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Interaction of naphthaleneytterbium with tetraphenyltin. Molecular structure of $Ph_3SnYb(THF)_2(\mu\text{-}Ph)_3Yb(THF)_3$

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Abstract

Reaction of naphthaleneytterbium with Ph_4Sn in THF yields $(Ph_3Sn)_2Yb(THF)_4$ and the heteroleptic complex $Ph_3SnYb(THF)_2(\mu\text{-}Ph)_3Yb(THF)_3$ (1), which can be isolated by crystallization from THF/ether solution. The product 1 forms triclinic crystals in space group $P\bar{1}$ with $a = 11.123(2)$, $b = 14.078(3)$, $c = 18.774(4)$ Å, $\alpha = 101.94(2)$, $\beta = 96.20(2)$, $\gamma = 109.85(2)°$, $Z = 2$. Least-squares refinement on the basis of 4955 reflections led to a final R value of 0.027. The molecule of 1 contains two Yb atoms connected by three bridging Ph rings. One of Yb atoms is bonded with Ph_3Sn unit (Sn–Yb bond length is 3.379(1) Å) and two THF molecules. The second Yb atom is surrounded by three molecules of coordinated THF. Coordination of each ytterbium atom is distorted octahedron. The proposed reaction scheme includes two-electron oxidation of naphthaleneytterbium, formation of $Ph_3SnYbPh$ and PhYbPh intermediates and their following association.

We have recently reported that the reaction of naphthaleneytterbium, $C_{10}H_8Yb(THF)_2$, with tetraphenyltin results in formation of two novel ytterbium complexes. One of them, $(Ph_3Sn)_2Yb(THF)_4$, has been characterized in [1]. Here we report the structure of the second product and discuss the possible scheme of the reaction [2].

Results

On shaking an equimolar mixture of $C_{10}H_8Yb(THF)_2$ and Ph_4Sn in THF at room temperature for 2 days, the precipitate of naphthaleneytterbium dissolves almost completely. The resulting mixture contains, besides free naphthalene, two main organometallic products, which can be separated by crystallization from THF/ether solution. One of them, $(Ph_3Sn)_2Yb(THF)_4$, is isolated in 36% yield as yellow crystals, sparingly soluble in ether [1]. The second product has been

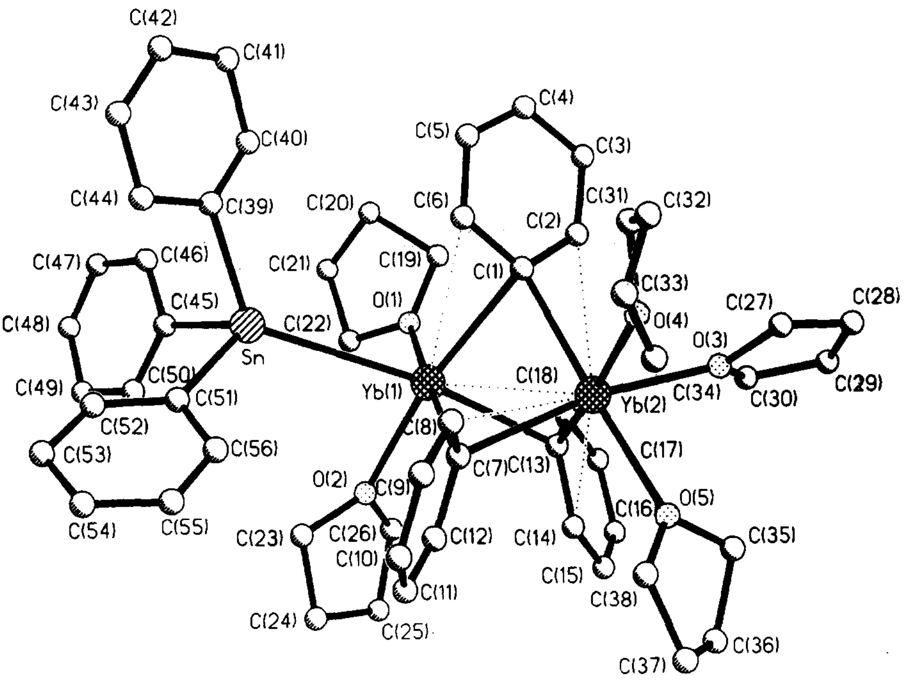


Fig. 1. Molecular structure of $Ph_3SnYb(THF)_2(\mu\text{-}Ph)_3Yb(THF)_3$ with atom labels.

identified as $Ph_3SnYb(THF)_2(\mu\text{-}Ph)_3Sn(THF)_3$ (1). It forms dark-ruby, almost black diamagnetic crystals on cooling the THF/ether solution. Compound **1** melts with decomposition on heating at 110–115 °C. The reaction of **1** with bromine gives bromobenzene, Ph_3SnBr and $YbBr_3$.

