
O(2)-C(38)B	1.670(111)	C(35)B-C(36)B	1.665(179)	C(81)-C(82)	1.517(95)
O(3)-C(39)	1.145(90)	C(36)A-C(37)	1.363(121)	C(82)-C(83)	1.435(118)
O(3)-C(42)	1.726(101)	C(36)B-C(37)	1.760(158)	C(83)-C(84)	1.586(97)
O(4)-C(43)	1.583(52)	C(37)-C(38)A	1.944(114)	C(85)-C(86)	1.497(91)
O(4)-C(46)	1.441(60)	C(37)-C(38)B	1.291(119)	C(86)-C(87)	1.424(73)
O(5)-C(77)	1.434(50)	C(39)-C(40)A	1.749(101)	C(87)-C(88)	1.381(177)
O(5)-C(80)	1.489(54)	C(39)-C(40)B	1.788(118)	C(89)-C(90)	1.869(155)
O(6)-C(81)	1.359(66)	C(40)A-C(41)A	1.034(108)	C(90)-C(91)	1.304(122)
O(6)-C(84)	1.329(62)	C(40)B-C(41)B	1.213(145)	C(91)-C(92)	1.559(140)

other aryl derivatives of lanthanoids: 2.45 Å in the anion [(2,6-Me₂C₆H₃)₄Ln]⁻ [15], 2.14 Å in the neutral molecule Cp₂YC₆H₄CH₂NMe₂-o [16] and 2.425–2.455 Å in (2-Me₂NCH₂C₆H₄)₃Lu [3].

Table 4

Bond angles (deg)