The molecular structure of **1** is represented in Fig. 1 together with the atomic numbering scheme. The bond distances and angles are given in Tables 2 and 3. Two Yb atoms in **1** are coupled by three Ph bridges. The Yb(1) is also bonded with Sn atom of Ph_3Sn group and two THF molecules. The Yb(2) atom is coordinated by three THF molecules. The coordination of each Yb atom is a distorted octahedron. The structure of the central fragment $Yb(\mu\text{-}Ph)_3Yb$ in **1** is similar to that in the molecule of $Ph_2Yb(THF)(\mu\text{-}Ph)_3Yb(THF)_3$ (**2**) described recently [3]. The structures of **1** and **2** differ from each other around the Yb(1) atom. In the **1** molecule, the Yb(1) atom is bonded with the Ph_3Sn fragment and two THF molecules, whereas in the **2** molecule Yb(1) is connected with two Ph groups and one THF molecule. The molecule of **1** can be regarded formally as an association of two units of divalent ytterbium, $Ph_3SnYbPh(THF)_2$ and $Ph_2Yb(THF)_3$, while the molecule of **2** looks like an association of $Ph_2Yb(THF)_3$ and $Ph_3Yb(THF)_2$, containing Yb^{II} and Yb^{III}, respectively.

The Yb–Sn bond length in **1** 3.379(1) Å is significantly longer than 3.305(1) Å found in $(Ph_3Sn)_2Yb(THF)_4$ [1]. It seems that this difference is a result of steric repulsion of the ligands at Yb(1) in **1**. The observed Sn–Yb bond length is somewhat longer than the sum of the radii of these atoms by Pauling of 3.12 Å [4].

The central $Yb(\mu\text{-}C)_3Yb$ moiety in **1** has the structure of distorted trigonal bipyramid. The dihedral angles between Yb(1)C(1)Yb(2), Yb(1)C(7)Yb(2) and

Yb(1)C(13)Yb(2) planes of 124.8, 117.2 and 118.0 ° respectively are close to those in molecule **2** (123.6, 121.7 and 114.7 °). The ranges of value of valent C–Yb–C angles at Yb(1) and Yb(2) atoms are 84.0(3)–87.8(3) and 85.3(3)–88.7(3) °, respectively. The corresponding values for **2** are somewhat smaller (79–81 °). The bridging Yb–C bonds in **1** are actually symmetrical: Yb(1)–C(1) 2.62(1), Yb(2)–C(1) 2.596(8); Yb(1)–C(7) 2.61(1), Yb(2)–C(7) 2.60(1); Yb(1)–C(13) 2.66(1), Yb(2)–C(13) 2.62(1) Å. The range of changing of these bond lengths in **1** of 2.60(1)–2.66(1) Å is significantly closer than that in **2** (2.48(3)–2.75(4) Å).

The bridging Ph rings in **1**, appear to be unsymmetrical relative to Yb(1) and Yb(2) atoms, as has been observed for **2**. The angles between average planes of C(1–6), C(7–12), C(13–18) rings and corresponding Yb–C(1,7,13)–Yb planes are 59.5, 70.2 and 68.2 °. In the two symmetrically independent molecules of **2** the analogous angles are 61, 73, 72 ° and 70, 79, 53 °. The arrangement of bridging Ph groups with respect to Yb(1) and Yb(2) atoms in the molecules of **1** and **2** differ noticeably. In the molecule of **2** all three bridging Ph groups are bent out from the C(1,7,13) plane to the side of the Yb(2) atom [3]. In **1** two rings C(7–12), C(13–18) are bent to the side of Yb(2) and one ring C(1–6) is bent to Yb(1). This can be seen from the deviation of the C(4), C(10) and C(16) atoms from the plane of C(1,7,13), which are −0.23, 0.35 and 0.32 Å, respectively.

The angles between bridging Yb(1)–C(1,7,13) bonds and average planes of corresponding Ph groups in the Yb(μ-Ph)$_3$Yb moieties of **1** and **2** differ. In **2** these angles are 18, 22 and 20 ° but in **1** they are essentially greater, 38, 30 and 30 °. The Yb(1) $\cdots$ C(6) of 3.12(1) and Yb(2) $\cdots$ C(2) of 3.18(1) Å distances in the bridging C(1–6) ring are practically the same, whereas in the two other groups these separations differ significantly: Yb(1) $\cdots$ C(8) 3.62(2), Yb(2) $\cdots$ C(12) 3.35(1) and Yb(1) $\cdots$ C(18) 3.41(1), Yb(2) $\cdots$ C(14) 3.15(1) Å. (The Yb(2) $\cdots$ C distances in **2** are noticeably shorter 2.90, 2.99 and 2.88 Å [3]). Thus, the bonding scheme of Ph groups in **1** may be represented as $\eta^2:\eta^2$; $\eta^1:\eta^2$; $\eta^1:\eta^1$.