O(1)–Yb(1)–C(1)	165.0(1.0)	Yb(2)–C(13)–C(18)	123.5(2.6)
Yb(1)–C(7)–C(8)	133.9(3.9)	C(14)–C(13)–C(18)	112.1(3.4)
O(1)–Yb(1)–C(7)	79.9(1.1)	C(13)–C(14)–C(15)	131.7(3.3)
C(1)–Yb(1)–C(7)	85.4(1.2)	C(14)–C(15)–C(16)	107.7(3.9)
O(1)–Yb(1)–C(13)	88.6(1.1)	C(15)–C(16)–C(17)	124.6(4.7)
C(1)–Yb(1)–C(13)	86.2(1.2)	C(16)–C(17)–C(18)	104.0(4.0)
C(7)–Yb(1)–C(13)	80.4(1.5)	C(13)–C(18)–C(17)	127.6(2.6)
O(1)–Yb(1)–C(19)	92.9(1.4)	Yb(1)–C(19)–C(20)	109.2(3.8)
C(1)–Yb(1)–C(19)	91.6(1.5)	Yb(1)–C(19)–C(24)	131.0(4.4)
C(7)–Yb(1)–C(19)	96.7(1.5)	C(20)–C(19)–C(24)	119.8(5.2)
C(13)–Yb(1)–C(19)	176.4(1.8)	C(19)–C(20)–C(21)	110.2(7.6)
O(1)–Yb(1)–C(25)	90.6(1.2)	C(20)–C(21)–C(22)	123.7(9.1)
C(1)–Yb(1)–C(25)	102.5(1.2)	C(21)–C(22)–C(23)	135.1(6.7)
C(7)–Yb(1)–C(25)	160.0(1.7)	C(22)–C(23)–C(24)	94.8(7.0)
C(13)–Yb(1)–C(25)	81.8(1.5)	C(19)–C(24)–C(23)	136.1(7.2)
C(19)–Yb(1)–C(25)	101.4(1.9)	Yb(1)–C(25)–C(26)	98.9(3.9)
O(2)–Yb(2)–O(3)	82.1(1.0)	Yb(1)–C(25)–C(30)	131.4(3.7)
O(2)–Yb(2)–O(4)	79.3(1.1)	C(26)–C(25)–C(30)	129.6(5.3)
O(3)–Yb(2)–O(4)	86.7(1.1)	C(25)–C(26)–C(27)	96.0(7.9)
O(2)–Yb(2)–C(1)	111.7(1.1)	C(26)–C(27)–C(28)	143.3(9.0)
O(3)–Yb(2)–C(1)	87.8(1.2)	C(27)–C(28)–C(29)	119.7(8.0)
O(4)–Yb(2)–C(1)	166.9(1.0)	C(28)–C(29)–C(30)	110.9(5.0)
O(2)–Yb(2)–C(7)	88.4(1.3)	C(25)–C(30)–C(29)	116.2(4.9)
O(3)–Yb(2)–C(7)	164.8(1.4)	O(1)–C(31)–C(32)	106.2(3.9)
O(4)–Yb(2)–C(7)	103.2(1.3)	C(31)–C(32)–C(33)	105.2(3.8)
C(1)–Yb(2)–C(7)	84.7(1.2)	C(32)–C(33)–C(34)	105.6(3.7)
O(2)–Yb(2)–C(13)	160.3(1.1)	O(1)–C(34)–C(33)	109.6(3.5)
O(3)–Yb(2)–C(13)	112.1(1.0)	O(2)–C(35)A–C(36)A	97.0(4.3)
O(4)–Yb(2)–C(13)	87.8(1.2)	O(2)–C(35)B–C(36)B	110.1(7.2)
C(1)–Yb(2)–C(13)	83.3(1.2)	C(35)A–C(36)A–C(37)	102.5(7.8)
C(7)–Yb(2)–C(13)	80.2(1.3)	C(35)B–C(36)B–C(37)	89.1(7.8)
O(5)–Yb(3)–C(47)	85.1(1.0)	C(36)A–C(37)–C(38)A	106.1(6.9)
O(5)–Yb(3)–C(53)	87.1(1.0)	C(36)B–C(37)–C(38)B	115.6(8.0)
C(47)–Yb(3)–C(53)	82.2(1.2)	O(2)–C(38)A–C(37)	84.8(4.9)
O(5)–Yb(3)–C(59)	168.9(1.2)	O(2)–C(38)B–C(37)	95.2(5.7)
C(47)–Yb(3)–C(59)	94.5(1.1)	O(3)–C(39)–C(40)A	108.6(6.8)
C(53)–Yb(3)–C(59)	81.9(1.2)	O(3)–C(39)–C(40)B	113.0(7.3)
O(5)–Yb(3)–C(65)	89.0(1.1)	C(39)–C(40)A–C(41)A	111.2(9.0)
C(47)–Yb(3)–C(65)	173.4(1.0)	C(39)–C(40)B–C(41)B	100.1(7.2)
C(53)–Yb(3)–C(65)	94.5(1.2)	C(40)A–C(41)A–C(42)	113.4(6.8)
C(59)–Yb(3)–C(65)	90.6(1.3)	C(40)B–C(41)B–C(42)	115.0(7.8)
O(5)–Yb(3)–C(71)	87.8(1.0)	O(3)–C(42)–C(41)A	92.6(5.5)
C(47)–Yb(3)–C(71)	85.5(1.3)	O(3)–C(42)–C(41)B	89.3(5.7)
C(53)–Yb(3)–C(71)	167.0(1.2)	O(4)–C(43)–C(44)	108.1(4.0)
C(59)–Yb(3)–C(71)	103.3(1.3)	C(43)–C(44)–C(45)	106.8(4.6)
C(65)–Yb(3)–C(71)	97.3(1.2)	C(44)–C(45)–C(46)	98.7(5.0)
O(6)–Yb(4)–O(7)	86.5(0.9)	O(4)–C(46)–C(45)	102.4(4.8)
O(6)–Yb(4)–O(8)	84.2(1.1)	Yb(3)–C(47)–Yb(4)	77.9(1.1)
O(7)–Yb(4)–O(8)	78.0(1.0)	Yb(3)–C(47)–C(48)	106.5(1.9)
O(6)–Yb(4)–C(47)	109.6(1.1)	Yb(4)–C(47)–C(48)	81.3(1.9)
O(7)–Yb(4)–C(47)	158.0(0.9)	Yb(3)–C(47)–C(52)	131.8(2.8)
O(8)–Yb(4)–C(47)	88.4(1.1)	Yb(4)–C(47)–C(52)	132.4(2.9)
O(6)–Yb(4)–C(53)	163.7(1.1)	C(48)–C(47)–C(52)	113.7(3.7)
O(7)–Yb(4)–C(53)	91.0(0.9)	C(47)–C(48)–C(49)	109.1(3.2)
O(8)–Yb(4)–C(53)	111.0(1.0)	C(48)–C(49)–C(50)	127.6(4.1)
C(47)–Yb(4)–C(53)	77.7(1.1)	C(49)–C(50)–C(51)	114.7(4.7)