In contrast to **2**, in the molecule of **1** the H atoms of bridging Ph groups were located objectively, from difference Fourier synthesis. The geometry of these C–H bonds has shown the absence of any interactions of these protons with Yb(1) or Yb(2) atoms.

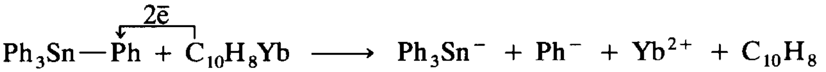
The Yb–O bond lengths in **1** lie in the closed range 2.454(8)–2.492(5) Å. In the molecule of **2** this range is wider, 2.30–2.53 Å. The valent angles O(3)–Yb(2)–O(4) 86.4(3), O(3)–Yb(2)–O(5) 81.1(2) and O(4)–Yb(2)–O(5) 86.7(2)° at the Yb(2) atom are closed to the angles of O(1)–Yb(1)–O(2) 85.5(2), Sn–Yb(1)–O(1) 85.4(2) and Sn–Yb(1)–O(2) 84.2(2)° at the Yb(1) atom.

Discussion

Thus, the interaction of naphthaleneytterbium with Ph$_4$Sn can be described by the following equation:

$$3\ \mathrm{Ph_4Sn} + 3\ \mathrm{C_{10}H_8Yb(THF)_2} \rightarrow \mathrm{Ph_3SnYb(THF)_2(\mu\text{-}Ph)_3Yb(THF)_3} + \mathrm{(Ph_3Sn)_2Yb(THF)_4} + 3\ \mathrm{C_{10}H_8}$$

It seems that the initial stage in this reaction is a transfer of two electrons from C$_{10}$H$_8$Yb(THF)$_2$ to the molecule of tetraphenyltin which leads to the splitting of one Ph–Sn bond and formation of Ph_3Sn^- and Ph^- anion moieties on the one hand and formation of free naphthalene and Yb^{2+} cation on the other hand. The nature of the initial naphthaleneytterbium complex remains unestablished [5] but both most possible variants of its arrangement (ionic or π-complex) after two-electron oxidation should result in the appearance Yb^{2+} cation in solution. If initial naphthaleneytterbium has ionic nature $C_8H_8^{2-}Yb^{2+}(THF)_2$ (contact ionic pair), the source of electrons could be naphthalene dianion $C_{10}H_8^{2-}$. In the second case, i.e. the neutral π-complex nature of naphthaleneytterbium, the electrons could leave from the highest occupied molecular orbital of the complex.



$$Ph_3Sn\text{—}Ph + C_{10}H_8Yb \longrightarrow Ph_3Sn^- + Ph^- + Yb^{2+} + C_{10}H_8$$

Coupling of anion moieties with Yb^{2+} cation in different combinations can lead to the formation of three types of complexes: Ph_3Sn–Yb–$SnPh_3$, Ph_3Sn–Yb–Ph and Ph–Yb–Ph. The first has been isolated and characterized in a previous report [1]. The last two compounds associate to give **1**. The reason for such association can be coordinating unsaturation of Yb atoms in these intermediates (in contrast to distannyl complex) and their poor steric crowding. Similar processes obviously take place in the reaction of Ph_2Hg with $C_{10}H_8Yb(THF)_2$, which leads to the binuclear complex $Ph_2Yb(THF)(\mu\text{-}Ph)_3Yb(THF)_3$ [3].

There is another possible scheme for these reactions. In this scheme the first part of the overall sequence involves the insertion of ytterbium into the Sn–Ph bond to form the Ph_3Sn–Yb–Ph complex, which undergoes a following disproportionation to give Ph_3Sn–Yb–$SnPh_3$ and Ph–Yb–Ph. The last stage in this case is, like the first scheme, association of Ph_3Sn–Yb–Ph and Ph–Yb–Ph, leading to the observed product **1**. The main difference between these schemes is the character of the intermediates formed in the initial stage of the process. According to redox properties of ytterbium and tin and some common rules previously established for similar reactions [6], the first scheme seems to be more probable.

Experimental

The instrumentation and general procedures employed for this study were identical to those given in a previous paper [1].

Synthesis of $Ph_3SnYb(THF)_2(\mu\text{-}Ph)_3Yb(THF)_3$ *(1)*

$C_{10}H_8Yb(THF)_2$ (1.14 g, 2.64 mmol) was added to a solution of Ph_4Sn (1.13 g, 2.64 mmol) in 30 ml of THF. The mixture was shaken at room temperature for 48 h, during which the precipitate of $C_{10}H_8Yb(THF)_2$ dissolved almost completely and the solution became a dark-brown. After centrifuging, the solution was decanted from the small amount of precipitate and THF was evaporated *in vacuo* to 10 ml. The addition of 20 ml ether precipitates dark-ruby almost black crystals of **1** (0.50 g, 44%, m.p. 110–115 °C(dec)). Anal. $C_{56}H_{70}O_5SnYb_2$ calc.: C, 52.22; H, 5.45; Yb, 26.87. Found: C, 52.33; H, 5.57; Yb, 26.70%. IR(nujol): 3020, 1420 (doublet), 1400, 1210, 1070, 1055, 1030, 990, 970, 875, 730, 720, 695, 670, 610, 450 (doublet) cm^{-1}.

Table 1

Atom coordinates ($\times 10^4$) and temperature factors ($Å^2 \times 10^3$)

Atom	x	y	z	U [a]
Yb(1)	274(1)	−1884(1)	7019(1)	29(1)
Yb(2)	1760(1)	594(1)	7754(1)	36(1)
Sn	−1374(1)	−4009(1)	7580(1)	34(1)
O(1)	−1431(6)	−2678(5)	5884(4)	60(3)
O(2)	1344(6)	−3011(5)	6404(4)	48(3)
O(3)	1822(6)	2101(4)	7272(4)	52(3)
O(4)	1388(7)	1549(5)	8906(4)	71(4)
O(5)	4115(6)	1697(5)	8207(4)	56(3)
C(1)	−730(8)	−476(6)	7459(6)	41(4)
C(2)	−1187(9)	130(7)	7074(6)	50(5)
C(3)	−2421(11)	164(9)	7042(8)	79(7)
C(4)	−3259(12)	−426(11)	7411(11)	126(10)
C(5)	−2874(12)	−1028(9)	7801(10)	119(9)
C(6)	−1629(10)	−1052(7)	7818(7)	66(6)
C(7)	2113(8)	−936(7)	8201(8)	43(5)
C(8)	1960(11)	−688(7)	8926(11)	61(7)
C(9)	2784(17)	−750(9)	9530(8)	92(8)
C(10)	3771(15)	−1092(12)	9401(12)	105(9)
C(11)	3952(11)	−1355(10)	8704(12)	86(9)
C(12)	3153(9)	−1264(7)	8120(7)	58(6)
C(13)	1749(8)	−453(6)	6427(6)	37(4)
C(14)	3126(8)	−143(6)	6485(6)	38(4)
C(15)	3725(10)	82(7)	5889(9)	50(5)
C(16)	3019(11)	12(8)	5218(8)	57(5)
C(17)	1696(10)	−287(7)	5137(7)	53(6)
C(18)	1096(8)	−503(6)	5733(8)	42(5)
C(19)	−2554(12)	−2404(9)	5745(9)	108(8)
C(20)	−3675(10)	−3381(12)	5604(9)	109(8)
C(21)	−3223(11)	−4199(9)	5269(9)	94(7)
C(22)	−1772(10)	−3748(8)	5473(7)	65(6)
C(23)	1792(10)	−3726(7)	6729(7)	64(6)
C(24)	3048(13)	−3622(11)	6479(9)	102(9)
C(25)	3119(13)	−3078(12)	5872(9)	109(10)
C(26)	1797(10)	−2990(7)	5717(6)	54(5)
C(27)	1691(15)	3003(8)	7729(8)	109(8)
C(28)	1938(13)	3790(8)	7334(9)	90(8)
C(29)	2068(20)	3318(11)	6605(10)	145(12)
C(30)	1840(12)	2232(8)	6533(7)	76(6)
C(31)	213(12)	1518(9)	9143(10)	96(8)
C(32)	553(21)	2070(16)	9936(11)	142(13)
C(33)	1703(24)	1855(11)	10171(9)	148(12)
C(34)	2393(15)	2006(10)	9535(10)	110(8)
C(35)	4831(9)	2345(7)	7779(7)	67(6)
C(36)	6077(11)	2202(11)	7809(10)	102(8)
C(37)	6346(10)	2065(12)	8568(9)	109(8)
C(38)	5030(11)	1632(10)	8773(8)	89(7)
C(39)	−3024(7)	−4183(6)	8176(6)	36(4)
C(40)	−3989(8)	−3804(7)	7967(6)	47(5)
C(41)	−5046(9)	−3926(8)	8333(7)	59(6)
C(42)	−5143(9)	−4395(9)	8914(8)	67(6)
C(43)	−4192(9)	−4773(8)	9125(6)	57(5)
C(44)	−3151(8)	−4658(7)	8757(6)	46(5)
C(45)	−2331(8)	−5586(6)	6803(5)	38(4)

Table 1 (continued)