Table 4 (continued)

O(6)–Yb(4)–C(59)	88.0(1.2)	C(50)–C(51)–C(52)	132.3(4.3)
O(7)–Yb(4)–C(59)	111.4(1.1)	C(47)–C(52)–C(51)	122.3(3.9)
O(8)–Yb(4)–C(59)	167.5(1.1)	Yb(3)–C(53)–Yb(4)	81.2(1.0)
C(47)–Yb(4)–C(59)	84.8(1.1)	Yb(3)–C(53)–C(54)	125.0(2.5)
C(53)–Yb(4)–C(59)	77.9(1.2)	Yb(4)–C(53)–C(54)	103.7(3.5)
Yb(1)–O(1)–C(31)	122.8(2.4)	Yb(3)–C(53)–C(58)	119.6(2.9)
Yb(1)–O(1)–C(34)	124.8(2.1)	Yb(4)–C(53)–C(58)	123.0(2.5)
C(31)–O(1)–C(34)	107.8(2.7)	C(54)–C(53)–C(58)	103.4(2.1)
Yb(2)–O(2)–C(35)A	115.1(2.6)	C(53)–C(54)–C(55)	127.5(4.6)
Yb(2)–O(2)–C(35)B	123.9(3.9)	C(54)–C(55)–C(56)	121.0(5.8)
Yb(2)–O(2)–C(38)A	125.6(4.1)	C(55)–C(56)–C(57)	122.5(6.4)
C(35)A–O(2)–C(38)A	117.3(4.6)	C(56)–C(57)–C(58)	110.9(5.1)
Yb(2)–O(2)–C(38)B	125.5(3.6)	C(53)–C(58)–C(57)	132.0(4.7)
C(35)B–O(2)–C(38)B	109.9(5.2)	Yb(3)–C(59)–Yb(4)	79.9(0.8)
Yb(2)–O(3)–C(39)	134.6(4.6)	Yb(3)–C(59)–C(60)	103.6(2.4)
Yb(2)–O(3)–C(42)	112.3(3.3)	Yb(4)–C(59)–C(60)	135.0(3.3)
C(39)–O(3)–C(42)	111.4(5.4)	Yb(3)–C(59)–C(64)	138.2(3.3)
Yb(2)–O(4)–C(43)	128.9(2.2)	Yb(4)–C(59)–C(64)	86.0(1.9)
Yb(2)–O(4)–C(46)	120.5(2.5)	C(60)–C(59)–C(64)	113.9(3.0)
C(43)–O(4)–C(46)	99.1(5.0)	C(59)–C(60)–C(61)	120.9(4.2)
Yb(3)–O(5)–C(77)	127.8(2.2)	C(60)–C(61)–C(62)	122.9(4.8)
Yb(3)–O(5)–C(80)	126.9(2.6)	C(61)–C(62)–C(63)	121.6(4.4)
C(77)–O(5)–C(80)	103.6(2.9)	C(62)–C(63)–C(64)	119.6(3.1)
Yb(4)–O(6)–C(81)	130.4(3.0)	C(59)–C(64)–C(63)	121.0(3.4)
Yb(4)–O(6)–C(84)	125.9(2.8)	Yb(3)–C(65)–C(66)	119.5(2.8)
C(81)–O(6)–C(84)	102.7(3.7)	Yb(3)–C(65)–C(70)	131.2(2.4)
Yb(4)–O(7)–C(85)	127.4(3.2)	C(66)–C(65)–C(70)	109.1(3.0)
Yb(4)–O(7)–C(88)	129.3(6.1)	C(65)–C(66)–C(67)	128.5(4.0)
C(85)–O(7)–C(88)	98.4(7.1)	C(66)–C(67)–C(68)	118.1(3.9)
Yb(4)–O(8)–C(89)	110.8(3.2)	C(67)–C(68)–C(69)	115.1(2.3)
Yb(4)–O(8)–C(92)	124.8(3.6)	C(68)–C(69)–C(70)	126.4(4.9)