Atom	x	y	z	U [a]
C(46)	−3680(9)	−6045(7)	6551(6)	52(5)
C(47)	−4243(10)	−7035(8)	6053(7)	66(5)
C(48)	−3509(12)	−7592(7)	5810(7)	71(6)
C(49)	−2190(11)	−7172(7)	6059(7)	67(6)
C(50)	−1614(8)	−6183(7)	6543(6)	49(4)
C51)	−142(7)	−4429(6)	8363(5)	34(4)
C(52)	−471(8)	−5419(6)	8493(6)	46(4)
C(53)	298(11)	−5634(8)	9028(7)	58(6)
C(54)	1416(10)	−4866(10)	9433(7)	64(6)
C(55)	1776(10)	−3894(9)	9311(7)	68(6)
C(56)	1012(8)	−3672(7)	8782(6)	46(4)

[a] Equivalent isotropic U defined as one-third of the trace of the orthogonalised U_{ij} tensor.

The solvent was removed *in vacuo* from the mother solution and the remaining solid was extracted with ether (30 ml, ×3). Cooling at −5 °C during 24 h yields yellow crystals of $(Ph_3Sn)_2Yb(THF)_4$ (0.37 g, 36%, m.p. 127–132 °C(dec.)). Naphthalene (0.096 g, 85%) was detected in the mother liquor.

Table 2

Bond lengths (Å)

Yb(1)–Yb(2)	3.226(1)	C(23)–C(24)	1.49(2)	C(11)–C(12)	1.39(2)
Yb(1)–Sn	3.379(1)	C(24)–C(25)	1.49(3)	C(13)–C(14)	1.43(1)
Yb(1)–O(1)	2.467(6)	C(25)–C(26)	1.52(2)	C(13)–C(18)	1.40(2)
Yb(1)–O(2)	2.467(7)	O(3)–C(27)	1.44(2)	C(14)–C(15)	1.40(2)
Yb(1)–C(1)	2.62(1)	O(3)–C(30)	1.44(2)	C(15)–C(16)	1.38(2)
Yb(1)–C(6)	3.12(1)	C(27)–C(28)	1.43(2)	C(16)–C(17)	1.37(2)
Yb(1)–C(7)	2.61(1)	C(28)–C(29)	1.44(2)	C(17)–C(18)	1.40(2)
Yb(1)–C(12)	3.35(1)	C(29)–C(30)	1.44(2)	C(39)–C(40)	1.41(1)
Yb(1)–C(13)	2.66(1)	O(4)–C(31)	1.41(2)	C(39)–C(44)	1.39(2)
Yb(1)–C(18)	3.41(1)	O(4)–C(34)	1.40(2)	C(40)–C(41)	1.40(2)
Yb(2)–O(3)	2.457(7)	C(31)–C(32)	1.47(2)	C(41)–C(42)	1.38(2)
Yb(2)–O(4)	2.454(8)	C(32)–C(33)	1.45(4)	C(42)–C(43)	1.39(2)
Yb(2)–O(5)	2.492(5)	C(33)–C(34)	1.50(3)	C(43)–C(44)	1.39(2)
Yb(2)–C(1)	2.596(8)	O(5)–C(35)	1.43(1)	C(45)–C(46)	1.40(1)
Yb(2)–C(2)	3.18(1)	O(5)–C(38)	1.43(2)	C(45)–C(50)	1.40(1)
Yb(2)–C(7)	2.60(1)	C(35)–C(36)	1.46(2)	C(46)–C(47)	1.40(1)
Yb(2)–C(8)	3.16(2)	C(36)–C(37)	1.49(3)	C(47)–C(48)	1.36(2)
Yb(2)–C(13)	2.62(1)	C(37)–C(38)	1.51(2)	C(48)–C(49)	1.37(2)
Yb(2)–C(14)	3.15(1)	C(1)–C(2)	1.41(2)	C(49)–C(50)	1.38(1)
Sn–C(39)	2.22(1)	C(1)–C(6)	1.39(2)	C(51)–C(52)	1.40(1)
Sn–C(45)	2.216(7)	C(2)–C(3)	1.38(2)	C(51)–C(56)	1.38(1)
Sn–C(51)	2.21(1)	C(3)–C(4)	1.37(2)	C(52)–C(53)	1.40(2)
O(1)–C(19)	1.44(2)	C(4)–C(5)	1.37(3)	C(53)–C(54)	1.36(1)
O(1)–C(22)	1.44(1)	C(5)–C(6)	1.39(2)	C(54)–C(55)	1.37(2)
C(19)–C(20)	1.46(2)	C(7)–C(8)	1.38(3)	C(55)–C(56)	1.39(2)
C(20)–C(21)	1.46(2)	C(7)–C(12)	1.39(2)		
C(21)–C(22)	1.49(2)	C(8)–C(9)	1.41(2)		
O(2)–C(23)	1.47(2)	C(9)–C(10)	1.37(3)		
O(2)–C(26)	1.44(1)	C(10)–C(11)	1.34(3)		

Table 3

Bond angles (degrees)

Yb(2)–Yb(1)–Sn	137.1(1)	Yb(1)–C(7)–C(9)	146.4(6)
Sn–Yb(1)–O(1)	85.4(2)	Yb(1)–C(7)–C(12)	109.6(8)
Sn–Yb(1)–O(2)	84.2(2)	Yb(2)–C(7)–C(12)	126.3(9)
O(1)–Yb(1)–O(2)	85.5(2)	C(7)–C(8)–C(9)	123.3(13)
Sn–Yb(1)–C(1)	106.7(2)	C(8)–C(9)–C(10)	119.6(16)
O(1)–Yb(1)–C(1)	90.3(3)	C(9)–C(10)–C(11)	119.4(18)
O(2)–Yb(1)–C(1)	168.0(3)	C(10)–C(11)–C(12)	120.2(14)
Sn–Yb(1)–C(7)	97.9(3)	C(7)–C(12)–C(11)	124.2(14)
O(1)–Yb(1)–C(7)	176.6(3)	Yb(1)–C(13)–Yb(2)	75.4(3)
O(2)–Yb(1)–C(7)	95.9(3)	Yb(1)–C(13)–C(14)	125.4(7)
C(1)–Yb(1)–C(7)	87.8(3)	Yb(2)–C(13)–C(14)	97.8(6)
Sn–Yb–C(13)	169.1(2)	Yb(1)–C(13)–C(18)	110.8(5)
O(1)–Yb(1)–C(13)	92.6(3)	Yb(2)–C(13)–C(18)	130.3(7)
O(2)–Yb(1)–C(13)	85.0(3)	C(14)–C(13)–C(18)	113.6(10)
C(1)–Yb(1)–C(13)	84.0(3)	C(13)–C(14)–C(15)	121.5(10)
C(7)–Yb(1)–C(13)	84.5(4)	C(14)–C(15)–C(16)	121.8(10)
O(3)–Yb(2)–O(4)	86.4(3)	C(15)–C(16)–C(17)	118.6(13)
O(3)–Yb(2)–O(5)	81.1(2)	C(16)–C(17)–C(18)	119.7(12)
O(4)–Yb(2)–O(5)	86.7(2)	C(13)–C(18)–C(17)	124.7(9)
O(3)–Yb(2)–C(1)	100.5(3)	O(1)–C(19)–C(20)	105.3(11)
O(4)–Yb(2)–C(1)	86.2(3)	C(19)–C(20)–C(21)	105.1(11)
O(5)–Yb(2)–C(1)	172.6(3)	C(20)–C(21)–C(22)	106.7(9)
O(3)–Yb(2)–C(7)	169.4(3)	O(1)–C(22)–C(21)	105.9(10)
O(4)–Yb(2)–C(7)	99.5(4)	O(2)–C(23)–C(24)	104.0(11)
O(5)–Yb(2)–C(7)	90.5(2)	C(23)–C(24)–C(25)	107.6(13)
C(1)–Yb(2)–C(7)	88.7(3)	C(24)–C(25)–C(26)	105.0(12)
O(3)–Yb(2)–C(13)	89.9(3)	O(2)–C(26)–C(25)	103.9(10)
O(4)–Yb(2)–C(13)	170.0(3)	O(3)–C(27)–C(28)	107.7(12)
O(5)–Yb(2)–C(13)	102.0(2)	C(27)–C(28)–C(29)	106.8(12)
C(1)–Yb(2)–C(13)	85.3(3)	C(28)–C(29)–C(30)	109.6(14)
C(7)–Yb(2)–C(13)	85.6(4)	O(3)–C(30)–C(29)	105.3(10)
Yb(1)–Sn–C(39)	126.2(2)	O(4)–C(31)–C(32)	107.3(13)
Yb(1)–Sn–C(45)	122.4(3)	C(31)–C(32)–C(33)	100.9(17)
C(39)–Sn–C(45)	93.8(3)	C(32)–C(33)–C(34)	100.9(16)
Yb(1)–Sn–C(51)	113.8(2)	O(4)–C(34)–C(33)	104.0(13)
C(39)–Sn–C(51)	97.6(4)	O(5)–C(35)–C(36)	105.8(11)
C(45)–Sn–C(51)	96.9(3)	C(35)–C(36)–C(37)	103.0(13)
Yb(1)–O(1)–C(19)	124.9(6)	C(36)–C(37)–C(38)	105.6(11)
Yb(1)–O(1)–C(22)	121.3(6)	O(5)–C(38)–C(37)	105.9(12)
C(19)–O(1)–C(22)	107.6(7)	Sn–C(39)–C(40)	119.8(8)
Yb(1)–O(2)–C(23)	125.4(7)	Sn–C(39)–C(44)	122.6(7)
Yb(1)–O(2)–C(26)	126.6(6)	C(40)–C(39)–C(44)	117.6(9)
C(23)–O(2)–C(26)	107.5(8)	C(39)–C(40)–C(41)	120.2(11)
Yb(2)– O(3)–C(27)	121.0(7)	C(40)–C(41)–C(42)	121.0(11)
Yb(2)–O(3)–C(30)	130.3(6)	C(41)–C(42)–C(43)	119.3(11)
C(27)–O(3)–C(30)	108.4(9)	C(42)–C(43)–C(44)	119.6(12)
Yb(2)–O(4)–C(31)	130.5(7)	C(39)–C(44)–C(43)	122.4(10)
Yb(2)–O(4)–C(34)	119.8(9)	Sn–C(45)–C(46)	122.2(7)
C(31)–O(4)–C(34)	107.5(11)	Sn–C(45)–C(49)	150.1(5)
Yb(2)–O(5)–C(35)	121.0(6)	Sn–C(45)–C(50)	121.5(5)
Yb(2)–O(5)–C(38)	129.2(6)	C(46)–C(45)–C(50)	116.2(7)
C(35)–O(5)–C(38)	108.1(8)	C(45)–C(46)–C(47)	120.4(10)
Yb(1)–C(1)–Yb(2)	76.3(3)	C(46)–C(47)–C(48)	121.6(9)