C(89)–O(8)–C(92)	123.2(3.8)	C(65)–C(70)–C(69)	122.1(3.4)
Yb(1)–C(1)–Yb(2)	78.9(1.0)	Yb(3)–C(71)–C(72)	124.4(2.7)
Yb(1)–C(1)–C(2)	128.8(2.2)	Yb(3)–C(71)–C(76)	131.1(2.7)
Yb(2)–C(1)–C(2)	87.1(2.9)	C(72)–C(71)–C(76)	104.5(3.3)
Yb(1)–C(1)–C(6)	102.1(2.6)	C(71)–C(72)–C(73)	131.6(4.0)
Yb(2)–C(1)–C(6)	126.6(2.5)	C(72)–C(73)–C(74)	114.3(3.9)
C(2)–C(1)–C(6)	125.0(3.7)	C(73)–C(74)–C(75)	120.8(3.9)
C(1)–C(2)–C(3)	118.5(4.0)	C(74)–C(75)–C(76)	119.9(4.1)
C(2)–C(3)–C(4)	123.6(4.4)	C(71)–C(76)–C(75)	127.4(3.7)
C(3)–C(4)–C(5)	115.4(3.8)	O(5)–C(77)–C(78)	108.0(3.4)
C(4)–C(5)–C(6)	123.2(4.2)	C(77)–C(78)–C(79)	105.9(3.8)
C(1)–C(6)–C(5)	114.2(4.2)	C(78)–C(79)–C(80)	98.9(4.0)
Yb(1)–C(7)–Yb(2)	80.8(1.5)	O(5)–C(80)–C(79)	106.1(4.1)
Yb(2)–C(7)–C(8)	98.0(3.0)	O(6)–C(81)–C(82)	97.7(4.9)
Yb(1)–C(7)–C(12)	113.4(3.1)	C(81)–C(82)–C(83)	102.1(6.3)
Yb(2)–C(7)–C(12)	118.1(2.8)	C(82)–C(83)–C(84)	97.4(6.0)
C(8)–C(7)–C(12)	107.7(4.4)	O(6)–C(84)–C(83)	107.1(5.0)
C(7)–C(8)–C(9)	136.3(4.9)	O(7)–C(85)–C(86)	98.3(7.1)
C(8)–C(9)–C(10)	113.7(5.5)	C(85)–C(86)–C(87)	114.0(4.8)
C(9)–C(10)–C(11)	126.5(5.6)	C(86)–C(87)–C(88)	96.2(7.6)
C(10)–C(11)–C(12)	111.9(5.5)	O(7)–C(88)–C(87)	114.9(9.3)
C(7)–C(12)–C(11)	122.1(5.1)	O(8)–C(89)–C(90)	83.8(6.0)
Yb(1)–C(13)–Yb(2)	79.3(1.3)	C(89)–C(90)–C(91)	111.5(6.5)
Yb(1)–C(13)–C(14)	131.8(3.0)	C(90)–C(91)–C(92)	107.0(6.9)
Yb(2)–C(13)–C(14)	86.4(2.6)	O(8)–C(92)–C(91)	104.7(5.9)
Yb(1)–C(13)–C(18)	114.2(2.1)		

The Yb–O(THF) bonds typically exhibit a wide range of distances, and the distances observed in **1** (2.30–2.53 Å) are not unusual. The O–Yb(1,3)–C(terminal) angles are close to 90° whereas the C(terminal)–Yb(1,3)–C(terminal) angles are slightly increased. The O–Yb–O angles at Yb(2,4) atoms are in the range 78.0–86.7°.

At the Yb(2) atom, there are two THF molecules disordered over two positions, but this disordering does not alter the fundamental aspects of the structure. The disorder is indicated in Table 2 by A and B atoms. In Fig. 1, one of the positions of the THF molecules is shown. The bond distances and bond angles are given in Tables 3 and 4.

Thus, the first X-ray diffraction study of phenyl derivatives of ytterbium obtained via $C_{10}H_8Yb(THF)_2$ shows that the structure of this compound appears to be more complex than the simple Ph_2Yb species, formation of which was proposed in the reactions of Ph_2Hg with metallic ytterbium.

In both reactions of $C_{10}H_8Yb(THF)_2$ with diphenylmercury and triphenylbismuth, in addition to compound **1**, a small amount of yellow crystals with m.p. 120–121°C (dec.) was isolated. The IR spectrum, value of $\mu_{eff} = 4.0\mu_B$, and ytterbium content correspond to the triphenylytterbium complex $Ph_3Yb(THF)_3$. The structure of this product is now under investigation.

Experimental

All manipulations were carried out with use of standard Schlenk procedures under vacuum. Solvents (THF, ether, toluene) were dried and deoxygenated by Na–benzophenone and vacuum transferred prior to use. Infrared spectra were recorded on a Perkin–Elmer 577 spectrometer. The magnetic moment was measured as described previously [17]. Chromatographic analyses were carried out using a Tsvet-104 instrument, non-volatile organic products were analyzed on a liquid chromatograph Milikhrom-1A. Naphthalenyttterbium $C_{10}H_8Yb(THF)_2$ was prepared according to the published procedure [9]. The ytterbium halides and hydroxide formed in the reactions were identified by titrimetric analysis.

*Synthesis of $Ph_2Yb(THF)(\mu-Ph)_3Yb((THF)_3)$ (**1**)*

$C_{10}H_8Yb(THF)_2$ powder (1.89 g, 4.22 mmol) was added to a solution of 1.50 g (4.22 mmol) of Ph_2Hg in 15 mL of THF. The reaction mixture was stirred for 2 h. The color of the mixture turned from black to red-brown. The mixture was centrifuged and a solution was decanted from the gray precipitate. Mercury (0.83 g, 98%) and ytterbium (0.15 g, 20%) were found in the precipitate. The solution was concentrated in vacuum to 4 mL, and 20 mL of hexane was added. The light-brown precipitate formed was reprecipitated from THF solution with hexane to give 1.39 g (81%) of a brown microcrystalline solid, m.p. 100–101°C (dec.). Elution of the precipitate with THF/ether (1 : 3) resulted in 0.12 g of a yellow solid of m.p. 120–121°C (dec.). Yb content 28.0% (calc. for $Ph_3Yb(THF)_3$ 27.88%). IR (Nujol): 3020w, 1405m, 1222w, 1070w, 1050m, 1030w, 1015s, 985w, 910br,w, 860br,s, 715m, 700s, 670s, 665w, 625m, 425m cm^{-1} . Upon cooling of the eluent to –5°C for 24 h, the product **1** precipitated as red crystals. Yield: 0.40 g (23%), m.p. 104–106°C (dec.), μ_{eff} (per Yb^{III}) $4.0 \pm 0.05 \mu_B$ (293 K). Anal. Found: C, 53.02; H, 5.61; Yb, 34.20%. $C_{46}H_{57}O_4Yb_2$ calc.: C, 54.16; H, 5.63; Yb, 33.93%. IR (Nujol):

3020w, 1405m, 1218w, 1065w, 1040w, 1025s, 980m, 910w, 870br,m, 730w, 715m, 700s, 670s, 610m, 430m cm^{-1} . The reaction of $\text{C}_{10}\text{H}_8\text{Yb}(\text{THF})_2$ (0.61 g, 1.37 mmol) with triphenylbismuth (0.60 g, 1.37 mmol) was carried out analogously: THF, 20°C, 6 h, yield of 1 0.45 g (64%).