Table 3 (continued)

Yb(1)–C(1)–C(2)	131.4(8)	C(47)–C(48)–C(49)	119.2(9)
Yb(2)–C(1)–C(2)	100.9(5)	C(47)–C(48)–C(50)	89.0(6)
Yb(1)–C(1)–C(6)	97.3(7)	C(49)–C(48)–C(50)	30.3(6)
Yb(2)–C(1)–C(6)	136.1(8)	C(48)–C(49)–C(50)	119.9(11)
C(2)–C(1)–C(6)	114.2(9)	C(45)–C(50)–C(49)	122.6(9)
C(1)–C(2)–C(3)	124.2(11)	Sn–C(51)–C(52)	124.2(5)
C(2)–C(3)–C(4)	118.4(14)	Sn–C(51)–C(56)	119.4(7)
C(3)–C(4)–C(5)	120.5(13)	C(52)–C(51)–C(56)	116.3(9)
C(4)–C(5)–C(6)	119.8(14)	C(51)–C(52)–C(53)	122.4(7)
Yb(1)–C(7)–Yb(2)	76.5(4)	C(52)–C(53)–C(54)	119.5(11)
Yb(1)–C(7)–C(8)	127.2(7)	C(53)–C(54)–C(55)	119.5(12)
Yb(2)–C(7)–C(8)	100.6(8)	C(54)–C(55)–C(56)	121.3(8)
		C(51)–C(56)–C(55)	121.0(9)

Reaction of 1 with bromine

To a solution of 0.167 g (0.130 mmol) of **1** in 2 ml of dimethoxyethane was added by cooling a solution of 0.4 ml bromine in 1 ml dimethoxyethane until the yellow colour of the mixture appeared. The organic layer was decanted from the precipitate of $YbBr_3$ (0.105 g, 98%). PhBr (0.056 g, 92%), was detected by GLC in the volatile products. The mixture of Ph_3SnBr, Ph_2SnBr_2 and $PhSnBr_3$ (0.052 g, 93%) was detected in non-volatile residue by LSC.

X-Ray analysis

X-Ray analysis was carried out with a Siemens R3/PC diffractometer at 193 K (λ(Mo-K_α); $\theta/2\theta$-scan in the range $2° \leqslant 2\theta \leqslant 50°$). 4955 reflections with $F > 3\sigma(F)$ were used in the structure determination and refinement. The crystals of **1** are triclinic: $a = 11.123(2)$, $b = 14.078(3)$, $c = 18.774(4)$ Å, $\alpha = 101.94(2)$, $\beta = 96.20(2)$, $\gamma = 109.85(2)$; $V = 2654.1(2.5)$, $Z = 2$, space group $P\bar{1}$. The structure was obtained using a combination of direct and difference map methods. The positions of Yb and Sn atoms were located by primary E synthesis, the positions of other atoms, including H atoms, by subsequent electron density syntheses. The absorption of X-rays in the crystal (μ(Mo-K_α) = 44.61 cm^{-1}) was taken into account by DIFABS program [7]. The structure was refined using full-matrix least-squares method with anisotropical thermal parameters for all non-hydrogen atoms and isotropical for hydrogen atoms. The final discrepancy factors: $R = 0.027$, $R_w = 0.027$. Coordinates of non-hydrogen atoms are given in Table 1, bond distances and angles in Tables 2 and 3 respectively, and coordinates of hydrogen atoms in Table 4.

Table 4

Hydrogen coordinates ($\times 10^3$) and temperature factors ($Å^2 \times 10^2$)

Atom	*x*	*y*	*z*	*U*
H(2)	−56(7)	60(5)	679(4)	7(2)
H(3)	−270(7)	60(5)	666(4)	9(2)
H(4)	−407(7)	−37(5)	745(4)	10(2)
H(5)	−339(8)	−144(7)	809(5)	14(3)
H(6)	−143(7)	−155(5)	805(4)	7(2)
H(8)	111(7)	−58(5)	883(4)	8(2)
H(9)	262(8)	−63(7)	1000(5)	7(3)
H(10)	431(8)	−122(7)	977(5)	13(3)
H(11)	475(7)	−156(5)	862(4)	8(2)
H(12)	329(8)	−143(7)	762(5)	8(3)
H(14)	366(7)	−13(5)	700(4)	9(2)
H(15)	485(7)	27(5)	593(4)	11(2)
H(16)	334(8)	12(7)	479(5)	9(3)
H(17)	112(7)	−33(5)	474(4)	7(2)
H(18)	12(7)	−79(5)	561(4)	7(2)
H(191)	−237(7)	170(5)	607(5)	9(2)
H(192)	−249(8)	−221(7)	526(5)	13(3)
H(201)	−450(7)	−336(5)	540(5)	10(2)
H(202)	−376(8)	−350(7)	608(5)	12(3)
H(211)	−364(8)	−440(7)	463(5)	12(3)
H(212)	−359(8)	−482(7)	535(5)	14(3)
H(221)	−123(7)	−416(5)	562(4)	9(2)
H(222)	−148(8)	−365(7)	494(5)	11(3)
H(231)	116(7)	−449(5)	646(4)	7(2)
H(232)	184(7)	−330(5)	710(4)	8(2)
H(241)	319(7)	−440(5)	633(4)	10(2)
H(242)	369(8)	−322(7)	688(5)	15(3)
H(251)	330(8)	−351(7)	537(5)	16(3)
H(252)	383(8)	−234(7)	600(5)	16(3)
H(261)	153(7)	−373(5)	528(4)	9(2)
H(262)	193(7)	−230(5)	557(4)	11(2)
H(271)	256(7)	323(5)	799(4)	10(2)
H(272)	102(8)	285(7)	796(5)	13(3)
H(281)	271(8)	434(7)	761(5)	19(3)
H(282)	130(8)	412(7)	738(5)	18(3)
H(291)	279(8)	358(7)	646(5)	19(3)
H(292)	148(8)	345(7)	628(5)	15(3)
H(301)	261(7)	212(5)	650(4)	7(2)
H(302)	113(7)	166(5)	625(4)	11(2)
H(311)	−35(7)	66(6)	894(4)	10(2)
H(312)	−43(7)	150(6)	880(4)	8(2)
H(321)	2(7)	197(5)	1027(4)	7(2)
H(322)	78(7)	303(5)	986(4)	12(2)
H(331)	235(7)	242(5)	1069(4)	11(2)
H(332)	146(8)	125(7)	1019(5)	13(3)
H(341)	253(7)	272(5)	951(4)	8(2)
H(342)	304(7)	150(5)	949(4)	10(2)
H(351)	484(8)	302(7)	801(5)	12(3)
H(352)	429(8)	209(7)	726(5)	12(3)
H(361)	677(8)	276(7)	769(5)	16(3)
H(362)	599(8)	159(7)	738(5)	14(3)
H(371)	682(8)	275(7)	895(5)	17(3)
H(372)	676(8)	167(7)	862(5)	12(3)

Table 4 (continued)

Atom	*x*	*y*	*z*	*U*
H(381)	497(7)	88(5)	853(4)	11(2)
H(382)	496(8)	183(7)	926(5)	14(3)
H(40)	−392(7)	−340(5)	756(4)	6(2)
H(41)	−567(7)	−366(5)	823(4)	8(2)
H(42)	−598(7)	−453(5)	915(4)	9(2)
H(43)	−427(7)	−516(5)	964(4)	7(2)
H(44)	−250(7)	−497(5)	895(4)	6(2)
H(46)	−422(7)	−568(5)	674(4)	5(2)
H(47)	−523(7)	−742(5)	586(4)	8(2)
H(48)	−377(7)	−833(5)	555(4)	7(2)
H(49)	−165(7)	−764(5)	587(4)	9(2)
H(50)	−65(7)	−592(5)	679(4)	7(2)
H(52)	−116(7)	−597(5)	819(4)	5(2)
H(53)	−17(7)	−650(5)	891(4)	10(2)
H(54)	204(7)	−491(5)	987(4)	8(2)
H(55)	254(7)	−322(5)	967(4)	8(2)
H(56)	128(7)	−301(5)	868(4)	6(2)

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