Reactions of 1 with HCl and H₂O

Gaseous HCl (0.27 g, 7.29 mmol) was condensed into the suspension of 1 (0.32 g, 0.32 mmol) in 5 mL of toluene. The color of the solid changed from red to blue. After heating the mixture in a boiling water bath for 1.5 h, the solid became white. The solvent and volatile products were removed by condensation in vacuum. The residue was identified as YbCl_3 , yield 0.17 g (99%). In the condensate, 0.12 g (98%) of benzene was found by the use of gas chromatography (GC).

Under similar conditions, 0.54 g (0.53 mmol) of 1 and 0.10 g (excess) of water gave 0.18 g (88%) of benzene, 0.02 g (14%) of THF and 0.23 g (98%) of ytterbium hydroxide.

Reaction of 1 with bromine

A solution of Br_2 (0.43 g, 2.72 mmol) in 3 mL of THF was added to a solution of 1 (0.45 g, 0.44 mmol) in 3 mL of THF. A yellow precipitate of YbBr_3 formed. After centrifuging, the solution was decanted from the precipitate. Yield of YbBr_3 : 0.36 g (100%). In the solution, 0.34 g (99%) of bromobenzene was determined by the use of GC.

Reaction of 1 with MeI

Methyl iodide (0.70 g, 0.45 mmol) was added to a solution of 1 (0.46 g, 0.45 mmol) in 5 mL of dimethoxyethane and the mixture was heated at 100°C for 1 h. The color of the mixture changed from brown to light yellow and a white solid precipitated. The solvent and volatile product were removed by condensation in vacuum and analyzed by the use of GC. Toluene (0.13 g, 62%) and THF (0.12 g, 92%) were found. The precipitate was identified as YbI_2 (0.13 g, 100%).

Reaction of 1 with CO₂

A solution of 1 (0.46 g, 0.46 mmol) in 12 mL of THF was shaken in a carbon dioxide atmosphere under ambient conditions for 4 h. The mixture was decolorized and a yellow insoluble solid deposited. The solvent was removed in vacuum and the residue was hydrolyzed with 10 mL of diluted HCl. The mixture formed after hydrolysis was extracted with ether (2×3 mL) and the extract was analyzed by the use of liquid chromatography. Benzophenone (0.13 g, 15%) and PhCOOH (0.14, 50%) were found.

X-Ray crystal structure determination

Single crystals of 1 were grown from solution in a THF/ether (1:3) mixture by cooling to -5°C . Intensity data were collected on a Siemens P3/PC diffractometer at -80°C . A summary of the crystal data is given in Table 1.

The structure was solved by using the usual combination of direct methods and difference Fourier techniques. The four Yb atoms were located in the initial E map and the remaining atoms in successive difference maps. Although the symmetry of the four Yb atoms is $P2_1/c$, the experimentally observed absences and

statistics of intensities indicate the $P2_1$ group. It quickly became obvious that several THF molecules were affected by disorder. Inspection of difference maps eventually resulted in the pattern of disorder indicated in the Tables by atoms A and B. The disorder involves two of the THF molecules at the Yb(2) atom.

The full-matrix least-squares refinement was completed by using a partial occupancy parameter ($\mu = 0.5$) for the disordered carbon atoms of the THF molecules, the anisotropic thermal parameter for the Yb atoms and the C atoms of Ph groups and isotropic thermal parameters for the oxygen and carbon atoms of the THF molecules.

Positions of the hydrogen atoms (besides the H atoms of the disordered THF molecules) were calculated by using the usual geometrical conditions. These were taken into account in the calculations of F_{cal} , but not refined.

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